

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 4, 1929.

I.—GENERAL; PLANT; MACHINERY.

Automatic control through temperature or pressure. C. J. SWAN (Ind. Eng. Chem., 1928, 20, 1152—1155).—The electric switch used consists of a sealed glass tube containing inert gases and a small quantity of mercury. The tilting of the tube causes the mercury to make or break the circuit. The movement is effected by the pressure within a metal bellows operating against an adjustable spring. For pressure control the apparatus to be controlled is connected to the bellows. For temperature control the bellows is charged with a liquid of suitable b.p. The tension of the spring regulates the sensitiveness of the instrument. Unless the unit to be controlled is electrical a motor valve is used. The device is suitable for controlling the supply of steam or hot water to dryers, for maintaining an even gas pressure with a number of boosters, and for maintaining constant levels in tanks.

C. IRWIN.

Gas flow through packed columns. S. P. BURKE and W. B. PLUMMER (Ind. Eng. Chem., 1928, 20, 1196—1200).—The pressure drop in a packed column $p/l = C(\rho V^2 S/f^3)$, where V is the linear velocity, S the surface per unit packed volume, and f the free volume per unit packed volume. C is a function of $\mu S/\rho V$, where μ is the viscosity of the gas and ρ the density. The pressure drop for air at 27° flowing with known velocity through columns packed with spheres of various sizes was determined. A further curve was then obtained by plotting C (values from above equation) against $\mu S/\rho V$. This curve expresses both viscous and turbulent flow. The principal difficulty in the practical application of the equations corresponding to this curve is in the accurate determination of f . They have, however, checked well with tests on scrubbers etc. Special (e.g., hollow) packing material requires empirical compensations to be applied.

C. IRWIN.

Loss calculations in dissolving, leaching, and extraction. C. V. IREDELL (Chem. Met. Eng., 1928, 35, 685—686).—In extraction of one solid from a mixture of solids by means of a liquid, loss is due to incomplete removal of the desired substance, and the extent of this loss can be controlled by means of suitable analysis. Provided that the one constituent only is extracted, that any change in the insoluble residue can be easily reversed, and that the extent of chemical change in the substance removed is known both in the solution and in the residue, then the % loss (L) is given by the equation $L = 100B(1-A)/A(1-B)$, where A is the weight of the desired substance per unit weight of dry raw material, and B is the weight of the same substance per unit weight of dry residue. When chemical change occurs modifications are introduced

into this equation, and their mathematical derivation is described.

R. H. GRIFFITH.

Suspension of macroscopic particles in a turbulent gas stream. S. P. BURKE and W. B. PLUMMER (Ind. Eng. Chem., 1928, 20, 1200—1204).—The force (R) acting between a sphere and a turbulent gas stream flowing past it is: $R = k\rho V^2 r^2$, where r is the radius of the sphere. For particles of other shapes πr^2 is replaced by other expressions corresponding to the maximum cross-section. The value of k has been determined by measuring the apparent loss in weight of spheres etc. suspended from a balance arm in an upward gas current and averages 0.00084. For irregular coke particles the average value was 0.00093. This value was confirmed by data obtained by measuring the suspension velocities for coke particles in a vertical air current. These results apply only to the case where the whole body of moving gas is in turbulent flow.

C. IRWIN.

Approximate determination of the absolute magnitude of the pores of porous materials. M. RABINOVITSCH and N. FORTUNATOV (Z. angew. Chem., 1928, 41, 1222—1226).—A number of pieces of the porous material of approximately the same shape and with as smooth a surface as possible are first dried at 120°, cooled in a desiccator over phosphorus pentoxide, and weighed. They are then attached to light wires and suspended in a closed vessel over water which is heated at 60—70°, whilst the air is pumped out of the vessel; the test-pieces are then allowed to fall into the water, and after cooling are removed, the adhering water is absorbed with filter paper, and the pieces are weighed again. The volume of water absorbed gives the volume of the pores. The pieces are then placed in a desiccator containing a large volume of sulphuric acid at 0° until a constant weight is obtained; from this weight the volume of the pores still filled with water at the vapour pressure in the desiccator is obtained. The mean radius of the pores that remain filled with water can then be calculated, assuming that they are small capillaries, from Minkovski's equation (cf. Anderson, A., 1914, ii, 632). Tests on various charcoals show that the pores of active carbon consist almost entirely of these micropores having a radius smaller than 10 μ . The proportion of the volume of the micro-pores to that of the total pores for various porous materials has been determined.

A. R. POWELL.

Apparatus for rapid sedimentation analysis. C. J. VAN NIEUWENBURG and W. SCHOUTENS (J. Amer. Ceram. Soc., 1928, 11, 696—705).—The apparatus for the continuous determination of the distribution curve of particle size consists of a sedimentation vessel in

the form of a Schöne tube, and a long conical underpart, which, in certain experimental circumstances, prevents the formation of vortices. The powdered material (20 g.), dried at 110°, is washed through a conical sieve (30 meshes per linear cm.) into the sedimentation vessel, the desired volume being obtained by the addition of distilled water. In this liquid a weighted, hollow glass body (wt. under water 3—5 g.), which is attached to a balance of regulated movement and sensitivity, is placed at a predetermined position. After compressed air has been admitted in order to render the suspension homogeneous, readings of the movement of the balance pointer are regularly taken. The measured increase in the weight of the suspended body at any moment is related to the weight of the particles which have settled. Two further points on the curve are obtained independently, (1) by measuring the residual solid matter in the suspension after the conclusion of the experiment, (2) by conducting an elutriation in the same apparatus. The radius of the particles is calculated by the application of Stokes' formula. An allowance for a "contraction factor," due to the reduction of the effective cross-section of the tube by the suspended body, is essential. During all experiments the temperature must be maintained constant.

A. T. GREEN.

Apparatus for testing filtering etc. media. LINSBAUER and VAŠÁTKO.—See II. Lovibond glasses. JUDD.—See VIII. Aeration of liquids. STICH.—See XVIII.

PATENTS.

Calcining furnace. J. H. KNAPP, Assr. to TATE, JONES & Co., Inc. (U.S.P. 1,686,565, 9.10.28. Appl., 17.12.24).—The furnace comprises an externally-heated drum; the means for introducing and withdrawing material are connected to the drum in an air-tight manner.

B. M. VENABLES.

Furnaces. C. V. A. ELEY (B.P. 299,527, 2.8.27).—In a furnace of the type having tubular or trough-like air passages below the firebars, the bottoms of the troughs are arranged as doors which can be lowered to discharge fine ash.

B. M. VENABLES.

Heat interchanger. O. E. FRANK, Assr. to O. E. FRANK HEATER & ENGINEERING Co., Inc. (U.S.P. 1,688,183, 16.10.28. Appl., 6.12.27).—The shell of a bundle-of-tubes-type heat exchanger is made in at least two parts so that a baffle for the outer fluid, which slides in grooves formed in the wall of the shell, can be easily removed.

B. M. VENABLES.

Heat-exchanging apparatus. P. LEVEQUE (B.P. 293,759, 10.7.28. Belg., 11.7.27).—Cast metal plates fit together in an air-tight manner by means of flanges and grooves. Fins on one side of the plates receive heat from one fluid and conduct it through the plate to fins on the other side in contact with the other fluid. The fins intercalate to form sinuous passages.

B. M. VENABLES.

Cooling system. W. H. CARRIER, Assr. to BUFFALO FORGE Co. (U.S.P. 1,687,542, 16.10.28. Appl., 28.7.21).—The system comprises a constant body of air in circulation, with liquid sprays in one part and a liquid eliminator in another part of the circuit.

B. M. VENABLES.

Rotary beater-type disintegrating and shredding mills. J. P. VAN GELDER (B.P. 284,230, 20.12.27. Austral., 25.1.27).—The flat ends of disintegrator blades are slit radially (or approximately so) and the tangs thus formed are set to either side alternately.

B. M. VENABLES.

Mixing apparatus. R. B. BROWN (U.S.P. 1,686,593, 9.10.28. Appl., 29.12.24).—A number of knives are fixed in staggered relation on a shaft and rotate within a casing. The material is admitted at the top of one end and exhausted at the bottom of the other end of the casing.

B. M. VENABLES.

Apparatus for mixing materials. A. B. and C. R. SMITH (B.P. 298,929, 16.7.27).—A number of sweeps or stirrers rotate in a stationary pan, and cylindrical or conical rollers co-operate with the upper surfaces of the sweeps.

B. M. VENABLES.

Drying apparatus. MASCHINENFABR. BUCKAU A.-G. ZU MAGDEBURG (B.P. 275,239, 27.7.27. Ger., 27.7.26).—Hot gases are supplied through a drum which rotates in a trough containing the material, and the trough is continued upwards as a casing to collect the vapour. The upper part of the casing is divided into a number of compartments with separate dampers to regulate the withdrawal of vapour.

B. M. VENABLES.

Drying machines. P. TURNER (B.P. 299,134, 20.8.27).—A number of rotating drums are arranged one above the other and are annular; the annuli are divided by radial partitions, and the inner cylindrical walls are perforated and serve for admission of drying fluid. The material itself enters the periphery at the upper part of a drum and leaves at the lower part, dropping into the next drum and so on.

B. M. VENABLES.

Apparatus for extracting moisture from materials. D. M. A. G. HAWES (B.P. 298,524, 8.7.27).—The material falls on to a rapidly rotating disc and is flung on to the interior wall of a less rapidly rotating element preferably shaped as two hollow cones base to base. It is pushed down the sloping face of the cone by means of fixed scrapers, and drops on to another rapidly rotating disc below and so on downwards through many stages. The drying medium is hot air or other gas.

B. M. VENABLES.

Apparatus for drying and handling grain etc. TOMLINSONS (ROCHDALE), LTD., and E. W. SMITH (B.P. 298,369, 10.10.27).—A rotary cylinder to which drying air is admitted has its peripheral wall composed of spaced slats arranged with such an overlap and angle that when the drum is rotated in one direction the material will be completely retained, but when reversed the material will be discharged through the spaces between the slats. A "feathering" device may be added to vary the openings.

B. M. VENABLES.

Separation of materials of different sp. gr. T. M. CHANCE (U.S.P. 1,685,521, 25.9.28. Appl., 28.6.27).—The materials are treated in a pulp comprising a mixture of a fluid and a comminuted solid of sp. gr. intermediate between the constituents to be separated. A number of treating vessels are arranged in staircase form with recirculation of the pulp.

B. M. VENABLES.

Treatment of foundry sand and other finely-divided materials. PNEULEC, LTD., and P. PRITCHARD (B.P. 298,384, 5.11.27).—A rotary drum screen has its screening surface constructed of a number of rods or wires loosely or tightly supported in end members, every or every alternate rod being wound with a helical wire forming a spacer. B. M. VENABLES.

Extracting substances from solid materials. N. NOBEL (B.P. 298,501, 4.7.27).—The extracting liquid flows in a closed circuit, and during part of the circuit the solid material flows with it, being conveyed solely by the flow of the liquid, and is then removed by rakes or similar means. Counter-current extraction may be effected by the use of several liquor circuits. During the extraction period the pulp is subjected to change of pressure, *e.g.*, by pumping-up siphons or by centrifugal means. B. M. VENABLES.

Filter. W. C. GRAHAM (U.S.P. 1,687,863, 16.10.28 Appl., 26.7.24).—Rocking filter trays have trunnions which are mounted on a carrier similar to two wheels abreast. The trunnions on one wheel are connected to a source of vacuum, and the other trunnions to a source of feed-pulp or wash-water according to their position. The passages through the trunnions connect below and above the filter surface in the trays respectively. B. M. VENABLES.

[Sand] filters. B. BRAMWELL (B.P. 298,835, 7.2.28).—The sand is cleaned in the filtering vessel by an upward jet of clean water in conjunction with an open-ended tube which keeps the whole sand in a state of turmoil and removes the silt with the overflow. Several cleaning jets and uptake tubes may be arranged in one vessel, and a single multiple-valve may effect the change of connexions from filtering to cleaning, and may be automatically operated by the increase of pressure due to the filter becoming choked. B. M. VENABLES.

Suction filter. FELD & VORSTMAN GES.M.B.H. (G.P. 452,514, 17.12.24).—The apparatus comprises a suction chamber, a filter surface, and a pressure chamber above it for receiving the material to be filtered, the upper part being hinged so that it can be tilted over to empty it by gravity and to facilitate cleaning and replacing the filter surface. A number of filter units may be arranged in a common pressure chamber. L. A. COLES.

Straining and filtering apparatus. J. G. MCKEAN and R. F. JONES (B.P. 298,856, 23.3.28).—A number of horseshoe-shaped plates are assembled with distance pieces to form narrow spaces between for the flow of liquid. Cleaning fingers rotated by a handle are also interleaved. B. M. VENABLES.

[Dust] filters. J. J. C. BRAND and B. LAING (B.P. 299,080, 19.7.27).—A cone of great height of filtering medium is folded back on itself many times forming a series of concentric, conical surfaces of alternate slope. The whole is hung in a cylindrical container by means of the upper circular edges or folds, and the lower circles are attached to weights or springs so that the filter fabric can be shaken. B. M. VENABLES.

Air filters for separating dust from dust-laden air. E. BUDIL (B.P. 275,257, 29.7.27. Ger., 31.7.26).—

The filter is constructed of an assemblage of polygonal tubes which are perforated with many slots, the material of the slot being bent inwards. B. M. VENABLES.

Apparatus for removing dust from gases. SOC. ANON. DES ÉTABL. NEU (F.P. 628,200, 28.1.27).—Centrifugal force set up in the gas stream by direction against a central cone drives the dust particles into annular cells surrounding the cone. L. A. COLES.

Device for separating dust, water, oil, etc. from air, gases, and vapours. HUNDT & WEBER, GES.M.B.H. (B.P. 298,855, 20.3.28. Ger., 10.11.27).—Within a separating vessel having a tangential supply pipe is a smaller device acting on the same principle, the supply pipes to the inner separator being downwardly inclined as well as tangential, and the final outlet for cleaned gas upwards at the axis of the inner separator. The bottom of the inner separator is held in place only by a spring, so that it can move downwards slightly to permit separated liquid and dirt to pass into the outer collector. B. M. VENABLES.

Combined filter and separator. A. C. HANDLEY (B.P. 299,648, 20.1.28).—An apparatus is described in which the bulk of the oil (from a mixture of oil and water) is separated by gravity and the remainder caught in a fibrous filter. B. M. VENABLES.

Removal in layers of material from vacuum filters, especially from drum filters. R. WOLF A.-G. (G.P. 452,392, 19.3.24).—The apparatus is provided with an outer, fixed scraper and with an inner scraper which penetrates gradually into the filtered material, and is returned automatically to its original position as soon as it reaches the innermost layer. L. A. COLES.

Wet treatment of subdivided materials involving filtration or screening. S. C. SMITH (B.P. 298,172, 5.4. and 18.6.27).—Rubber-woven fabric or perforated sheet is used as a filtering (or percolating) medium either by itself or as a corrosion- and erosion-resisting lining or backing to other filter media. B. M. VENABLES.

Apparatus for distillation of solid matters. H. DUPUY (B.P. 289,425, 22.3.28. Fr., 27.4.27. Addn. to B.P. 198,661; B., 1924, 497).—In the process previously described the steam is superheated by passing over incandescent coal, and an injector is used as the circulating pump. B. M. VENABLES.

Apparatus for separating liquids of different densities. F. PINK (B.P. 298,683, 29.6.27).—The apparatus is of the type through which the mixed fluid flows so slowly (less than 2 ft./min. for oil and water) that stream-line flow is obtained and the two liquids are separated by gravity. B. M. VENABLES.

Apparatus for sterilising liquids. N. V. HUYGEN & WESSEL'S INGENIEURSBUREAU (B.P. 290,555, 25.8.27. Holl., 14.5.27).—The whole steriliser is rocked, and the inner container for the goods is preferably spring-mounted within the outer container for the heating and cooling medium. B. M. VENABLES.

Treatment of liquids with reagents. R. JEANNIN and R. M. BERLINE (F.P. 627,822, 20.1.27).—Reagents

mixed with porous or non-porous, inactive, finely-divided material are brought in intimate contact with the liquid. L. A. COLES.

Apparatus for expelling gases from liquids. A. VOGT (G.P. 452,265, 17.10.24).—A vessel attached to a source of reduced pressure is provided with up- and down-flow pipes both dipping into the same container.

L. A. COLES.

Apparatus for distillation, rectification, or evaporation. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,084, 20.7.27).—The vapour outlet from a still is bent into a hook shape, the discharge being downwards into the reflux tube, so that the vapour has to double back upwards before it can finally exhaust. The reflux tube may continue downwards as a screw column, and a cooler may be provided in the upper part.

B. M. VENABLES.

Treatment of gases with liquids in scrubbing towers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299, 075, 20.6.27).—The tower has no real filling, but is provided with a spiral slide down which the liquid flows in a sheet, the centrifugal force being counteracted by sloping the surface radially as well as helically. The hand of the helix may be changed part way down, and various additional devices, e.g., for temperature regulation, are claimed.

B. M. VENABLES.

Gas washers. M. MALLET (B.P. 297,765, 29.3.28. Fr., 27.9.27).—In an apparatus comprising several comparatively low towers side by side, the circulating pumps draw from sumps intermediate the towers, and to ensure that all the liquid is circulated the level of the liquid in the intermediate sumps is maintained below that in the sumps under the towers.

B. M. VENABLES.

Treatment of a metallic, organic, or other compound, or a gas, by a gaseous agent serving to enter into reaction therewith. SOC. INTERNAT. DES PROC. PRUDHOMME HOUDRY (B.P. 274,846, 12.7.27. Fr., 23.7.26).—A substance is treated by a gaseous reagent in the absence of a true catalyst, but after the gas has passed through an activating ("atomic-rendering") filter which is confined in a separate chamber. The activating filter contains substances similar to those usually used as catalysts.

B. M. VENABLES.

Separation of gases and vapours by means of solid adsorbents. SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIÈRES (Addn. F.P. 31,976, 4.1.26, to F.P. 607,683; B., 1927, 210).—Vacuum apparatus is attached to the outlet of the second cooler in the apparatus described previously so that the water boils at a lower temperature (30–50°) in the evaporator.

L. A. COLES.

Recovery of adsorbed gases from solid adsorbents. I. G. FARBENIND A.-G., Assecs. of G. and R. WIETZEL (G.P. 452,456, 8.4.22).—The gases are recovered in fractions by heating the material and then treating it with steam under reduced pressure. L. A. COLES.

Effecting reactions under pressure. M. TORNOW (G.P. 452,216, 16.1.27).—The reaction vessels are constructed of natural stone.

L. A. COLES.

Colour-estimating apparatus. O. ROSENHEIM, E. H. J. SCHUSTER, and TINTOMETER, LTD. (B.P.

299,194, 31.10.27).—Standard slips of transparent material are uniformly coloured individually, and a number of them are assembled in one plane to form a series, several of which may be mounted behind each other. Every series is movable in both directions in its own plane so that any part can be brought across the line of sight, which is to one side of the centre line of the instrument, while the line of sight to the object to be compared is correspondingly at the other side.

B. M. VENABLES.

Penetrometers. E. and E. A. GRIFFITHS (B.P. 298,662, 12.7.27).—A blunt-nosed plunger surrounded by a guard ring is presented to the surface of the material to be tested. A spring is inserted between the plunger and the operating handle, and the deflexion of the spring is indicated by a pointer which is automatically locked at its reading (of the load) when the plunger has penetrated a predetermined distance.

B. M. VENABLES.

Grinding and pulverising mill. B. SCHERBAUM (U.S.P. 1,691,210, 13.11.28. Appl., 23.12.25. Ger., 31.12.24).—See B.P. 245,435; B., 1926, 695.

Valves for high-pressure and high-temperature purposes. O. D. REDDING (B.P. 286,273, 1.3.28. U.S., 2.3.27).

Apparatus for screening or washing coal, coke, ballast, ores, grain, etc. W. J. FIDDES (B.P. 299,102, 30.6.27).

[Closing device for] filter presses for extracting liquids from viscid substances. B. GOWSHALL (B.P. 298,282, 12.7.27).

Centrifugal apparatus (G.P. 452,486).—See VII. **Gas purification** (G.P. 452,394 and 452,437).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Bristol and Somerset coalfield. Carbonisation of "Parkfield large gas" coal. ANON. (Dept. Sci. Ind. Res., Fuel Res. Survey Paper No. 12, 1928, 39 pp.).—Carbonisation tests have been carried out on a 750-ton consignment of "Parkfield large gas" coal, of which the proximate analysis gave: moisture 1.5%, volatile matter 34.3%, fixed carbon 56.6%, ash 7.6%. In the laboratory assay at 600° the coal yielded a swollen and porous coke similar to that given by typical Yorkshire and Durham gas coals. The coal worked satisfactorily in both vertical (Glover-West) and horizontal retorts; owing to its highly swelling character it presented some difficulty in first starting up in the vertical retorts, but this was overcome either by starting up with another coal or by reducing the temperature of the top combustion chambers. The yields of products per ton of coal from the vertical retorts when using 5% of steam were: coke 13.7 cwt., gas 16,030 cub. ft. (511 B.Th.U./cub. ft.), tar 11.5 gals., ammonium sulphate 22.8 lb. The best results with the horizontal retorts were obtained with charges of 12½ cwt. carbonised for a period of 12 hrs., giving exactly the rated throughput for the setting. Tests were carried out with both producer-gas and water-gas heating. The yields per ton of coal were: coke 14.0 cwt. (including 3.66 cwt. used in the producer), gas

12,930 cub. ft. (568 B.Th.U./cub. ft.), tar 10·4 gals., ammonium sulphate 26·7 lb. The coal did not lend itself to easy carbonisation in low-temperature vertical retorts owing to holding up of the charge. The difficulty was largely overcome by briquetting the fines (with 7% of pitch and 24% of coke breeze) before charging to the retort. Both the high- and low-temperature cokes gave trouble when used for steam-raising purposes under a Lancashire boiler and as producer fuels in a suction-gas plant owing to the fusible nature of the ash.

A. B. MANNING.

Composition of coal—its rational analysis. W. FRANCIS and R. V. WHEELER (J.C.S., 1928, 2967—2979).—A rational analysis of a coal can be given in terms of free hydrocarbons, resins, organised plant entities, and ulmin compounds; chemical compositions of these substances, with the exception of the ulmins, do not vary very much, whether these ingredients form part of a lignite, bituminous coal, or semi-anthracite, but the composition of the ulmin compounds varies, depending on the coals from which they are obtained. The difference in character of these ulmins may be denoted by determining their reactivity with oxygen, which is obtained directly from the amount of oxygen combining with them in a given time and indirectly from the rate of production of alkali-soluble ulmins under standard treatment with an oxidising agent. Examined in this way the ulmins show a graduation in properties in the series: peat, lignite, bituminous coal, anthracite. Rational analyses are given of a series of coals from the Pittsburg seam (U.S.A.), ranging in character from semi-bituminous to semi-anthracite, as well as for a number of coals of younger formation. For the Pittsburg seam in particular the "reactivity index," i.e., degree of oxidation of the ulmins, is shown to afford a measure of its rank or "degree of coalification." Methods of estimating the proportions of hydrocarbons, resins, organised plant entities, and ulmins are given, together with the determination of the "reactivity index" of the last-named.

C. B. MARSON.

Spontaneous heating of coal. J. D. DAVIS and D. A. REYNOLDS (U.S. Bur. Mines, Tech. Paper No. 409, 1928, 74 pp.).—The tendency of a coal to self-heat increases as the size of the particles decreases; fine coal is therefore dangerous to store. Self-heating is more pronounced with coals of low geological rank; these should be stored so that segregation of fines is prevented and foreign matter excluded as much as possible. As to what effect moisture in coal has on spontaneous heating, the authors consider that this is probably determined by the conditions of storage; if those parts of the pile liable to develop spontaneous heating could be wetted, then it might be prevented because the heat required to vaporise the water would be more than the oxidising coal could supply, but wetting down the whole surface of the pile changes the conditions of ventilation and may favour heating at points not reached by the water. Occluded gases probably have little effect. The tendency to self-heat increases with the temperature of initial exposure, hence storage near steam pipes and such like is to be avoided. Finely-divided pyrites increases the risk of spontaneous heating, and should

therefore be absent from storage coals. Oxidation of the coal substance is the main cause; certain organic constituents may contribute more to the heating than others, but no single constituent exerts a predominating influence, to the exclusion of the rest. Spontaneous heating takes place in two stages; the first, which operates at room temperature, is associated with the physical absorption of oxygen and the formation of a coal-oxygen complex; as the temperature rises, this complex decomposes, yielding carbon monoxide, carbon dioxide, and water, which forms the second stage. This stage generates more heat than the first; there is no sharp transition point from one stage to another.

C. B. MARSON.

Causes of oxidation of coal. D. J. W. KREULEN (Chem. Weekblad, 1928, 25, 642—646).—The humic acid formed by heating dry coal in air for 2 hrs. at 175° increases fairly regularly with the proportion of real volatile matter, but there are considerable deviations. The readiness with which a coal oxidises is bound up also with its friability and resistance to grinding. Methods of expressing these factors quantitatively are described.

S. I. LEVY.

The sulphur problem in burning coal. J. F. BARKLEY (U.S. Bur. Mines, Tech. Paper No. 436, 1928, 7 pp.).—The tendency of a coal to clinker, often associated with its content of sulphur, is really due to the presence of pyrites which, when present, gives ferrous sulphide and ferrous silicates—the active clinkering agents. On combustion, usually not more than 10% of the sulphur originally present in a coal is retained by the ash, the remainder passing into the flue gases as sulphur dioxide and trioxide. The method of calculating the temperature of the flue gases at which condensation of (a) water, (b) sulphuric acid will occur is described, as it is considered that such condensation is necessary before appreciable corrosion of iron and steel will take place.

W. H. BLACKBURN.

Laboratory apparatus for testing filtering and decolorising media. A. LINSBAUER and J. VASÁTKO (Z. Zuckerind. Czechoslov., 1928, 53, 25—30).—A small filter has been designed primarily for the examination of decolorising carbon according to the layer method, the active surface taking 2—10 g. of carbon per 100 cm.² Principal advantages claimed are constant working temperature and rate of flow, and an equal pressure over the whole of the filtering surface. Filtration proceeds through the layer horizontally, this being considered to be the only correct means of layer application of decolorising carbons. Liquor used for the tests is clarified, passed through a filter-press, and stored in a tank at a suitable height above the filter.

J. P. OGILVIE.

Wet gas purification. FERBERS (Gas- u. Wasserfach, 1928, 47, 1133—1137).—In dry purification of gas with iron oxide, the greatest drawback is the difficulty of employing material in a sufficiently finely-divided state to give rapid reaction; this has been overcome by suspending the oxide in water, the mixture containing 1 or 2 g. of iron hydroxide per litre in the presence of alkali. A plant capable of treating 100,000 m.³ of gas per day has been built for the Mont Ceniz mine and

contains three washers, 18 m. in useful height and 250 cm. in diam. A test lasting 14 days gave the following results: throughput of gas 67,975—88,585 m.³ per day; hydrogen sulphide in outlet gas 6—0.6 mg./m.³, corresponding with a purification efficiency of 99.97% overall; air used 4—4.5%; daily consumption of caustic soda 250—275 kg., and of iron oxide 80 kg. The sulphur recovered contained 37.5% of water; the working temperature was 35°, and the cost of the process 0.153 pfennig/m.³ of gas treated.

R. H. GRIFFITH.

Removal of tar fog from gases. L. ECK (Chem. Fabr., 1928, 657).—For small-scale experimental purposes tar fog is most readily separated from gas streams by washing, frictional-impact condensation, or electrical precipitation methods. The first type of separator is seldom used, but apparatus of the cyclone (Pelouze-Audouin or Lodge-Cottrell) class can readily be applied.

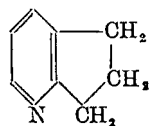
R. H. GRIFFITH.

Indene and styrene. Crude materials in industrial quantities. R. L. BROWN (Ind. Eng. Chem., 1928, 20, 1178—1180).—Thirteen representative samples of the oily condensate which collects in carburetted water-gas mains and attached appliances have been analysed. The fresh, dry liquid contains up to 15% of styrene and 35% of indene. Fractions containing 75% of indene are obtainable by rough fractionation, and with more efficient fractionation fractions containing up to 75% of styrene can be obtained. The remaining 25% consists almost entirely of stable, aromatic hydrocarbons which can be removed by polymerisation of the styrene or by the formation of simple derivatives. The naphthalene contents of the oils are given and the methods of analysis and identification of the components are described. Potential supplies and industrial uses of styrene are discussed (cf. B., 1924, 48).

E. H. SHARPLES.

Basic nitrogen compounds from Fushun shale tar. II. T. EGUCHI (Bull. Chem. Soc. Japan, 1928, 3, 227—243; cf. B., 1927, 696).—A method for the separation of various methylpyridines based on carefully controlled fractional precipitation with picric acid and purification of the various picrate fractions has been worked out and applied to the isolation, in the pure state, of these derivatives in Fushun shale oil. The following have been isolated and their physical constants determined: 4-methylpyridine, b.p. 145.45—145.47°/763.9 mm., d_4^{25} 0.9502 (vacuum), n_D^{25} 1.5029 (picrate, m.p. 167°); 3-methylpyridine, b.p. 144.17—144.18°/763.0 mm., d_4^{25} 0.9518 (vacuum), n_D^{25} 1.5038 (picrate, m.p. 149°); chloroplatinate, m.p. 208°; chloroaurate, m.p. 187°; double mercurichlorides B.HCl.HgCl₂, m.p. 145—146°, B.HCl.2HgCl₂, m.p. 146°, B.HCl.3HgCl₂, m.p. 170°, where B is 1 mol. of the base); 2:3-dimethylpyridine, b.p. 160.7°/760 mm., d_4^{25} 0.9419, n_D^{25} 1.5057 (picrate, m.p. 188°; double mercurichloride, B.HCl.6HgCl₂, m.p. 194°); 2:5-dimethylpyridine, b.p. 156.82—156.88°/758.1 mm., d_4^{25} 0.9261 (vacuum), n_D^{25} 1.4982 (picrate, m.p. 167°); chloroplatinate, m.p. 214°; double mercurichloride, B.HCl.6HgCl₂.H₂O, m.p. 203°. From the fraction of shale tar b.p. 170—172° were isolated 3:5-dimethylpyridine, b.p. 171.6°/760 mm., d_4^{25} 0.9385, n_D^{25} 1.5032; 2:3:6-trimethylpyridine, b.p. 172.88—172.91°/761.2 mm., d_4^{25} 0.9220 (vacuum), n_D^{25} 1.5018

(picrate, m.p. 147—148°; chloroplatinate, decomp. 220°; chloroaurate, m.p. 139°; double mercurichlorides, B.HCl.2HgCl₂.H₂O, m.p. 104°, B.HCl.6HgCl₂, m.p. 165°), and 2:4:6-trimethylpyridine. From the fraction b.p. 178—180° were isolated 3:4-dimethylpyridine, b.p. 178.82°/759 mm., d_4^{25} 0.9537 (vacuum), n_D^{25} 1.5099 [picrate, m.p. 163°; chloroplatinate, m.p. 276° (decomp.); double mercurichloride, B.HCl.3HgCl₂.2H₂O, m.p. 130.5—131°; chloroaurate, m.p. 187°], and 2-methyl-4-ethylpyridine, b.p. 179—180°, d_4^{25} 0.9130, n_D^{25} 1.4953. From the fraction b.p. 186—190° was isolated 2:3:5-trimethylpyridine, b.p. 186.75°/761.1 mm., d_4^{25} 0.9310 (vacuum), n_D^{25} 1.5057 (picrate, m.p. 184°; chloroplatinate, m.p. 212°; double mercurichloride, B.HCl.3HgCl₂, m.p. 110°), and 2:4:5-trimethylpyridine, b.p. 190.04—190.05°/766.1 mm., d_4^{25} 0.9330 (vacuum), n_D^{25} 1.5054 (picrate, m.p. 161°; chloroplatinate, m.p. 192°; double mercurichloride B.HCl.3HgCl₂.2H₂O, m.p. 112°). From the fraction b.p. 200—202° was isolated 2:3:4:6-tetramethylpyridine, b.p. 203.9°/771 mm., d_4^{25} 0.9229, n_D^{25} 1.5084 (picrate, m.p. 107°). From the fraction b.p. 187—188° was isolated 2:6-dimethyl-4-ethylpyridine, b.p. 187.5—188°/758 mm., d_4^{25} 0.9089, n_D^{25} 1.4964 (syrupey picrate; chloroplatinate, m.p. 203°), and from the fraction b.p. 189—190° 2:3-dimethyl-6(?)ethylpyridine, b.p. 190—190.5°/764 mm., d_4^{25} 0.9107, n_D^{25} 1.4970, was obtained. From the final fraction, b.p. 200—202°, a new base pyridane (probably represented by the annexed formula), b.p. 199.8°/761 mm., d_4^{25} 1.029, n_D^{25} 1.541 (picrate, m.p. 181°), was isolated. From a comparison of the various accurate b.p. data it is found that the rise in b.p. produced by a methyl group in the



α -position is about one half that produced by a methyl group in the β - or γ -positions in the pyridine ring, the b.p. of the various methylpyridines being expressed by the empirical formula $T = 115.3^\circ + 14m + 28m' + C$, where T is the b.p. of the methylpyridine, m the number of α -methyl groups, and m' the number of β - or γ -methyl groups, C is a variable correction term, being 4° for 2:3-substitution, and 8° for 3:4-substitution, these values being reduced to one half if the two side-chains are situated in the 1:4-positions.

J. W. BAKER.

Benzene for precipitation of asphaltenes from mineral oil. H. BURSTIN and J. WINKLER (Przemysl Chem., 1928, 12, 445—463).—Benzene used for the precipitation of asphaltenes should consist of saturated aliphatic hydrocarbons possessing the following properties: b.p. 65—95° (70% boiling from 65—80°), d_4^{15} 0.685—0.695, raffination number < 20, n_D^{20} < 1.3950, aniline point > 64°, and the fraction boiling from 80° to 95° should have d_4^{15} 0.700 and n_D^{20} > 1.4000.

R. TRUSZKOWSKI.

Preparation of contact substances from the solar oils of Grozni. M. D. TILITSCHIEV and A. I. DUMSKI (Neft. Choz., 1926, 11, 738—743).—Acids formed by the action of fuming sulphuric acid on solar and machine oil distillates are used as catalysts in the saponification of fats, the condensation of phenols with formaldehyde, etc. After treatment with the acid, the residual oil was extracted with aqueous alcohol; the effect of repeated extraction was studied. The acid causes resinification

and then sulphonation of the aromatic hydrocarbons. The residue consists chiefly of naphthene hydrocarbons. The higher is the density of the original oil, the greater is the yield (13–22%) of contact substance.

CHEMICAL ABSTRACTS.

Oxidation of mineral oils in presence of soluble catalysts. G. S. PETROV, A. I. DANILOVICH, and A. Y. RABINOVICH (Papers Karpov Chem. Inst., Bach Mem. Vol., 1927, 157–168).—Manganese naphthenates were most, and zinc naphthenates least, active in effecting the oxidation of mineral oils; copper and lead salts were intermediate. Manganese salts of sulpho-acids of high mol. wt. were even more active. Oils refined with sulphuric acid oxidise to about the same extent as non-refined oils; the former yield a larger amount of soluble acids, whilst the latter yield more oxy-acids. Distilled oils highly refined with fuming sulphuric acid do not oxidise in absence of catalysts even when treated with air at 150° for 40 hrs., but darken as a result of decomposition.

CHEMICAL ABSTRACTS.

Determination of the composition of petroleum. B. TARASSOV (Neft. Choz., 1928, 11, 67–70).—Unsaturated hydrocarbons are determined approximately by measuring the critical solution temperature with aniline before and after treatment with 80% sulphuric acid; on removal of the sulphuric acid with aqueous alkali, naphthenic acids are removed. Aromatic hydrocarbons can be determined by nitration; suitable formulæ for converting nitro-derivatives into hydrocarbons are given for each fraction.

CHEMICAL ABSTRACTS.

Mol. wt. of petroleum products. B. KAMINER (Neft. Choz., 1926, 11, 219–223).—The following mol. wts. were obtained ebullioscopically in benzene: crude petroleum 250–263; gasoline (b.p. up to 190°) 119–122; kerosene (b.p. 190–259°) 166–170; gas oil (259–264°) 227–231; solar oil (264–310°) 288–297; machine oil (310–320°) 411–420; cylinder oil (322–330°) 474–481; tar 711–732.

CHEMICAL ABSTRACTS.

Fifteen years of the Burton process [for cracking heavy petroleum oils]. R. E. WILSON (Ind. Eng. Chem., 1928, 20, 1099–1101).—The development of the Burton process is historically reviewed. G. CLAXTON.

Liquid-phase cracking processes. E. OWEN (Chem. Met. Eng., 1928, 35, 677–681).—A review of the development of oil-cracking processes and plant; the older Burton, Coast, and Fleming methods are being replaced by others such as those of Jenkins, Isom, and Dubbs.

R. H. GRIFFITH.

Dossor mineral oil. B. G. TUICHININ and S. N. PAVLOVA (Papers Karpov Chem. Inst., Bach Mem. Vol., 1927, 215–29).—The characteristics of the three horizontal layers of the Dossor (Caucasus) oil fields are described.

CHEMICAL ABSTRACTS.

Higher hydrocarbons from methane. H. M. STANLEY and A. W. NASH (Nature, 1928, 122, 725).—Fischer's statement (B., 1928, 844) that under suitable conditions of heating methane yields higher hydrocarbons is confirmed.

A. A. ELDRIDGE.

Higher hydrocarbons from methane. R. V. WHEELER (Nature, 1928, 122, 773).—A claim for priority (cf. Fischer, B., 1928, 844). The production of benzene

during the decomposition of ethylene is believed to be by way of ethylene and butadiene.

A. A. ELDRIDGE.

Determination of hydrocarbon vapours in air by active carbon. E. POSNER (Z. anorg. Chem., 1928, 174, 290–294).—The determination of hydrocarbon vapours by adsorption on active carbon is unaffected by carbon monoxide and methane, but acid and alkaline gases should first be removed. The preparation of the apparatus takes about 20 hrs., and the actual determination up to about 1 hr., with a rate of flow of 60 litres per hr.

H. F. GILLBE.

Cresol-naphthene soap solutions. KOGAN.—See XII. **Brewers' pitch.** KUTTER.—See XVIII.

PATENTS.

Treatment or purification of coal or other carbonaceous material. SOC. ANON. D'OUGRÉE-MARIHAYE (B.P. 295,312, 16.9.27. Fr., 9.8.27).—Low-ash coals are separated by a "float-and-sink" method in which the crushed material (0.5–10 mm.) is impregnated with liquors, e.g., solutions of calcium chloride, of successively increasing strengths up to that of the density requisite for the separation. After the separation the liquors are used in the reverse order for washing the coal, each liquor moving in a closed cycle, first wetting the coal and so becoming weaker, then washing the coal and thereby being restored to substantially the original strength.

A. B. MANNING.

Apparatus for distillation of coal and other solid fuels. R. FEIGE (B.P. 279,120, 15.10.27. Ger., 15.10.26).—A retort for the carbonisation of powdered fuel is provided with a central vertical shaft which can be rotated and to which are attached a number of flat or funnel-shaped plates. Interposed between these plates are stationary funnel-shaped surfaces attached to the outer wall of the retort. The material falling on one of the moving plates is projected outwardly in a thin layer by the centrifugal action. After passing over the edge of the plate it falls by gravity down the stationary plate on to the centre of the succeeding rotating plate, and so on through the retort. The retort is double-walled and is heated by the passage of hot gases through the annular space so formed. These gases may then pass up through the retort itself counter-current to the flow of the material. Pipes may be provided for the introduction of hydrogen, hydrocarbons, etc. to different zones of the retort if desired. The apparatus may be modified to work under pressure or under a vacuum, or to permit the material to be subjected to an electrical discharge.

A. B. MANNING.

Low-temperature carbonisation of bituminous coals etc. C. H. LANDER and J. F. SHAW (B.P. 299,201, 11.11.27).—A vertical retort for the low-temperature carbonisation of coal has the horizontal cross-section in the form of an elongated figure of which the opposite sides are parallel, and is tapered vertically in such a way that the rate of increase of the cross-sectional dimensions is greater at the lower end of the retort than at the upper. The retorts are conveniently built up of channel-shaped end-sections and plates which are interposed between them. Each complete section may be made to

engage with that immediately below it by connexions of the spigot and socket type. A. B. MANNING.

Vertical retorts and the like. C. H. LANDER and J. F. SHAW (B.P. 299,202, 11.11.27; cf. B.P. 299,201, preceding).—The lower end of a retort of the type previously described (*loc. cit.*) is not rigidly secured to the outlet duct below but rests thereon in such a manner that it is free to expand laterally. The relative dimensions of these two parts are such that when the retort is fully expanded the lower end is not larger in cross-section than the adjacent part of the outlet duct. Thus the lower end of the retort may be provided with a skirt or socket which rests within a trough or spigot on the upper end of the outlet duct. To facilitate relative motion between the two parts the surfaces in contact may be coned. A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. I. G. FARRENIN. A.-G. (B.P. 276,001, 11.8.27. Ger., 11.8.26).—Carbonaceous material is converted into valuable hydrocarbons by destructive hydrogenation in presence of catalysts containing up to 10% of silver (either free or combined), the remainder of the catalyst being boron, aluminium, silicon, titanium, vanadium, tantalum, or molybdenum, either free or combined, or cobalt compounds, or mixtures thereof. Hydrogen in excess or gases containing, or giving rise to, hydrogen are employed at pressures above 50 atm., and the reaction is carried out at above 400°.

G. CLAXTON.

Coke ovens. SIMON-CARVES, LTD., and J. H. BROWN (B.P. 298,285, 14.7.27).—In coke ovens of the cross-regenerator or other type, the air for each gas burner (or group of burners) is taken from the atmosphere at a point below the structure, the inlets having valves controlled from outside the structure and preferably connected in groups for operating by a single controlling means which may be combined with the gas-reversing gear. From the valves the air passes upwards in conduits through the regenerator sole flues.

B. M. VENABLES.

Ovens of the rotary, annular sole type. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H. (B.P. 293,449, 13.6.28. Ger., 7.7.27).—The material to be heated is spread in the thinnest possible layer on the sole, and the latter is divided into several segments each provided with charging and discharging zones. The dust-like product may be removed by a blast of gas, one for each segment, and recovered in a cyclone or other separator, one separator being common to all the segments.

B. M. VENABLES.

Adsorption media. LURGI-GES. F. WÄRMETECHNIK M.B.H. (B.P. 280,505, 9.9.27. Ger., 11.11.26).—The flue dust from combustion plants is used as an adsorption medium, after being subjected to selective separation, e.g., by centrifugal or electrical action, combined with known washing processes. A. B. MANNING.

Absorptive carbon. SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 624,277, 3.3.26).—Carbonaceous material is saturated with a mixture of sulphuric and phosphoric acids, heated at a temperature below the activation temperature, compressed, and then strongly heated to complete carbonisation. A. R. POWELL.

Active carbon. SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 626,983, 7.4.26).—Material rich in cellulose is impregnated with an activating compound prior to carbonisation. A. R. POWELL.

Treatment of coke. C. L. WAGGONER and F. B. THACHER, Assrs. to BY-PRODUCTS COKE CORP. (U.S.P. 1,685,654, 25.9.28. Appl., 9.4.28).—The porous coke, produced by high-temperature distillation and free from volatile constituents, is mixed with a moisture-absorbing alkaline-earth compound. F. G. CLARKE.

Manufacture of coke and electrodes therefrom, chiefly for use in electrometallurgy. SOC. ANON. D'OUGRÉE-MARIHAYE (B.P. 295,314, 28.10.27. Fr., 9.8.27).—Coal is purified by the process of B.P. 295,312 (B., 1929, 7) and carbonised in a strongly heated retort, or briquetted and carbonised in a current of heated nitrogen or steam, producing a partly graphitised coke containing less than 1% each of ash, sulphur, and volatile matter, suitable for electrodes. A. KEY.

Gas generators. E. C. R. MARKS. From BERNITZ FURNACE APPLIANCE CO. (B.P. 299,627, 2.12.27).—A gas generator is lined with hollow bricks the cavities of which communicate with each other in such a way as to form a continuous space between the inner and outer walls. The bricks corresponding with the lower half of the fuel bed are perforated to permit the circulation of gases between the wall space and the fuel bed. A few of the bricks in the upper zone may also be perforated, and vertical ducts may be provided for conducting air and steam to the upper section of the lining; or the upper zone of the wall space may be entirely separated from the lower and used as an air preheater.

A. B. MANNING.

Manufacture of water-gas. H. NIELSEN and B. LAING (B.P. 299,485, 28.5.27).—A water-gas plant for use with powdered fuel comprises a combustion chamber surmounted by a reaction chamber. The latter contains a suitable arrangement of refractory material to act as a heat accumulator, so that sufficient heat becomes stored therein during the "blow" to carry on the water-gas reaction for any desired length of time when the air supply is replaced by superheated steam.

A. B. MANNING.

Manufacture of carburetted water-gas. P. DVORKOVITZ (B.P. 299,482, 27.4.27).—Water-gas is enriched by the addition of oil-gas which is produced in a separate gasifier maintained at a constant temperature.

A. B. MANNING.

Manufacture of gas from [heavy] oils. C. CHILOWSKY (B.P. 293,007, 20.10.27. Fr., 27.10.26).—Processes described in B.P. 255,423, 271,899, and 271,907 (B., 1927, 805, 867) are improved so that fouling of the apparatus and catalyst is prevented.

C. B. MARSON.

Purification of fuel and other gases. KOPPERS Co., Assees. of (A) D. L. JACOBSON and H. A. GOLLMAR, (B) H. A. GOLLMAR (B.P. 280,165 and 286,633, [A] 14.7. and [B] 16.7.27. U.S., [A] 5.11.26, [B] 7.3.27).—(A) Hydrogen sulphide and other acidic constituents are removed from fuel gases by washing with an alkaline solution of a compound of arsenic, tin, or other metal of

which the sulphide is soluble in an alkali or alkali sulphide solution. The solution is reactivated by aeration, and, after being freed from the sulphur which is liberated, is recirculated. A solution of sodium arsenite containing the equivalent of about 0.5% As_2O_3 may be used, but is preferably sulphided and aerated once before use in the plant. (B) The strength of the wash liquid is maintained by the addition, at a uniform rate, of a mixture, preferably in solution, of the alkali and arsenic (or other metal) compound, containing 1—2 atoms of alkali metal to each atom of arsenic. A. B. MANNING.

Apparatus for removing tar from gases evolved in the dry distillation of wood. J. R. RONGIER (F.P. 624,127, 5.11.26).—The apparatus comprises one or more condensers terminating in bell-shaped vessels which dip into annular troughs in which the tar is collected and which are provided internally with horizontal perforated plates containing openings for the removal of uncondensed vapours. A. R. POWELL.

Manufacture of ammonium sulphate from gas-works' liquor or the like. S. ROBSON (B.P. 299,934, 8.11.28).—Crude ammonia liquors are distilled and the resulting distillate is brought to a concentration of 7—12% NH_3 so that when maintained at 50° a current of air and/or inert gases passing through it volatilises somewhat more than twice as much ammonia, by vol., as water vapour. These gases are then brought into contact with sulphur gases (e.g., sulphur trioxide) as described in B.P. 289,950 (B., 1928, 523) to produce ammonium sulphate directly in one operation.

R. J. W. LE FÈVRE.

Recovery of calcium ferrocyanide from gas-purification residues. RÜTGERSWERKE A.-G., and L. KAHL (Dan. P. 34,820, 27.8.24. Ger., 2.10.23).—The residues are extracted with aqueous calcium hydroxide solution, and the solution, after filtration, is worked up to Prussian-blue by the usual process. L. A. COLES.

Absorbent filling material for use in acetylene storage containers. M. ROHRSCHEIDER (B.P. 299,681, 24.4.28).—Finely-divided or shredded leather, which has been freed from acids, substances soluble in acetone, and water, is used as a filler for acetylene containers. The leather may be admixed with a powdered or granular heat-resistant material, e.g., pumice stone or kieselguhr.

A. B. MANNING.

Manufacture of porous masses for storing explosive gases. GAS ACCUMULATOR Co. (UNITED KINGDOM), LTD., ASSEES. of AUTOGEN GASACCUMULATOR A.-G. (B.P. 281,718, 5.12.27. Ger., 4.12.26).—Inorganic porous substances to be used as filling materials for containers for storing explosive gases are impregnated with a finely-divided heat-resisting material (e.g., kieselguhr), which fills up all dangerous voids therein.

A. B. MANNING.

Treatment of gases or vapours for the recovery or extraction of hydrocarbons therefrom. P. M. SALERNI (B.P. 299,481, 26.4.27).—A number of long, narrow, spiral passages are arranged in parallel and are attached to an axis which can be rotated. The gas and oil, or other absorbing medium, are passed together along the passages, the centrifugal force maintaining the oil in a layer against the surface of the passages, along

which the gases travel in a state of turbulence. A central rotor, rotating in a direction opposite to that of the spiral device, and comprising a series of vanes on a central shaft, impels the gas and oil into the inner openings of the spiral passages. The oil is fed to distributing channels through nozzles extending radially from the shaft. The gases after emerging from the passages are whirled round by suitably placed vanes in order to free them from oil spray. A. B. MANNING.

Treatment of shale and the like. W. H. HAMPTON (U.S.P. 1,687,763, 16.10.28. Appl., 11.12.19).—Finely-divided bituminous solids are digested with a heavy mineral oil at 260—370°. The evolved vapours are mixed with steam, subjected to cracking at higher temperatures, and the products fractionally condensed. The residual digestion mixture is treated with a lighter oil and separated from any solids. From the residual oil a heavy fraction is obtained which, mixed with a heavy fraction from the cracking process, is used for the digestion of further material. H. S. GARLICK.

Extracting the oily bases from crude shale oil. D. T. DAY (U.S.P. 1,685,315, 25.9.28. Appl., 11.12.24).—The crude shale oil is treated with a dilute solution of an inorganic acid, and the acid layer containing the bases is separated and treated with alkali. The liberated bases are separated from the aqueous solution, redissolved in dilute acid, re-liberated by means of alkali, extracted by agitation with a hydrocarbon solvent, and finally removed from this solvent by extraction with an inorganic acid. C. O. HARVEY.

Production of low-boiling hydrocarbons. W. GESSMAN and E. W. SHALDERS (B.P. 299,820, 30.6.27).—A mixture of unsaturated hydrocarbons, preferably acetylene, with a gas containing carbon monoxide and hydrogen is passed at normal pressure and without heating over a metal catalyst which is irradiated at intervals with ultra-violet light. The catalyst consists of pure, powdered, electrolytic copper mixed with powdered tungsten and powdered pumice, which mixture is treated with a dilute (10%) solution of caustic alkali and then dried thoroughly. G. CLAXTON.

Cracking of [petroleum] oils. J. G. ALTHER, ASST. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,685,476, 25.9.28. Appl., 20.8.20. Renewed 9.6.26).—By using a series of heating coils of continuously decreasing diameter, the velocity of the oil under treatment is increased as the temperature of the heating chamber diminishes. The smallest coil discharges the oil into a vaporising chamber. R. BRIGHTMAN.

Distillation of petroleum oils. J. E. BELL, ASST. to SINCLAIR REFINING Co. (U.S.P. 1,683,151, 4.9.28. Appl., 1.4.24).—The feed is introduced at or near the top of a fractionating tower having a helical passage and flows continuously down the helix, meeting the vapours from the still. The heavier reflux and the unvaporised portion of the feed enter the still from the foot of the tower, the lighter vapours being collected. Effective heat interchange and refluxing of the oil render close fractionation possible. R. BRIGHTMAN.

Decolorising, clarifying, and purifying petroleum oils. J. L. KAUFFMAN and I. A. CLARK (U.S.P.

1,684,035, 11.9.28. Appl., 28.11.24).—The oil is agitated with a finely-divided filtering material (*e.g.*, untreated clay) by means of superheated steam, and, after heating the mixture to a temperature not exceeding the b.p. of the oil and cooling, the filtering material is separated therefrom. C. O. HARVEY.

Treating slack wax. B. L. SOUTHER and W. A. GRUSE, Assrs. to GULF REFINING CO. (U.S.P. 1,685,008 and 1,685,058, 18.9.28. Appl., [A] 17.2.26, [B] 30.8.26).—(A) The magma of solid slack wax containing oil is finely comminuted until it is fluid and the liquid and solid ingredients are separated mechanically. (B) A liquid having preferential solvent action on the oil is added to the comminuted material to facilitate the mechanical separation. F. G. CLARKE.

Refining of [petroleum] oils. S. TILMSTRA, Assr. to ROXANA PETROLEUM CORP. (U.S.P. 1,684,159, 11.9.28. Appl., 6.12.26).—Oil which has been treated with sodium plumbite solution is freed from lead by washing with sodium polysulphide solution. C. HOLLINS.

Recovery of vapours [waste petroleum gases]. R. T. OSBORN, Assr. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,685,501, 25.9.28. Appl., 11.7.22).—Gases or vapours which are themselves uncondensable at atmospheric temperature and pressure are passed into vapours from a still operating below cracking temperature and pressure, whereby a mixture is obtained which may be substantially completely condensed. C. O. HARVEY.

Atomisable mobile fuel product. A. W. BURWELL, Assr. to ALOX CHEM. CORP. (U.S.P. 1,684,125, 11.9.26. Appl., 2.3.27).—A fuel oil is oxidised with air at 120–140° and under 250 lb./in.² in presence of 0.1% of manganese stearate; water-soluble products are removed, and the remainder, saponified with sodium hydroxide, is added to 50 pts. of fuel oil and water is driven off, leaving a colloidal solution which is capable of holding in suspension an equal weight of powdered coal (100–200-mesh). C. HOLLINS.

[Stream divider for] oil [distillation] apparatus. C. F. TEARS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,688,839, 23.10.28. Appl., 18.6.23. Renewed 28.5.28).—The apparatus comprises an enlarged container with an oil inlet and with one ordinary outlet and several relatively small manifolded outlets, all the outlets terminating as standpipes in the container with their upper ends at the same level. L. A. COLES.

Manufacture of alkyl halides, glycols, and alcohols from cracked gases of hydrocarbon oils. L. BLANCHET (F.P. 610,431, 1.5.25).—Additional to B.P. 251,652 (B., 1927, 836) is the following: A cracked gas, after washing with oils to remove pentane and amylene, b.p. 30–37°, is cooled to 5° with slight pressure, and the residual gas is freed from butane and butylenes by further cooling under higher pressure; propane and propylene are similarly removed, or the propylene may be absorbed in sulphuric acid. Ethylene (and propylene, if desired) is converted by hypochlorous acid into chlorohydrin, which is either distilled in a vacuum or hydrolysed to glycol; the remaining gas is converted by chlorine into alkyl chlorides and hydrolysed to alcohols. The liquefied fractions, pentane

and amylene, butane and butylene, are treated with hydrogen chloride and chlorine to give amyl chlorides and butyl chlorides, from which the acetates and finally the alcohols are obtained. C. HOLLINS.

Apparatus for the distillation of coal and similar carbonaceous substances. C. H. PARKER (U.S.P. 1,689,152, 23.10.28. Appl., 27.1.28. U.K., 25.9.26).—See B.P. 287,830; B., 1928, 394.

Apparatus for distilling solid combustible carbonaceous material. O. HUBMANN (U.S.P. 1,690,933, 6.11.28. Appl., 8.10.25. Ger., 4.11.24).—See B.P. 242,618; B., 1926, 971.

Formation of [reconstructed] carbonaceous fuel. W. B. RULON (B.P. 299,611, 27.10.27).—See U.S.P. 1,656,364; B., 1928, 219.

Gas purification. F. W. SPERR, JUN., Assr. to KOPPERS CO. (U.S.P. 1,690,437—8, 6.11.28. Appl., 6.6.24. Renewed 23.1.28).—See B.P. 235,126—7; B., 1925, 662.

Cracking of hydrocarbons. C. P. TOLMAN (B.P. 299,957, 17.9.27).—See U.S.P. 1,643,036; B., 1927, 836.

Apparatus for burning pulverised fuel. GASIFIED FUEL, LTD., H. E. HAZLEHURST, and O. MARGETSON (B.P. 299,930, 11.4.27).

Reducing excess gas pressure in the lower part of freshly charged vertical chambers or retorts. PINTSCH & DR. OTTO GES.M.B.H. (B.P. 288,337, 5.4.28. Ger., 7.4.27).

Burners for gas firing. R. EICKWORTH (B.P. 275,654, 5.8.27. Ger., 6.8.26).

Combined filter and separator (B.P. 299,648). Treatment of gases with liquids (B.P. 299,075). Gas washers (B.P. 297,765).—See I. Hydrogen from gaseous mixtures (B.P. 299,558).—See VII. Preventing explosions (U.S.P. 1,686,051).—See XI.

III.—ORGANIC INTERMEDIATES.

Separation of alcohols and phenols from oil mixtures. H. SCHMIDT (Chem.-Ztg., 1928, 52, 898).—The oil is heated at 80–100° under slightly reduced pressure with the theoretical quantity of boric acid to produce the borate B(OR)₃. When no further water distils over the temperature is raised and the unchanged oil distilled under low pressure. The residual borate is then hydrolysed with sodium hydroxide and the liberated alcohol or phenol is recovered by distillation in steam. The method affords a rapid means of separating primary, secondary, and tertiary alcohols from admixture with one another; sufficient boric acid is first added to combine with the primary alcohol, and the unchanged mixture of secondary and tertiary alcohols is distilled off under low pressure, treated with just sufficient boric acid to combine with the secondary alcohol, and the process repeated. A. R. POWELL.

Indene and styrene. BROWN.—See II.

PATENTS.

Production of phosphoric esters of aliphatic alcohols. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 300,044, 12.1.28).—Phosphoryl chloride

vapours are passed, preferably at 25—40 mm. pressure, into an aliphatic alcohol at 50°, and the mixture is boiled under reflux for about 1 hr. so that hydrogen chloride is removed as formed. Triethyl, tri-*n*-butyl (b.p. 135°/5 mm.), tri-*isobutyl* (b.p. 112°/4—5 mm.), and triamyl (b.p. 143—144°/2.5 mm.) phosphates are described.

C. HOLLINS.

Manufacture of concentrated acetic acid from calcium acetate. L. H. FAUQUE (F.P. 612,595, 10.3.26).—Crude calcium acetate paste (30% water) is saturated in a stoneware-packed tower with hydrogen chloride, warmed until liquid, and transferred to two containers, where dry calcium acetate equivalent to the hydrochloric acid is added, and the mixture is distilled, giving 70—80% acetic acid and a residue of tar and saturated calcium chloride solution.

C. HOLLINS.

Production of pure α -hydroxycarboxylic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,040, 28.12.27).—Cyanohydrins are hydrolysed by a solution of hydrogen chloride in an inert solvent (dioxan, chloroform, etc.) together with sufficient water for the reaction. Preferably the cyanohydrin is prepared from hydrogen cyanide, the aldehyde or ketone, and alkali, in dioxan (or a mixture of dioxan and chloroform), and gaseous hydrogen chloride is then added to the warm solution. The preparation of lactic, mandelic, and α -hydroxybutyric acids is described.

C. HOLLINS.

Purification of lactic acid. J. A. VON WÜLFING (G.P. 446,865, 4.2.25).—Crude concentrated lactic acid is mixed with active carbon, silica gel, etc. to form a loose powder which on extraction with ether gives lactic acid free from coloured and odorous impurities and from higher fatty acids.

C. HOLLINS.

Reduction of organic compounds. L. ZECHMEISTER and P. ROM (G.P. 446,867, 26.11.25).—Organic compounds in methyl-alcoholic solution or suspension are reduced with magnesium and aqueous ammonium chloride without addition of an activator (cf. A., 1926, 720).

C. HOLLINS.

Manufacture of primary amines. FABR. DE PROD. CHIM. ORGANIQUE DE LAIRE, Assecs. of G. MIGNONAC (B.P. 282,083, 9.12.27. Fr., 9.12.26).—Oximes or nitriles are hydrogenated in presence of nickel with the addition of ammonia, which largely prevents formation of secondary and tertiary amines. Acetonitrile, ammonia, and hydrogen, passed over reduced nickel at 180°, give ethylamine in 90% yield; benzylamine is similarly obtained from benzonitrile, and isopropylamine from acetoxime. Phenylacetoneitrile in alcoholic ammonia containing reduced nickel gives 85% of β -phenylethylamine when shaken with hydrogen.

C. HOLLINS.

Manufacture of thiuram monosulphides. A. J. MAXIMOV, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,682,920, 4.9.28. Appl., 18.9.26).—Thiuram monosulphides (e.g., tetramethylthiuram sulphide, m.p. 120—130°) are obtained in good yield by stirring the corresponding disulphide in aqueous suspension with sodium cyanide.

R. BRIGHTMAN.

Manufacture of indoles. I. G. FARBENIND. A.-G., Assecs. of O. NICODEMUS (G.P. 446,544, 26.3.25).—Suitable alkylated arylamines are passed with oxygen or a

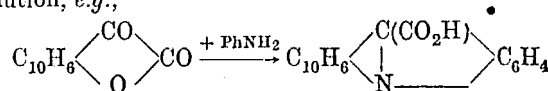
nitro-compound over silica gel or highly active carbon, especially the phosphoric acid-carbon of G.P. 407,406, to which may be added oxygen carriers such as compounds of cerium, vanadium, manganese, or copper, at 300—400°. Diethylaniline yields 1-ethylindole, b.p. 247°, 2-methyl-1-ethylindole, b.p. 265°, and di-indyls; dimethyl-*o*-toluidine gives 15% of 1-methylindole, b.p. 240°, and traces of higher indoles.

C. HOLLINS.

Preparation of derivatives of isatoic anhydride. BADISCHE ANILIN- & SODA-FABR. (F.P. 603,970, 5.6.25. Ger., 28.6.24. Cf. G.P. 433,147; B., 1927, 215).—The following is additional to the G.P.: The isatoic anhydrides from *o*-aminobenzoic acids or their *N*-monoalkyl or -monoaryl derivatives (other than anthranilic acid) yield compounds with cellulose, starch, sugar, etc. which couple with diazo compounds. Isatoic anhydrides made by the action of carbonyl chloride or chloroformic esters on 2-amino-3-naphthoic acid and on 5-nitro-anthranilic acid are described.

C. HOLLINS.

Preparation of naphthacridine-*ms*-carboxylic acids and derivatives. I. G. FARBENIND. A.-G., Assecs. of K. FRIES (G.P. 446,543, 5.7.26).—A β -naphthacoumarandione is heated with a primary arylamine in acid solution, e.g.,



The products are decarboxylated by heating above their m.p. The following are described: pheno- β -naphthacridine and its 12-carboxylic acid, m.p. 284°, from aniline (cf. Stolle and others, A., 1923, i, 1125); 10-methoxypheno- β -naphthacridine, m.p. 157°, and its 12-carboxylic acid, m.p. 236°, from *p*-anisidine; 10-acetamidopheno- β -naphthacridine-12-carboxylic acid, m.p. 292°, from *p*-aminoacetanilide, which is converted by heat and hydrolysis into 10-aminophenonaphthacridine, m.p. 238°; $\beta\beta$ -dinaphthacridine-14-carboxylic acid, m.p. 240°, and $\beta\beta$ -dinaphthacridine, m.p. 216°, from β -naphthylamine.

C. HOLLINS.

Preparation of anhydrides of organic acids. F. DOERINCKEL and M. SCHLIMMANN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,691,647, 13.11.28. Appl., 21.1.27. Ger., 28.1.26).—See B.P. 289,959; B., 1928, 515.

Production of gels. Production of esters from organic halides. Hydrolysis of ester-forming compounds. E. E. AYRES, JUN., and E. H. HAABSTAD, Assrs. to B.A.S. CO. (U.S.P. 1,691,424—6, 13.11.28. Appl., [A, B, C] 21.10.24. Renewed [B] 20.4.27).—See B.P. 241,889; B., 1927, 859.

Production of anthraquinonesulphonic acids. J. THOMAS, Assr. to SCOTTISH DYES, LTD. (U.S.P. 1,691,679, 13.11.28. Appl., 10.1.23. U.K., 18.1.22).—See B.P. 200,851; B., 1923, 922 A.

Treatment of compounds by gaseous reagents (B.P. 274,846).—See I. Reduction of dyes etc. (B.P. 299,899).—See IV. Formic and acetic acids (G.P. 452,138).—See VII. Oxidation of aldoses (B.P. 293,322).—See XVII. Chloral (F.P. 612,396). Phenol ethers (G.P. 446,606).—See XX.

IV.—DYESTUFFS.

PATENTS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G., Assocs. of A. LÜTTRINGHAUS, P. NAWIASKY, and A. EHRHARDT (G.P. 451,122, 6.9.22).—Dihydroxydibenzanthrone is treated with the toluenesulphonic ester of glycol, glycerol, ethylene chlorohydrin, or other polyhydric alcohols in which the hydroxyl groups have been partly replaced by halogen or an arylsulphonic acid residue. Blue to green-blue vat dyes result. C. HOLLINS.

Manufacture of vat dyes of the anthracene series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,972, 30.9.27).—The glyoxylidenebisanthrones of B.P. 296,817 (B., 1928, 848) are fused with alkali to give fast bordeaux to violet vat dyes. C. HOLLINS.

Manufacture of stable diazo-salt preparations. I. G. FARBENIND. A.-G. (B.P. 275,245, 27.7.27. Ger., 2.8.26. Cf. B.P. 273,352; B., 1927, 647).—The fission of diazonium hydrogen 1:5-naphthalenedisulphonates into normal salt and free acid in solution is prevented by admixture of 1:5-naphthalenedisulphonic acid. Other diazonium sulphonates are similarly stabilised.

C. HOLLINS.

Manufacture of insoluble azo dyes for acetate silk. I. G. FARBENIND. A.-G., Assocs. of K. HOLZACH and R. METZGER (G.P. 450,920, 24.1.26).—The dye, *m*-aminobenzaldehyde \rightarrow 2:4-dihydroxyquinoline, dyes acetate silk greenish-yellow, becoming violet-brown on treatment with phenylhydrazine. *m*-Aminobenzaldehyde \rightarrow phenylmethylpyrazolone gives fast yellow shades. C. HOLLINS.

Manufacture of reduction products of vat dyes and other reducible organic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,899, 4.5.27).—Dry leuco-compounds suitable for conversion into sulphuric esters are prepared by adding the vat dye and 98% formic acid to a solution of sulphur dioxide in pyridine, dimethylaniline, or other organic base. Reduction takes place at 25–70°, according to the vat dye used; benzil, isatin, etc. may also be reduced. The soluble leuco-ester may be prepared directly from the leuco-compound without isolation.

C. HOLLINS.

Aminodiarylsulphonepyrazolone azo dyes. H. SCHWEITZER and W. NEELMEIER, Asssts. to GRASELLI DYESTUFF CORP. (U.S.P. 1,685,071, 18.9.28. Appl., 5.1.26. Ger., 8.1.25).—See B.P. 245,765; B., 1926, 734).

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Influence of acids and alkalis on the formation of mucilage from cellulose. ANON. (Papier-Fabr., 1928, 26, 722–728).—Mineral acids and alkalis assist mucilage formation in the hollander and reduce considerably the time necessary to reach a given degree of grinding, but the properties (strength etc.) of the papers obtained are so adversely affected that the use of these assistants cannot be considered. More satisfactory results are obtained by the use of organic acids (e.g., acetic acid) or of salts such as sodium nitrite, which accelerate mucilage formation during grinding without causing a diminution

in the strength of the cellulose. Their use results in a reduction of the time of grinding by 30% in the case of the knife hollander and 20% in that of the stone hollander for the same degree of grinding. When no such additions are made the time required to give a degree of grinding of 90° in the stone hollander is half that required for the knife hollander. The cost of such accelerating agents increases the cost of production of the cellulose in spite of reductions due to the decreased time necessary, and the cheapest way of obtaining rapid mucilage formation is still by use of the stone grinder. B. P. RIDGE.

Wood. IV. **Chemical composition of pulp woods from North Korea.** Y. UYEDA and T. MORITA (J. Cellulose Inst., Tokyo, 1928, 4, 233–234).—Analyses are given of “Ezomatsu” (*Picea jezoensis*), “Tōshirabe” (*Abies nephrolepsis*), and “Chōsenmomi” (*Abies holophylla*). These woods have a total cellulose content (by chlorination) of 50–51% on the weight of air-dry wood (10% moisture), 90% of which is α -cellulose. D. J. NORMAN.

Relationship between amphoteric properties and purity of cellulose and its derivatives. K. KANAMARU (J. Cellulose Inst., Tokyo, 1928, 4, 259–284).—The amphoteric character of cellulose is not a property of the cellulose itself but is due to the presence of impurities, among the more important of which are degraded celluloses such as oxycellulose and hydrocellulose, and traces of alkali loosely combined in the double-layer state with the cellulose. D. J. NORMAN.

Viscose. XVIII. **Spinning.** VIII. G. KITA, R. TOMIHISA, and J. ONOHARA. IX. G. KITA, J. ONOHARA, and K. MASUI (J. Cellulose Inst., Tokyo, 1928, 4, 235–237, 237–241).—VIII. Results are given showing that by maturing alkali-cellulose in hydrogen instead of air the viscose prepared therefrom shows a higher viscosity, is more easily spun, and is capable, after suitable ripening, of giving stronger threads (2 g. per denier). Attempts to produce viscose threads of equal strength by shortening the time of maturing in air to 6 hrs. at 25° were not successful.

IX. The time of maturing of the alkali-cellulose for the production of the strongest viscose threads depends on the duration of the steeping operation. Thus, with a steeping time of 3, 7, 14 days (at 7–11° in each case), the optimum maturing time of the alkali-cellulose at 25° was, respectively, 3, 12, and 4 hrs. Under these conditions it is possible to prepare viscose threads of as high strength as is shown by viscose prepared from alkali-cellulose matured in an atmosphere of hydrogen.

D. J. NORMAN.

Chemistry of the cellulose determination. C. E. PETERSON and M. W. BRAY (Ind. Eng. Chem., 1928, 20, 1210–1213).—A study of the changes in chemical composition of spruce wood during the Cross and Bevan chlorination process has been made, and the following constituents have been determined in the chlorinated residues of 12 samples: cellulose, total pentosans and pentosans in the cellulose, methoxyl content, and lignin. The reaction involved follows the same general course as those taken by the reactions occurring with the ordinary pulping reagents, but higher yields and greater delignification result. In spruce wood, lignin is

not completely removed even after a number of protracted chlorinations, and some of the pentosans in the cellulose are removed during the first and each succeeding chlorination, whilst the pentosans not in the cellulose are destroyed very rapidly during the process of cellulose isolation. For maximum reactivity the sample of spruce to be chlorinated should contain a quantity of water at least equal to its own weight evenly distributed through the wood. Since the pentosan content of the cellulose does not increase in the various steps of the chlorination process, the results indicate that cellulose is not decomposed into substances yielding furfuraldehyde during the process of its isolation (cf. A., 1923, ii, 441).

E. H. SHARPLES.

Copper and silver numbers as factors for the evaluation of cellulose products. J. RINSE (Ind. Eng. Chem., 1928, 20, 1228—1230).—The determination of copper numbers is reviewed and modifications of existing methods are suggested to improve the accuracy of, and diminish the time required for, the analysis. Braidy's method gives relative values, but it has no high degree of accuracy, and both this and the Fehling's solution procedure fail to give conclusive results. The copper numbers may differ largely from the true reducing figures, as the cellulose itself is attacked by the caustic solution and the time of boiling is too short for complete oxidation of the original impurities. Götze's method for the determination of silver numbers is modified, and the results show that reaction is nearly complete after 24 hrs., and that the values are much nearer the true value than are the copper numbers. The method is simpler and standardisation is less necessary.

E. H. SHARPLES.

Determination of the sulphur of viscose. T. VITALE and T. MARESCA (Annali Chim. Appl., 1928, 18, 461—465).—The determination is made in a Mahler or Parr bomb, absorbent carbon of known sulphur content being used. One fourth of the total amount of sodium peroxide is mixed with 1 g. of potassium chlorate, 0.2 g. of benzoic acid, and 0.25 g. of the animal charcoal. On to this layer the viscose (0.4—0.5 g.) is allowed to fall in separate drops from a Lunge-Rey oleum pipette, the next layer consisting of 0.25 g. of charcoal, and the final one of the remaining three fourths of the sodium peroxide. The subsequent operations are as usual. The barium sulphate may be weighed or determined nephelometrically by means of Parr's photometer, the error in the latter case being $\pm 3\%$. An accuracy of 99% may be attained by oxidation with alkaline hydrogen peroxide solution (cf. Petersen, A., 1903, ii, 690).

T. H. POPE.

Effect on rosin sizing of heating the moist paper. E. OMAN (Papier-Fabr., 1928, 26, 705—707).

—If rosin-sized paper of high moisture content (e.g., at least 50%) be heated to a certain temperature (e.g., about 75°), the size is adversely affected to a less or greater degree, and may even completely disappear. The extent of this deterioration of the size is dependent on the p_H value of the contained water, being least for the range p_H 4—5 and increasing with both increase and decrease of the p_H outside this range. The presence of alum in the pulp water does not prevent this deteriora-

tion since the p_H of the latter is the deciding factor. The above facts are put forward to explain why Stöckigt and Klingner obtained unsized paper in some of their sizing experiments.

B. P. RIDGE.

Fat from silk cocoons. JELAKOV.—See XII.

PATENTS.

Producing fibre and yarn from unretted flax plants. M. WADDELL and H. C. WATSON (B.P. 299,519, 29.7.27).—After drying the flax stalks and separating the gummy constituents, woody parts, and seeds, the fibres are tightly twisted in the form of a rove on an open reel, where they are retted by bacteria and/or chemical action, washed to remove impurities, and finally spun into yarn.

F. R. ENNOS.

Extraction of cellulose or paper pulp from fibrous vegetable matter containing the same. (Sir) G. C. GODFREY, E. C. BENTHALL, E. S. TARLTON, H. F. WHEELER (BIRD & Co.), G. L. SCOTT, and E. SPENCER (B.P. 283,910, 5.7.27. India, 20.1.27).—A semi-continuous process working on the reverse-washing principle is described for the alkaline digestion of fibrous vegetable material, e.g., bamboo, grasses, etc., according to which the raw material is subjected to successive treatments at increasing temperature, pressure, and concentration of liquor until digestion is complete. The partly used liquor from each stage of the process is blown over into the next but one digester in operative sequence working at a lower pressure, the total volume of liquor and the concentration of active alkali being at the same time adjusted, if necessary, by the addition of wash liquor from a completed cook. The digesters are preferably arranged in a double-ring system. (Cf. B.P. 291,064—5; B., 1928, 925.)

D. J. NORMAN.

Preparing and separating the cellulose material, suitable for paper-manufacturing, from plant raw material with pith fibre. H. KUMAGAWA and K. SHIMOMURA (B.P. 299,740, 30.6.27).—The plant raw material is beaten in a stream of water in a beater or hollander; the water-soluble materials dissolve while the short pithy fibres float on the surface of the stream and the long thin ones, suitable for paper-making, remain suspended and are removed by a suitable device.

F. R. ENNOS.

Treatment of cellulose. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,512, 28.7.27).—A preliminary treatment of cellulose in the manufacture of cellulose acetate consists in passing it in sheet form through glacial acetic acid.

F. R. ENNOS.

Cellulose composition. O. DROSSBACH and O. JORDAN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,677,753, 17.7.28. Appl., 2.7.26. Ger., 24.7.25).—Aliphatic esters of maleic or fumaric acid are solvents and plasticisers for cellulose nitrate, acetate, or ethers.

C. HOLLINS.

Manufacture of paper and compositions therefor. H. E. POTTS. From F. W. ATACK (B.P. 299,817, 19.4.27).—Liquid emulsions which dilute immediately with water without breaking are obtained by emulsifying a grease solvent, e.g., a chlorinated hydrocarbon, with water by means of Turkey-red oil, optionally with addition of a

small proportion of fatty acid or of ammonia, and stabilising the emulsion with a colloidal silicate, *e.g.*, bentonite. The product is suitable, after dilution, as a detergent in laundering, carpet cleaning, etc., or to remove pitch from the wire and felts in papermaking.

D. J. NORMAN.

Manufacture of paper. G. A. MOURLAQUE (B.P. 299,965, 24.9.27).—The apparatus used consists of a single boiler arrangement having an interior hearth and lixiviator. Vegetable matter is soaked in water, subjected to the action of ozone, heated under pressure with a solution of potash obtained from the cinders of the fuel used in the hearth and of lime prepared by heating limestone therein, and finally bleached, all operations being performed in the lixiviator. After removal of the pulp, the solids in the residuary liquor are recovered by evaporation and calcination in the hearth of the apparatus.

F. R. ENNOS.

Strengthening and rendering impermeable paper or similar materials. L. R. MECRE (B.P. 275,617, 29.7.27. Fr., 4.8.26).—After a preliminary immersion in alkali the paper is impregnated first with an aqueous solution containing two or more of the following: salep, gum tragacanth, alga gum, to which alum or resin may be added, and then with a solution of borax, ammonium sulphate or carbonate, or boric acid. Colouring or other substances are added during the process, if desired, and the gum-like materials are finally rendered insoluble in the usual way.

F. R. ENNOS.

Production of stencil sheets for use in duplicating. A. DE WAELE (B.P. 298,705, 22.7.27).—In the manufacture of stencil sheets by coating Yoshino paper with gelatinising organic colloids, *e.g.*, nitrocellulose, the use of large quantities of organic solvents is avoided by applying the coating in the form of an aqueous emulsion. To prepare the emulsion, collodion cotton, in admixture with softening agents, *e.g.*, castor oil, is treated with sufficient amyl acetate to give a stiff jelly, and this is beaten in a colloid mill with water containing about 10% on the weight of the nitrocellulose of gelatin or agar. Emulsifying agents such as sulphonated sperm oil or ammonium salts of ricinoleic acid may be used, and bentonite may be added to stabilise the emulsion.

D. J. NORMAN.

Treatment of fibrous material [paper]. A. LUTZ, Assr. to R. C. LEE (U.S.P. 1,682,390, 28.8.28. Appl., 29.3.21. Renewed 26.5.26. Ger., 2.7.15).—In the sizing of paper, cardboard, etc. a size containing sodium hydroxide or lactic acid is caused to penetrate the material completely at 40°; a hardening treatment with hydroxymethylformamide follows, and the material is wound in roll form while hardening proceeds.

C. HOLLINS.

De-inking paper. A. R. LUKENS, Assr. to RICHARDSON Co. (U.S.P. 1,680,949, 14.8.28. Appl., 22.6.25).—The addition of an alkali sulphonate, after or during the treatment with alkali, shortens the time required to wash the pulp free from alkali and ink constituents.

F. G. CLARKE.

[Patterned woven] fabric making. A. BODMER, Assr. to HEBERLEIN & Co. A.-G. (U.S.P. 1,688,798—9, 23.10.28. Appl., [A] 27.11.26, [B] 14.7.28. Ger., [A, B] 5.12.25).—See B.P. 262,477; B., 1927, 361.

Sulphonated cellulose derivatives and their manufacture. G. KRÄNZLEIN, A. VOSS, and F. BRUNNTRÄGER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,682,382, 28.8.28. Appl., 25.7.27. Ger., 10.9.26).—See B.P. 277,317; B., 1928, 853.

Manufacture of cellulose xanthate. P. MORO (U.S.P. 1,689,958, 30.10.28. Appl., 18.3.26. Fr., 31.3.25).—See F.P. 597,336; B., 1926, 401.

Spinning of artificial silk. II. SCHMIDT and E. HUBERT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,689,895, 30.10.28. Appl., 4.4.27. Ger., 11.6.26).—See B.P. 289,942; B., 1928, 520.

Spinning of artificial [silk] filaments. R. SAJITZ and F. POSPIECH, Assrs. to CHEM. FABR. POTT & Co. (U.S.P. 1,689,894, 30.10.28. Appl., 14.6.26. Ger., 17.6.25).—See B.P. 280,608; B., 1928, 85.

Manufacture of artificial silk, artificial horse-hair, etc. [from cellulose derivatives]. H. DREYFUS (U.S.P. 1,688,531, 23.10.28. Appl., 28.6.23. U.K., 11.8.22).—See B.P. 210,108; B., 1924, 251.

[Spinning] apparatus for manufacture of artificial threads or filaments. BRIT. CELANESE, LTD., and J. BOWER (B.P. 299,405, 26.7.27).

Manufacture of artificial textile fibres [possessing wave or curl]. W. SEVER, JUN., and J. B. SPEAKMAN (B.P. 299,291, 3.4.28).

Treatment [manufacture of ribbons or webs from pieces] of fabrics made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 293,858, 15.7.27. U.S., 15.6.28).

Machine for making transparent paper or sheet cellulose from aqueous cellulose solutions. C. RUZICKA (B.P. 300,477, 15.5.28).

Treatment of gut etc. (B.P. 299,535).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyeing of fibrous materials. J. NÜSSLEIN, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,684,881, 18.9.28. Appl., 23.11.26. Ger., 7.12.25).—Aromatic amines for the production on the fibre of oxidation dyes are applied as an aqueous dispersion, *e.g.*, with Turkey-red oil, or sodium diisopropyl-naphthalenesulphonate or *N*-dibenzylaniline-sulphonate, or with organic solvents. R. BRIGHTMAN.

Production of fast tints on vegetable fibres [dyeing cotton with azo dyes containing chromium]. Soc. CHEM. IND. IN BASLE (B.P. 271,897, 27.5.27. Switz., 27.5.26).—Dyeings fast to washing are obtained on cotton directly by use of the chromium compounds of dyes made by diazotising an *o*-aminophenol (other than an amino-salicylic acid) or an anthranilic acid and coupling with the usual components. The chromium compound of the dye, 4-chloro-5-nitro-2-aminophenol \rightarrow J-acid, gives bluish-green shades; 41 other couplings are mentioned.

C. HOLLINS.

Production of effect threads. I. G. FARBENIND. A.-G. (F.P. 623,704, 26.10.26. Ger., 13. and 25.11.25).—Cotton effect threads in woollen material are left undyed in an acid dye bath to which alkylated, arylated, aralkylated, or cycloalkylated aromatic (or partly hydrogenated

aromatic) sulphonic acids, particularly benzylnaphthalenesulphonic acids, have been added. C. HOLLINS.

Dyeing of textile goods [with vat dyes]. I. G. FARBENIND. A.-G. (B.P. 272,941, 16.6.27. Ger., 18.6.26. Addn. to B.P. 270,307).—In the process of the prior patent a boiling hyposulphite vat liquor to which formaldehyde or acetone has been added is employed. More level dyeing results, and the spent dye liquor may be brought up to strength with fresh dye and used again. C. HOLLINS.

Dyeing by means of leuco-ester compounds of dyes of the indigo series and other vat dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,743, 26.7.27).—The streakiness resulting from drying the dyed goods on a dipping frame before development is avoided if dextrin, glue, starch, etc. is added to the dye bath. C. HOLLINS.

Dyeing of cellulose esters. I. G. FARBENIND. A.-G., Assecs. of F. WEBER (G.P. 446,220, 13.8.25).—An aminonaphthoic acid is coupled on the fibre or is diazotised and developed on the fibre; e.g., 2:3-aminonaphthoic acid is diazotised and developed with β -naphthol (strawberry-red), H-acid (bluish-red), phenylmethylpyrazolone (yellowish-orange), or 2:3-hydroxynaphthoic acid (bluish-pink); or is coupled on the fibre with diazotised *p*-nitroaniline (red); or 2:6-aminonaphthoic acid is diazotised and developed with 2:3-hydroxynaphthoic β -naphthylamide (yellow-reddish-brown) or 2:3-hydroxynaphthoic acid (seal-red). C. HOLLINS.

Dyeing of cellulose esters. I. G. FARBENIND. A.-G., Assecs. of K. H. MEYER and H. HOFF (G.P. 446,539, 11.6.24. Addn. to G.P. 428,176; B., 1926, 785).—The process of the prior patent is used with coloured, insoluble, weakly basic, organic compounds generally, e.g., 3-aminobenzanthrone (red), 6-anilino-*N*-methylpyridonanthrone (bluish-red), aminoacridone (fluorescent yellow), diaminophenanthraquinone (violet), 5:5'-diaminoindigo (blue), 1-anthraquinonylhydroxylamine (brownish-red), 1:5-dihydroxylaminoanthraquinone (red-brown), coeramidonine (yellow). C. HOLLINS.

Dyeing etc. of materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD. G. H. ELLIS, and H. C. OLPIN (B.P. 299,349, 20.7.27).—Acetate silk etc. is dyed with acetoacetyl (benzoylacetyl etc.) derivatives of colouring matters which contain amino-groups. Examples are: the acetoacetyl derivatives of aminoazobenzene (greenish-yellow), 2:4-dinitro-4'-aminodiphenylamine (golden-yellow), 1:4-diamino-2-methylanthraquinone (orange). C. HOLLINS.

Dyeing of cellulose acetate. I. G. FARBENIND. A.-G., Assecs. of F. WEBER (G.P. 452,213, 15.1.26).—Acetate silk is dyed with azo dyes formed by coupling a non-sulphonated diazo compound with β -naphthylamine or β -naphthol-8-sulphonic acid; e.g., 2:5-dimethoxyaniline \rightarrow crocein acid (yellow); 5-nitro-*o*-anisidine \rightarrow phenyl- β -naphthylamine-8-sulphonic acid (seal-red); *o*-chloroaniline \rightarrow phenyl acid (reddish-yellow); 2:5-dimethoxyaniline \rightarrow crocein acid (pink). C. HOLLINS.

Dyeing, printing, or stencilling of materials made with or containing cellulose derivatives [esters and ethers]. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 299,343, 19.7.27).—Unsulphonated azo dyes having a naphthylenediamine as end-component dye acetate silk, immunised cotton, etc. Examples are: *p*-nitroaniline \rightarrow 1:5-naphthylenediamine (orange-red), 5-nitro-*o*-anisidine \rightarrow 5-acetamido- α -naphthylamine (red). Certain couplings are disclaimed. C. HOLLINS.

Printing with vat dyes. I. G. FARBENIND. A.-G. (B.P. 279,864, 26.10.27. Ger., 26.10.26).—A cellulose ether or ester, particularly methylcellulose, is added as a thickener to vat-dye printing pastes. The printing material may be stored for a considerable time before steaming. Concentrated pastes suitable for printing from a photographically engraved roller are obtainable. C. HOLLINS.

Dyeing of wool and/or silk. C. SHAW (U.S.P. 1,691,500, 13.11.28. Appl., 7.11.27. U.K., 26.11.26).—See B.P. 277,833; B., 1927, 874.

Dyeing of cellulose esters and ethers. A. GOESCHKE-ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,691,517, 13.11.28. Appl., 4.11.26. Switz., 14.11.25).—See B.P. 261,423; B., 1928, 121.

Colour estimating (B.P. 299,194).—See I. Azo dyes for acetate silk (G.P. 450,920).—See IV. Colour cinematography (B.P. 283,560 and 289,827).—See XXI.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of selenium in sulphuric acid. Z. REICHSTEIN (Trans. Inst. Pure Chem. Reagents [Russ.], 1927, 6, 27—30; Chem. Zentr., 1928, I, 230).—Selenium is determined colorimetrically by means of 4% aqueous codeine phosphate solution. The reaction is more sensitive with acid above about 75% concentration than with dilute acid. Iron gives a similar coloration to selenium, and must therefore be adjusted to the same concentration in the acid under test and in the standard solutions. Arsenic affects the coloration only when present in high concentration. The test is sensitive with 0.0015% Fe and 0.00005% SeO₂. L. A. COLES.

Leucite industry from the chemico-physical point of view. N. PARRAVANO (Giorn. Chim. Ind. Appl., 1928, 10, 501—511).—The importance of the Blanc process for the utilisation of Italian leucite (cf. B., 1925, 240) is shown by the fact that the laval masses of six extinct volcanoes of Central and Southern Italy contain 105,340 million tons of material, representing 8786 million tons of potash. The leucite, separated electromagnetically from the basaltic mass, is treated with hydrochloric or nitric acid, the products being silica and a solution containing aluminium and potassium chlorides or nitrates. Results are now given of determinations of the variation in solubility of these different salts with the temperature and acid concentration, and diagrams are shown for the systems potassium chloride (nitrate)–aluminium chloride (nitrate)–hydrochloric (nitric) acid–water at various temperatures. The heating curves of AlCl₃.6H₂O and Al(NO₃)₃.6H₂O are also

given. Investigation of the X-ray spectra shows that the alumina obtained by heating these salts differs at low temperatures from that derived from bauxite by the Bayer process, but becomes identical with the latter when heated; indications that the dehydration of hydrated alumina occurs by stages are obtained.

T. H. POPE.

Reduction of tricalcium phosphate by carbon. K. D. JACOB and D. S. REYNOLDS (Ind. Eng. Chem., 1928, 20, 1204—1210).—In laboratory tube experiments on the reduction of tricalcium phosphate free from silica by means of pure carbon, conducted in an atmosphere of nitrogen, volatilisation of phosphorus commenced at 1150° and was completed in 1 hr. at 1325°, or in 10 min. at 1500°. The rate of reaction was reduced considerably if the depth of the mixture exceeded 1 cm. Tricalcium phosphate has undoubtedly definite dissociation pressures at high temperatures, and probably the rapid reduction of liberated phosphorus pentoxide by carbon removes the vapour phase and accelerates further dissociation, which follows the course of a unimolecular reaction in the range 1250—1400°. Less than 0.2% of the total phosphorus was converted into calcium phosphide at temperatures up to 1500°.

C. A. KING.

Magnesia-graphite reactions at high temperatures. F. T. CHESNUT (Chem. Met. Eng., 1928, 35, 687).—In a specially designed furnace a sintered magnesia crucible fitted internally with an Acheson graphite sleeve was subjected to a 100-kw. load by induction, observations being made by a sight hole and by means of test rods of refractory oxides. A temperature estimated at about 3000° was reached, and under these conditions much gas is evolved on account of the dissociation of the magnesia. On cooling, the magnesia had not visibly vitrified.

R. H. GRIFFITH.

Reduction of thoria by hydrogen. B. SEGALL (Chem.-Ztg., 1928, 52, 897).—A review of recent researches on the behaviour of thoria in an atmosphere of hydrogen at high temperatures, both alone and in contact with tungsten.

A. R. POWELL.

Thermodynamic properties of oxygen and nitrogen. R. W. MILLAR and J. D. SULLIVAN (U.S. Bur. Mines, Tech. Paper No. 424, 1928, 20 pp.).—By using available experimental data and graphical methods of integration and extrapolation, values of the volume, heat content, and entropy, per g.-mol., are calculated for oxygen and nitrogen over temperature and pressure ranges of 80—300° Abs. and 1—60 atm., respectively. The results are expressed in tables and also as Mollier charts.

F. J. DENT.

Corrosive action of sulphur monochloride. E. H. HARVEY (Chem. Met. Eng., 1928, 35, 684).—Pure metals in lump, stick, or foil form were covered with excess of sulphur monochloride and stored in bottles, closed with Bunsen valves, in diffused light at room temperature for one year; the metal was then washed with carbon tetrachloride, dried, and weighed. Chromium and cobalt were entirely unaffected, whilst the change with nickel, cadmium, lead, and silver was very small, but aluminium, arsenic, antimony, copper, and manganese were badly attacked. It is suggested that the use of drums of alloy

or plated steel would eliminate existing difficulties in the transport of this substance.

R. H. GRIFFITH.

Determination of mercuric iodide. MORAW.—See XX.

PATENTS.

Production of formic acid, acetic acid, and hydrofluoric acid. H. FISCHER (G.P. 452,138, 4.10.21).—The acids are generated in drum-shaped vessels provided with stirring apparatus which by its own rotation or by that of the drum lifts up cylindrical rollers.

L. A. COLES.

Production of hydrofluoric acid. F. SCHUCH (F.P. 628,062, 1.12.26).—Fluorides are heated with mineral acids in two or more vessels attached to one another, the second being wider than the first.

L. A. COLES.

Storage and transportation of acid mixtures in steel drums. J. H. GRAVELL (U.S.P. 1,678,775, 31.7.28. Appl., 11.12.25).—An element between mercury and iron in the electrochemical series, *e.g.*, tin or arsenic, is added to the acid to prevent corrosion. Compounds of the elements, *e.g.*, arsenic trioxide or sodium arsenite, may be used. Known substances having an inhibiting action such as organic bases may also be added.

F. G. CLARKE.

Treatment of caustic solutions for the production of solutions and of solid caustic soda of a high degree of purity. D. A. PRITCHARD, and UNITED ALKALI Co., LTD. (B.P. 299,995, 21.10.27).—Evaporated caustic soda liquors or solutions are treated with a salt or salts of sulphuric acid or with sulphuric acid itself (these reagents are preferably added in the anhydrous form), whereby the major portion of the sodium chloride originally present is precipitated. Liquors containing as little as 0.2 pt. of sodium chloride per 100 pts. of Na₂O in solution may be obtained, and are capable of producing solid caustic soda of a high degree of purity on evaporation.

R. J. W. LE FÈVRE.

Production of mixed crystals containing potassium and ammonium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,984, 13.10.27).—The solid deposit is separated from an aqueous solution saturated with the chlorides and/or the nitrates of potassium and ammonium (and sodium salts, if desired) which is in equilibrium with at least three solid phases of these salts. To the separated solution solid potassium and/or ammonium nitrate or the corresponding chlorides or both kinds of salts and sodium salts, or other salts, if desired, are added, and the resulting deposit of mixed crystals is removed. Soluble phosphates may also be added to the saturated solution.

R. J. W. LE FÈVRE.

Production of ammonium phosphates from mono- or di-calcium phosphate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,796, 29.6.27).—Mono- or di-calcium phosphate or mixtures of the two may be converted into ammonium phosphate by treatment, while stirring, with ammonia in the presence of water. A mixture of calcium phosphate and ammonium monohydrogen phosphate is thereby formed. The reaction is facilitated by raising the temperature and/or pressure. The resulting tricalcium phosphate is recovered, converted by treatment with acids into mono-

and/or di-calcium phosphate, and the product returned to the first stage of the process. R. J. W. LE FÈVRE.

Making oxy-compounds of phosphorus [and fertiliser]. C. G. MINER, ASSR. to PHOSPHORUS HYDROGEN Co. (U.S.P. 1,686,873, 9.10.28. Appl., 27.10.23).—A mixture of phosphatic and potash minerals is reduced, and the liberated potassium and phosphorus are condensed as potassium phosphide. The latter is treated with water or steam, preferably in the presence of nitrogen, whereby a mixture of nitrogen and hydrogen and a solution of potassium hydrogen phosphate are obtained. A complete fertiliser is produced by treating the phosphate solution with ammonia, obtained from the nitrogen-hydrogen mixture by the Haber or Claude process. F. G. CLARKE.

Production of commercial borax from rasorite by a wet method. T. M. CRAMER, ASSR. to PACIFIC COAST BORAX Co. (U.S.P. 1,685,214, 25.9.28. Appl., 22.1.27).—Borax is produced from a natural borate, which contains 2–5 mols. of water and is difficultly soluble, by heating it under pressure with water, preferably by means of live steam in a closed vessel. F. G. CLARKE.

Preparation of silica from silicate solutions. P. SPENCE & SONS, LTD., T. J. I. CRAIG, and A. KIRKHAM (B.P. 299,483, 28.4.27).—Aqueous solutions of alkali silicates, containing about 10% SiO_2 and, if necessary, sufficient alkali carbonate to ensure a molecular ratio after treatment of not less than 2 pts. of alkali carbonate to 1 pt. of silica originally present, are treated gradually (about $\frac{1}{2}$ hr.) at 95–70° or lower with a warm (40–50°) solution of alkali hydrogen carbonate and/or carbon dioxide sufficient to convert all the alkali present into the normal carbonate. Precipitation of the silica in a flocculent, easily filterable, opaque condition is completed by cooling to about 25°. To remove the alkali still remaining after filtering and washing, the product is given further washings with dilute acid and water. The silica, after being dried and sifted, has a bulk density of 0.1. Alkali carbonate is recovered from the mother-liquor and re-used as desired. P. E. L. FARINA.

Production of pure, finely-divided kieselguhr. K. PERL & DR. F. STEINITZER CHEM.-TECH. INST. (G.P. 446,411, 19.9.26).—Kieselguhr is triturated with strong acids before or after treatment with material for destroying organic substances, after which it is calcined, treated with oxidising agents, and washed. L. A. COLES.

Production of lead chloride and nitrates from material containing lead and small quantities of gypsum. SILESIA VER. CHEM. FABR. (G.P. 450,737, 25.11.25. Addn. to G.P. 449,739; B., 1928, 815).—The gypsum is decomposed by treatment with hot lead chloride solution and the lead sulphate formed is washed free from calcium chloride. L. A. COLES.

Utilisation of waste iron pickle. A. J. EVANS (B.P. 299,919, 8.8.27).—The precipitation of hydrated peroxide of iron from iron liquors is facilitated by addition of small quantities of protective colloids (liquid glue, size, etc.) to the mixtures of these liquors with alkaline-earth carbonates. Agitation and aeration then cause a

permanent froth to be formed whereby the mixture is brought into intimate contact with atmospheric oxygen as rapidly as possible. R. J. W. LE FÈVRE.

Production of aluminium sulphate from crystalline aluminium chloride. I. G. FARBENIND. A.-G., ASSEES. of E. C. MARBURG and F. ROSSTEUSCHER (G.P. 452,457, 13.1.24).—Crystalline aluminium chloride is heated at not above 200° to expel the greater part of the hydrogen chloride, steam being applied if necessary, and residual acid is removed by treating the residue with sodium carbonate or bicarbonate, or with ammonia or ammonium carbonate. The residual alumina is dissolved in sulphuric acid of suitable concentration. L. A. COLES.

Manufacture of aluminium sulphate. L. G. PATROUILLEAU, and ALUMINE & DÉRIVÉS (F.P. 627,334, 10.1.27).—Bauxite is treated with sulphuric acid, and the resulting solution is electrolysed hot between iron electrodes and subsequently diluted to precipitate basic aluminium sulphate. A. R. POWELL.

[Colloidal] chromic hydroxide. J. HÄUSLER and B. KOHNSTEIN (Austr.P. 106,829, 28.1.21).—An acid chromic acid solution and an alkaline solution of sucrose are separately atomised into a mixing chamber whereby a fog is obtained from which colloidal chromic hydroxide may be recovered. A. R. POWELL.

Anhydrous magnesium chloride. I. G. FARBENIND. A.-G. (F.P. 624,736, 17.11.26. Ger., 7.12.25).—Crystallised magnesium chloride is dehydrated in a current of a dry gas containing hydrogen chloride, the temperature being regulated according to the hydrogen chloride content of the gas mixture. A. R. POWELL.

Preparation of tungsten oxide gel. W. A. PATRICK and E. H. BARCLAY, ASSRS. to SILICA GEL CORP. (U.S.P. 1,683,695, 11.9.28. Appl., 29.3.27).—Nitric acid (6.38N) is added to an 8% solution of sodium tungstate, $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$, the addition being stopped at each appearance of a precipitate until the latter redissolves on stirring, the final acidity being 0.1–0.5N. The sol sets on keeping to a hydrogel, or may be mixed with a silicic acid sol to produce a mixed hydrogel. C. HOLLINS.

Centrifugal apparatus for the simultaneous neutralisation and drying of salts containing acids. W. VOGEL (G.P. 452,486, 1.7.26).—Gases containing ammonia are blown through the material by a propeller or fan attached to the shaft driving the drum. L. A. COLES.

Production of hydrogen, or hydrogen-nitrogen mixtures, or hydrogen-carbon monoxide mixtures. M. CASALE-SACCHI (B.P. 299,492, 25.7.27).—Carbon monoxide as such or in gases containing it is decomposed completely into carbon dioxide and hydrogen by bubbling through water containing catalysts such as reduced copper, nickel, iron, thallium, platinum, palladium, or metallic oxides or salts of weak inorganic acids, or mixtures thereof, at such a temperature (180–360°) and pressure (20–150 atm.) that the water is kept liquid; thence the gases pass through a heat exchanger to the apparatus for removal of carbon dioxide. Hydrogen or a hydrogen-nitrogen mixture is thus obtained

suitable for hydrogenation, the synthesis of ammonia, etc. By modifying the process incomplete catalysis is possible with the production of hydrogen-carbon monoxide mixtures suitable for organic syntheses (methyl alcohol etc.). The compressed state of the gases is an advantage in the process, and also favours the carbon dioxide elimination.
P. E. L. FARINA.

Recovery of hydrogen from gaseous mixtures rich in same. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 299,558, 29.9.27).—The impure hydrogen, containing about 8% of methane (and homologues) and small quantities of potential catalyst poisons, is washed at -150° and at a pressure up to 400 atm. with a suitable organic solvent, of which liquid methane, obtained in the process, is preferred. Final traces of the impurities are removed by adsorption in active charcoal, silica, alumina gel, natural substances like chabasite, brown coal, etc., either separately or together. The hydrogen remaining is exceptionally pure and particularly suitable for the synthesis of ammonia. The liquid methane and cold hydrogen may be used to precool the impure gas and the adsorbents. When methane has been used in the preliminary washing the adsorbent is sufficiently regenerated by raising the temperature to -80° .
P. E. L. FARINA.

Production of phosphorus. I. G. FARBERIND. A.-G. (F.P. 628,179, 28.1.27. Ger., 2.2.26).—Phosphates are heated with reducing agents and alumina or material containing it.
L. A. COLES.

Manufacture of phosphorus and aluminium. E. COLLET (N.P. 41,453, 17.9.24).—A mixture of 10 pts. of bauxite, 15 pts. of phosphate rock, 3 pts. of coke, and 1 pt. of sodium carbonate is heated in the electric furnace whereby phosphorus distils and aluminium is obtained.
A. R. POWELL.

[Mould for] production of roll sulphur. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., Asses. of W. VON DIETERICH, F. ADEN, and J. LOOSER (G.P. 451,796, 4.2.26).—The moulds are constructed of metal, preferably of aluminium or its alloys.
L. A. COLES.

Control of rate of oxidation [manufacture of sodium monoxide]. H. R. CARVETH, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,685,520, 25.9.28. Appl., 29.1.26. Ger., 6.6.25).—See B.P. 264,724; B., 1927, 218.

Treatment of gases bearing gaseous synthetic ammonia. W. H. KNISKERN, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,690,585, 6.11.28. Appl., 16.8.23).—See B.P. 220,655; B., 1925, 283.

Manufacture of alkali hypochlorites. A. OPFÉ (U.S.P. 1,689,748, 30.10.28. Appl., 26.6.26. Ger., 17.4.26).—See B.P. 258,821; B., 1926, 1013.

Treating earthy minerals. R. W. HYDE, Assr. to DWIGHT & LLOYD METALLURGICAL CO. (U.S.P. 1,688,422, 23.10.28. Appl., 4.4.25).—See B.P. 275,463; B., 1927, 777.

Preparation of sulphur of a high degree of dispersion. H. SCHRADER and H. SCHOELLER (U.S.P. 1,688,357, 23.10.28. Appl., 14.8.24. Ger., 16.8.23).—See B.P. 220,597; B., 1925, 99.

Treatment of gases with liquids (B.P. 299,075). Treatment of compounds or gases by a gaseous reagent (B.P. 274,846).—See I. Ammonium sulphate from gas-works' liquor (B.P. 299,934). Calcium ferrocyanide from gas residues (Dan.P. 34,820).—See II. Titanium oxide (B.P. 299,835).—See XIII.

VIII.—GLASS; CERAMICS.

Effect of temperature change on the colour of red and yellow Lovibond [tintometer] glasses. B. D. JUDD (U.S. Bur. Stand. Res. Paper No. 31, 1928, 8 pp.).—From determinations of the spectral transmission at 15° and 40° of the Lovibond glasses 35Y and 7.2R, separately and in combination, the effect of an increase of temperature of 25° on glasses within the range 35Y to 35Y + 7.2R was found to be practically equivalent to the addition of only 0.2 Lovibond red unit. Two samples of cottonseed oil gave even smaller colour changes over the same temperature range, and, if these were typical, temperature variations of less than 15° need not be considered in colour grading.
A. COUSEN.

Viscosity of glass. S. ENGLISH (J. Soc. Glass Tech., 1928, 12, 106—113 r).—A critical examination of the available methods of determining viscosity. The viscosity range of glass for working was taken as $10^{2.5}$ — $10^{3.5}$ c.g.s. units, and for the annealing point at 10^{13} .
A. COUSEN.

Solidification of amorphous matter [glass]. S. ENGLISH (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 3—6).—A brief synopsis of earlier work of the author on the viscosity of soda and soda-lime glasses. The experimental methods adopted are described and some of the results obtained are shown in a series of graphs.
A. R. POWELL.

Annealing of glass from the point of view of factory operation. E. A. COAD-PRYOR (J. Soc. Glass Tech., 1928, 12, 204—213).—An examination of the principles underlying efficient lehr design.
A. COUSEN.

Relationship between physical properties of glasses and their suitability for manipulation by machine. F. W. ADAMS (J. Soc. Glass Tech., 1928, 12, 114—119 r).—Limiting compositions of colourless and green glass for working on flow and suction-feed machines are given, and conditions conducive to the production of homogeneous glass. The physical properties which the glass should possess to work successfully in the machine are also briefly treated.
A. COUSEN.

Effect of oxidising and reducing agents on selenium glass colours. A. SILVERMAN (J. Amer. Ceram. Soc., 1928, 11, 81).—Selenium produces a ruby colour in zinc-potash glasses in the presence of cadmium sulphide and a reducing agent; an amber shade in lead glasses with an oxidising agent; a pink colour in lime glasses with an oxidising agent without cadmium sulphide; and an orange shade when cadmium sulphide and a reducing agent are present.
F. SALT.

Loss of silica during glass melting. S. R. SCHOLES (J. Amer. Ceram. Soc., 1928, 11, 79).—Silica is the

component which suffers greatest loss during the melting of glass; the finer are the particles of silica introduced, the greater is the loss. When round-grained and dustless sand is used, the silica in the glass is lower by 0.64% (average) than the value calculated from the batch; when sharp-grained, dusty sand is used the loss is greater, viz., 1.13%. This difference is suggested as the reason why sharp-grained sand is the easier to melt.

F. SALT.

Acid-resistant enamels. A. MALINOVSKY (J. Amer. Ceram. Soc., 1928, 11, 110—113).—Three enamel batches formulated by Kerstan (Ceram. Ind., 1927) were made up and tested for resistance to hydrochloric acid in various strengths. All three were badly attacked.

F. SALT.

Vacuum slip process of removing air from clay bodies. G. W. LAPP (J. Amer. Ceram. Soc., 1928, 11, 61—67).—The body slip is passed to the top of a vacuum treating tank before going to the filter press. The slip flows in a thin layer down the sides of the vacuum tank, and is thus exposed to the full action of the vacuum. The benefits of the vacuum treatment to the manufacturing process and to the finished ware are pointed out.

F. SALT.

Determination of workability of plastic clays. S. J. McDOWELL (J. Amer. Ceram. Soc., 1928, 11, 99—102).—Apparatus is described which measures the amount of deformation of plastic clay specimens under a given load (8000 g.) applied vertically. It is simple and robust in construction, and has given good results in practice.

F. SALT.

Warpage study of terra-cotta clays. R. M. MURPHY (J. Amer. Ceram. Soc., 1928, 11, 103—104).—Several types of terra-cotta clays, in bars measuring 9 in. by 1 in. by 1 in., were tested for warpage during drying and firing. Clays of low plasticity reduce the drying shrinkage and warpage; moderate amounts of a sandy clay reduce the firing warpage and shrinkage. Plastic clays have high dry shrinkage and warpage, but they add strength to the body.

F. SALT.

Equations and tables for shrinkage, expansion, and design calculations [in ceramics]. A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1928, 11, 715—723).—Formulae and data eliminating the use of certain incorrect equations are given. To facilitate calculations four-figure tables have been computed.

A. T. GREEN.

Use of fused silica as raw material in the manufacture of porcelain. A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1928, 11, 82—89).—The effect of substituting fused silica for ground quartz sand on the firing behaviour and thermal expansion of a porcelain body was studied. Two bodies were examined, one containing 20% of flint sand, the other 20% of fused quartz. In a reducing atmosphere the fused silica body matured 3—4 cones lower than the other, forming a very dense product. The thermal expansion of the fused silica body when fired under reducing conditions to cone 8 was much lower and more regular than that of the normal body fired to cone 11 under oxidising conditions. At cone 11 small crystals resembling mullite appeared in

the fused silica body. The name "fusilain" is suggested for this body.

F. SALT.

Measurement of thermal expansion of pottery bodies. T. YOSHIOKA (J. Japan. Ceram. Assoc., 1927, 35, 568—572).—A new dilatometer is described comprising a silica tube, a mechanism to rotate a mirror, a lamp, and a scale.

S. KONDO.

Analysis of bauxite and of refractories of high alumina content. G. E. F. LUNDELL and J. I. HOFFMAN (U.S. Bur. Stand. Res. Paper No. 5, 1928, 13 pp., and Bur. Stand. J. Res., 1928, 1, 91—104).—Although the method follows that usually adopted in the analysis of silicates, precautions are necessary when the material has a high content of alumina. Raw bauxite should be dried for 2 hrs. at 140°, and is then hygroscopic. It may be decomposed almost entirely by a mixture of hydrofluoric, sulphuric, and nitric acids, though fusion is necessary with calcined samples. Silica is determined after two evaporations with either hydrochloric or sulphuric acid followed by heating the precipitate at not below 1200°, the usual correction for silica in the ammonia precipitate being applied. Alumina is determined by difference by subsequent treatment of the mixed oxides of aluminium, iron, titanium, zirconium, phosphorus, vanadium, and chromium, precipitated by means of ammonia. The solution of these oxides is treated with 10% caustic soda solution containing a little sodium peroxide, precipitating the oxides of iron, titanium, and zirconium, and chromium is determined colorimetrically in the filtrate, which is then treated with ammonia. After filtering, the ammonia precipitate is dissolved in 10% nitric acid together with a few drops of hydrogen peroxide, and the tint of the solution compared with that of a standard vanadium solution under similar conditions. After oxidation with potassium permanganate phosphorus is precipitated with ammonium molybdate and titrated or weighed. Separation of iron is effected by treating the alkaline solution of iron, titanium, and zirconium oxides with hydrogen sulphide, platinum having been removed first in faintly acid solution. The filtrate containing titanium and zirconium, acidified to give a 10% sulphuric acid solution and cooled in ice water, is precipitated with a cold 6% aqueous solution of cupferron. The curdy precipitate, collected on a paper-pulp filter, is heated at 1200°, weighed as zirconia and titania, and fused with potassium pyrosulphate, zirconium then being precipitated as the phosphate. A new and larger sample (1—5 g.) is generally required for the determination of lime and magnesia. The silica-free solution is nearly neutralised with caustic soda and poured into a 10% solution of caustic soda containing carbonate, the presence of iron, either natural or introduced, aiding agglomeration of calcium and magnesium. The impure precipitate is dissolved, precipitated with ammonia, and lime and magnesia are separated from the filtrate by ordinary methods. Alkalis can be determined satisfactorily by Smith's method. A more rapid method suitable for routine work and depending on the precipitation of aliquot portions of the solution by different reagents is also given.

C. A. KING.

PATENTS.

Strengthened glass. P. H. HEAD (B.P. 299,900, 30.7.27 and 19.4.28).—Diacetone alcohol, alone or mixed with industrial alcohol, tetrachloroethane, *cyclohexanone*, etc., is used for effecting adhesion between celluloid and glass sheets.

L. A. COLES.

Shaping of silica. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of H. P. HOLLNAGEL (B.P. 292,624, 23.6.28. U.S., 23.6.27).—Hollow articles are formed by causing silica rendered plastic by heating at about 1400° to flow over the surface of a rotating convex mould by the application of local pressure.

L. A. COLES.

IX.—BUILDING MATERIALS.

Cause and prevention of kiln and dry-house scum and of efflorescence on face-brick walls.

L. A. PALMER (U.S. Bur. Stand. Tech. Paper No. 370, 1928, 22, 51 pp.).—A distinction is made between efflorescence, kiln scumming, and dry-house or "fixed" scumming. Two sets of tests on various makes of face-bricks have been made, and several types of lime and cement mortars are described. In the first series the bricks alone are stood for 6 months in trays containing about 1 in. of water; in the second, three bricks are built up into small panels with the various mortars and subjected to the same treatment. The soluble salts which cause the efflorescence are for the most part in the form of sulphates, and the results of the tests indicate that both the brick and mortar materials contribute to the efflorescence on a face-brick wall. In an attempt to reduce the efflorescent effect by incorporating a water-repellent substance with the mortar materials, it is shown that the addition of 2% of ammonium or calcium stearate is very effective. The formation of sulphates during drying and burning is next considered. If the waste heat from the kilns is utilised in the dryer, small amounts of sulphur dioxide which are present may be oxidised and adsorbed on the surface of the bricks. Quantitative tests made in a small laboratory oven show that the formation of soluble sulphates in the dryer is minimised by completing the drying process as rapidly as possible. Conditions which make this possible are high temperature, low humidity (and consequently good draft), and a minimum amount of mixing water in the clay. The formation of sulphates during burning was found to be rapid during the water-smoking period and also from 370—540°. This range should be passed through as quickly as possible. At the higher soaking temperature, however, the sulphates begin to be eliminated, and more rapidly in a reducing atmosphere. If this latter condition is impracticable, the soaking temperature should be raised and the process extended. Dry-house scum is caused by soluble salts already in the clay which accumulate on the surface during drying. They are chiefly sulphates of sodium and potassium, which melt before their decomposition and elimination is effected. This "fixed" scum is minimised by rapid drying, a strongly reducing atmosphere at 870—930° (below their m.p.), and by barium treatment of the clay. It is recommended that the soluble salts of barium rather than barium carbonate be used. A brick containing not more than 0.05% of water-soluble sulphuric

anhydride in the outer $\frac{1}{8}$ in. of its exposed surface is not likely to develop efflorescence.

J. A. SUGDEN.

Properties of breeze and clinker aggregates [for concrete], and methods of testing their soundness. F. M. LEA (Dept. Sci. Ind. Res., Building Res. Bull. No. 5, 1928, 17 pp.).—Whether a particular breeze or clinker is suitable for use as an aggregate depends not so much on the amount of combustible matter present, which is not harmful if well carbonised, but on whether it contains uncarbonised coal, particularly of high oxygen content. Such coals undergo a progressive expansion when in contact with the setting cement, and this expansion may be checked by a rapid drying of the mixture, only to assert itself again should it become wetted later. Failures of clinker concretes are rarely due to the presence of sulphur compounds, though limits of not more than 0.45% S (as sulphide) and 0.4% S (as sulphate), or 0.75% of total sulphur excluding sulphates, are indicated. The corrosion of reinforcement in concretes is due to permeability, and the accelerating effect of sulphur compounds is secondary. A test for the resistance to cracking consists in forming a pat of a mixture of 3 pts. of breeze (through 76-mesh) and 1 pt. of a mixture (1:1) of Portland cement and plaster of Paris. The upper surface of the pat formed on a glass plate 4 in. square should be of convex shape and about $\frac{1}{2}$ in. deep at the centre. After setting for 3—4 hrs. in a moist atmosphere the pat is immersed in water and examined for visible cracks during 1—4 days. The unsuitability of breeze is indicated also by its excessive absorption of methylene-blue.

C. A. KING.

Fuel economy in burning Portland cement clinker. R. D. PIKE (Ind. Eng. Chem., 1928, 20, 1155—1163).—Whilst progress has been made in fuel economy by the use of waste-heat boilers, there has been little advance in reducing the consumption of fuel in the cement kiln itself. It is shown by consideration of theoretical heat balances that in a properly designed kiln using the dry process a saving of 40—50% over the wet process should be achieved. Partial corroboration of this view is obtained from the working results of dry shaft kilns.

C. A. KING.

Studies on Portland cement compounds by the X-ray diffraction method. W. C. HANSEN (J. Amer. Ceram. Soc., 1928, 11, 68—78; cf. Harrington, A., 1927, 715).—Methods of preparing compounds of lime with alumina and silica which may occur in Portland cement are examined, and the preparation of $5\text{CaO}, 3\text{Al}_2\text{O}_3$, $3\text{CaO}, \text{Al}_2\text{O}_3$, $3\text{CaO}, \text{SiO}_2$, $\gamma\text{-}2\text{CaO}, \text{SiO}_2$, and $\beta\text{-}2\text{CaO}, \text{SiO}_2$ is described. In an attempt to produce $\alpha\text{-}2\text{CaO}, \text{SiO}_2$, a product was obtained which consisted mainly of twinned crystals, but the X-ray diffraction pattern of which was identical with the β -form. Two mixtures of lime, alumina, and silica were heated at four different temperatures, and the products were studied by the X-ray method. The compounds identified in these products were $3\text{CaO}, \text{SiO}_2$, γ - and $\beta\text{-}2\text{CaO}, \text{SiO}_2$, $3\text{CaO}, \text{Al}_2\text{O}_3$, and CaO .

F. SALT.

PATENTS.

[Portland] cement manufacture [by the wet process]. W. A. SCHMIDT, Assr. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,688,882, 23.10.28. Appl., 21.9.27).

—The flue dust is recovered, allowed to react with water, and then introduced into the raw-mix slurry.

F. G. CLARKE.

Production of dolomite cement. F. W. BROWN, Assr. to G. R. AUSTIN (U.S.P. 1,682,479, 28.8.28. Appl., 10.11.24).—Dolomite free from iron and silica is rapidly heated to 760°, and maintained at 870° until carbon dioxide is removed from the magnesia but not from the lime; the cooled product is then dyed. F. G. CLARKE.

Manufacture of porous masses from mineral substances capable of setting. J. MEYER and E. ASMUS (B.P. 292,468, 7.6.28. Ger., 18.6.27).—Finely-powdered alloys of calcium with zinc and/or magnesium, preferably containing 70–80% Ca, are added to cement etc.

L. A. COLES.

Manufacture of wall board. A. E. WHITE. From UPSON Co. (B.P. 300,041, 30.12.27).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Utilisation of burnt pyrites as an iron ore. C. FICAI (Giorn. Chim. Ind. Appl., 1928, 10, 450–459, 511–521).—A description is given of the plant and method used by the Montecatini Co. at Porto Maghera, where burnt pyrites is used for the preparation of about 400 tons per day of agglomerated ore, mostly utilised by the iron industry. The agglomeration of finely-divided iron ore, in particular of burnt pyrites, by the briquetting process consists in heating the material, previously compressed into blocks, to about 1350°. The various hypotheses advanced in explanation of the changes taking place, viz., (1) formation of silicates in the fused and solid states, (2) semi-fusion (sintering) of the iron oxides, and (3) recrystallisation of these oxides, are examined. Hypothesis (1) is excluded as a result of microscopical examination and in virtue of the possibility of agglomerating pure ferric oxide, and (2) by reason of the difference between the m.p. of the oxides (about 1550°) and the actual temperature of agglomeration, which in certain circumstances can be effected even at about 1200°. The experimental results favour hypothesis (3), and it appears that the red, earthy hematite contained in the burnt pyrites is transformed by the heating into a black, crystalline ferric oxide, magnetite being formed as an intermediate product possessing catalytic properties.

T. H. POPE.

Some constituents of basic open-hearth slag. J. M. FERGUSON (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927–8, [6], 9–18).—Three well-crystallised constituents have been isolated from basic slags of low phosphorus content to which fluorspar has been added to decrease the viscosity, viz., tricalcium silicate in white, orthorhombic plates, tricalcium ferrite in lustrous, black, monoclinic needles (*d* 4), and a blood-red magnetic constituent crystallising in the cubic system. The last-named contains 30–32% CaO, 13.5–14.5% MnO, 5–6% FeO, 5% MgO, 5% Al₂O₃, 32–34% Fe₂O₃, and 8–5% SiO₂. The mechanism of the formation of these compounds in the slag and their function in the refining of iron are briefly discussed.

A. R. POWELL.

Production of high-alumina slags in the blast furnace. T. L. JOSEPH, S. P. KINNEY, and C. E. WOOD (U.S. Bur. Mines Tech. Paper No. 425, 1928, 32 pp.).—Prior to furnace experiment m.p. determinations showed that slags containing 47–48% Al₂O₃, 41–42% CaO, 4–5% SiO₂, together with the usual traces of impurities, were within operating possibilities, though the ternary eutectic (49.5% CaO, 43.7% Al₂O₃, 6.8% SiO₂) has a relatively low viscosity in a considerable range of temperature above the m.p. In a two-weeks' test slags were produced covering a range of composition 6–30% SiO₂, 35–52% CaO, 18–53% Al₂O₃, about 2% TiO₂, about 1% Fe₂O₃, and 0.5% S. No operating difficulties were encountered. Calcium aluminates desulphurise metal rapidly at 1500–1525°, but high-alumina slags lose the avidity for sulphur at lower temperatures. During the experimental run silicon was oxidised from the scrap charge, though this would be less pronounced in full-sized furnaces. The making up of the charge is largely a matter of local costing, but the silica in the coke ash places a limit on the permissible silica in other materials.

C. A. KING.

Microscopical studies of a passive layer in carbon steel and the resulting etch structure. C. BENEDICKS and P. SEDERHOLM (Z. physikal. Chem., 1928, 138, 123–134).—In studying the etch figures produced on a carbon tool-steel by alcoholic nitric acid solutions, an unusual fissured effect was noticed which is attributed to the existence of an extremely thin passive layer; this is produced even with etch fluids containing only 0.1% of nitric acid. The probable origin of this film is discussed.

F. G. TRYHORN.

Secondary hardening of alloy steel. J. H. ANDREW (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927–8, [6], 7–8).—The secondary hardening of hypereutectoidal nickel-chromium steels on tempering above 400° is ascribed to the removal of iron carbide from solid solution, leaving a solid solution which undergoes a critical transformation (γ to α) at 150° with the formation of the hard martensite phase. No secondary hardening takes place when the composition of the steel is such that the γ — α transformation takes place above the decomposition temperature of martensite.

A. R. POWELL.

High-speed steel. J. S. G. PRIMROSE (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927–8, [6], 29–34).—An account of the microstructure, heat-treatment, and working properties of high-speed tungsten steels.

A. R. POWELL.

Manufacture of ferromanganese at the Makievka Works in Central Russia. V. G. KOTELNIKOV (Rev. Mét., 1928, 25, 541–556).—An account is given of the operation during a period of 18 months of a blast furnace producing high-grade ferromanganese from Russian ores. The best results were obtained by the use of a charge giving an acid slag containing 30% SiO₂, 32–34% CaO, 18–20% MnO, 2% MgO, and 9–10% Al₂O₃. The manganese ore should contain at least 45% Mn and not more than 10–14% SiO₂, and the temperature of the blast should be between 750° and 830°. A rapid descent of the charge in the furnace gives a better yield than the usual slow working; it is recommended that the time the ore is in the furnace should not exceed

14 hrs. With an ore containing 46–48% Mn, ferromanganese containing 76–77% Mn can be obtained under the above conditions with a coke consumption of 2.4 tons/ton of metal. The extraction of manganese as metal exceeds 70%; 18% remains in the slag and 12% passes into the flue dust. The latter contains about 30% Mn and 28% SiO₂ and is utilised, together with the slag produced, in the smelting of iron ores in the blast furnace for the production of pig iron. Operating with a basic slag results in higher losses of manganese in the slag and a lower content of manganese in the metal.

A. R. POWELL.

Accelerated laboratory corrosion test methods for zinc-coated steel. E. C. GROESBECK and W. A. TUCKER (U.S. Bur. Stand. Res. Paper No. 10, 1928, 41 pp.).—In duration tests consisting of spraying galvanised steel sheets with a normal solution of sodium chloride or ammonium chloride, the results of the former showed consistent relationship between the "life" of the coating and the thickness of zinc, though the spray method caused local corrosion depending on the distribution of the condensed spray over the surface. A simulated atmospheric attack consisting of a cycle of 5 hrs.' exposure to a warm, moist, gaseous mixture of, by vol., 5 pts. of carbon dioxide, 1 pt. of sulphur dioxide, and 94 pts. of air, followed by 1 hr. of water spraying and 18 hrs. of air drying, resulted in a progressive removal of the coating in the reasonable period of 3–4 weeks. "Copper-steel" (0.2% Cu) produced no observable effect on the life of the zinc coating, though known imperfect coatings, *e.g.*, grey-galvanised, stringy, and blistered, were consistent in showing a lower life than regular sheets of good quality. No attempt was made to correlate results with the life in actual working practice.

C. A. KING.

Recovery of copper and zinc from brass scrap. I. A. KAZARNOVSKI and V. M. ZVENIGORODSKAYA (Papers Karpov Chem. Inst., Bach Mem. Vol., 1927, 101–109).—The oxides formed on calcination are dissolved in sulphuric acid and the solution is boiled with brass turnings; precipitation of copper is practically complete.

CHEMICAL ABSTRACTS.

Determination of sulphur in copper alloys containing tin. H. LEYSANT (Z. anal. Chem., 1928, 75, 169–182).—Methods for determining small quantities of sulphur are examined and details are given. Sulphur may either be oxidised and ultimately precipitated as barium sulphate, or, assuming that sulphur is present entirely as sulphides (as is apparently the case here), the alloy may be treated with a suitable acid and the evolved hydrogen sulphide absorbed in a suitable medium. The whole of the sulphur is completely oxidised by fuming nitric acid, but much of the sulphur is contained in the insoluble stannic oxide. This precipitate must therefore be separately examined, or the whole taken to dryness several times with hydrochloric acid. It is unnecessary to remove copper before precipitation of barium sulphate. The resulting barium sulphate is usually contaminated with tin etc., and requires treatment with fusion mixture and reprecipitation of sulphate. Removal of tin etc. by treatment with ammonia or hydrogen sulphide prior to precipitation of sulphate

leads to untrustworthy results. Accurate results are obtained when tin is removed as bromide by repeated evaporation with hydrobromic acid, but care must be taken to avoid loss by spurting during the evaporations. Oxidation by bromine is dependent on the actual experimental conditions, but satisfactory results are obtained when a saturated solution of potassium chlorate is poured over the material and a considerable excess of bromine is added. When reaction has ceased, hydrochloric acid is added and sulphate precipitated from the boiled, diluted solution. Oxidation methods are, however, tedious, and the use of large quantities of reagents necessitates the carrying out of a large number of blank determinations. The whole of the sulphide-sulphur is evolved when the alloy is treated with a solution of hydrogen bromide. Suitable absorbing media are brominated hydrochloric acid, in which case sulphur is precipitated as barium sulphate, and a solution of cadmium acetate. In the latter case sulphide is determined iodometrically, or converted into cupric sulphide by treatment with a solution of copper sulphate and weighed as oxide. The whole of the sulphur is not evolved when hydrobromic acid is replaced by hydrochloric acid. This is especially true when the alloy is in the form of coarse turnings etc. J. S. CARTER.

Mechanism of crystal growth by annealing. T. SUTOKI (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 857–876).—The crystal growth of aluminium containing 0.4% Fe and 0.2% Si has been followed after straining to 1–2% elongation and annealing at 570–630°. Crystals of macroscopic size in contact with one another do not grow, however prolonged the annealing, but a single crystal in contact with strained microcrystals grows steadily at their expense. Repeated heating and cooling produces many macrocrystals if the rate of cooling is rapid, but intermediate sudden coolings by quenching cause the formation of very large crystals surrounded by minute grains. From annealing experiments on tin containing 1.5% Sb the crystal growth of unstrained metals has been shown to proceed along concave grain-boundaries in such a manner that the radius of curvature decreases with time of annealing. In this case large crystals may grow at the expense of smaller ones, and small crystals at the expense of larger ones, and a crystal may grow on one side and be encroached upon on another side. Thus it appears that to cause crystal growth on annealing the presence of some minute gaps in the grain-boundary or of nuclei along slip-planes caused by straining is necessary.

A. R. POWELL.

Production of tungsten in rod or powder form from a single crystal. I. TARJÁN and P. TÚRY (Chem. Runds. Mitteleuropa Balkan, 1927, 4, 165–168; Chem. Zentr., 1928, I, 237–238).—The tenacity of tungsten wire used in the electric incandescence lamp is largely dependent on its production from a single crystal. Reduction of tungsten trioxide is best effected by heating at 700–950° in hydrogen; the size of the particles, which should average 0.1μ, should not be greater than 1.0μ. The tungsten, containing some thorium, is cast into rods weighing 85 g. in an atmosphere of hydrogen and heated for a short time only

in the neighbourhood of its m.p. Crystal formation takes place in cylindrical surfaces starting at the interior and working outwards. L. A. COLES.

Corrosive action of sulphur chloride. HARVEY.—See VII. **Effect of lead and zinc fumes on soil.** LUNDEGÅRDH.—See XVI.

PATENTS.

Blast furnaces. J. P. DOVEL (B.P. 299,941, 25.8.27).—Hollow bronze cooling blocks are inserted into the lining of a blast furnace and rigidly bolted to the shell, so that as the refractory lining erodes a new lining formed from the furnace charge constituents collects and bonds about the cooling elements. In the upper part of the furnace, as far as the temperature permits, detachable metallic wear plates are fitted and spaced to allow of free expansion. For the proper formation of the stock column the charging bell should have an area, at its base, equal to about one third that of the shaft at the stock line. C. A. KING.

[Open-hearth] furnace. R. H. STEVENS, Assr. to BETHEHEM STEEL CO. (U.S.P. 1,689,563, 30.10.28. Appl., 2.9.21).—The forward end of a tuyère port provided with a flue connexion to a regenerator projects within the rear end of a tuyère entrance port likewise provided with a flue connexion to another regenerator. Additional openings controlled by valves are arranged near the furnace end of the regenerators, and means are provided for controlling the flow of gases to the ports.

J. S. G. THOMAS.

[Mill] furnaces [for steel bars]. D. WILLIAMS (B.P. 298,507, 9.7.27).—The furnace comprises a combustion chamber, heating chamber, and flues under the heating chamber through which the products of combustion pass to and fro before reaching the stack. Over the firebridge between the fire and goods chamber is a perforated wall. B. M. VENABLES.

Welding high-carbon steel. J. H. DEPPELER, Assr. to METAL & THERMIT CORP. (U.S.P. 1,686,603, 9.10.28. Appl., 29.8.25).—A thin sheet of iron with a low carbon content is placed between the two parts to be joined, and the edges are then butt-welded by aluminothermic methods. A. R. POWELL.

Increasing the yield point in hollow bodies from steel alloys, particularly austenitic steel alloys. F. KRUPP A.-G. (B.P. 292,937, 5.6.28. Ger., 23.6.27).—Austenitic steels in the form of hollow rotary bodies are worked in a hot state and turned or expanded, *e.g.*, on a mandril, so that the stress induced by this means exceeds the original yield point. C. A. KING.

[Nickel-chromium] alloy steel and articles made therefrom. F. B. DEHN. From THOMPSON PRODUCTS INC. (B.P. 298,250, 8.4.27).—The steel contains 11.5–13% Cr, 7–8% Ni, 2–3% Si, and 0.25–0.35% C. The structure is rendered non-austenitic by annealing at 870–900° for 6 hrs. and cooling at the rate of 30°/hr. for 5 hrs. After working into the desired shape the alloy is further annealed at 620–675° and air-cooled or quenched in oil. A. R. POWELL.

Stable-surface iron-chromium-silicon alloy. P. A. E. ARMSTRONG, Assr. to LUDLUM STEEL CO. (U.S.P.

1,686,223, 2.10.28. Appl., 19.2.24).—An iron alloy containing more than 13% Cr, 0.3–5.0% Si, and less than 0.05% C is prepared by adding a preheated ferrochromium alloy to degasified molten iron. The ingots are forged hot and rolled first at 1040–1260°, then heavily rolled at about 600°. A. R. POWELL.

Iron alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 299,502, 27.7.27).—An iron alloy, suitable for the manufacture of armoured wire, having a tenacity of the order of 100 tons/in.², developed by cold work, contains up to 15% of copper and silicon in about equal proportions; a suitable composition is 3–6% Cu, 3–6% Si, and remainder iron, with or without the addition of a small quantity of a deoxidiser such as manganese. M. E. NOTTAGE.

Method of making alloy steels. C. MCKNIGHT, JUN., Assr. to INTERNAT. NICKEL CO. (U.S.P. 1,689,043, 23.10.28. Appl., 8.6.25. Renewed 13.3.28).—High-silicon scrap metal is melted under oxidising conditions to reduce the silicon content below a critical point not exceeding 0.1%, then subjected to reducing conditions with the addition of alloying materials. C. A. KING.

Metallurgical furnace. H. BUTT, Assr. to RILEY STOKER CORP. (U.S.P. 1,687,470, 9.10.28. Appl., 25.3.25).—The combustion chamber is provided with a retort to which the fuel is supplied and with a "fuel support" (fire-grate) to the side thereof (away from the firebridge) on to which the fuel overflows from the retort. Air passes through the fire-grate and a smaller amount is forced into the retort, producing a large quantity of combustible gas. The roof of the furnace is close to the bed of fuel to restrict combustion, and sloping to reflect heat into the charge in the retort; it is low over the fire-bridge, and immediately after dips down over the charge in the heating chamber so as to cause the gases to mix thoroughly and burn as a long flame.

B. M. VENABLES.

Smelting apparatus. C. E. GLESSNER (U.S.P. 1,686,912, 9.10.28. Appl., 9.6.27).—Crushed ore is fed uniformly into the top of a "centrifugal extractor" having a vertical axis and annular grooves. The bottom of the extractor is sealed by a bath of the metal produced. Air is drawn downwards through the apparatus.

B. M. VENABLES.

Centrifugal concentrating and amalgamating apparatus of the vertical type. F. D. LEWIS (U.S.P. 1,684,870, 18.9.28. Appl., 17.10.27).—The material is fed through an axial pipe to the bottom of a rotating tapering "cylinder" on the interior of which are formed annular grooves, and passes upwards into a bowl also provided with grooves and rotating. A disc sliding on the feed pipe controls the opening between the cylinder and bowl. B. M. VENABLES.

Concentration of ores and minerals by flotation. W. A. DOUGLASS, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,674,166, 19.6.28. Appl., 25.6.26).—Thionurethanes, NHR·CS·OR', are used with frothing agents for ore flotation; examples in which R = H, Me, or Et, and R' = Me, Et, Prⁿ, or Buⁿ are mentioned. C. HOLLINS.

Apparatus for treating [metallic] fumes. M. I. DORFAN, Assr. to ALLIS-CHALMERS MANUF. CO. (U.S.P.

1,685,229, 25.9.28. Appl., 21.11.23).—Combustible gases, escaping from the mouths of a bank of retorts for reducing zinc or other ores, enter combustion zones formed by hoods into which air is introduced. The products of combustion are passed from the hoods to a separator to deposit solids. The forward and rear walls of each hood are movable in order to control the air supply and to give access to the retorts. F. G. CLARKE.

Treatment of complex minerals containing tin, and/or copper etc. and associated metals. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING (B.P. 299,750, 30.7.27).—Complex tin ores containing sulphides of iron, copper, etc. are treated with a solution containing persulphates and chlorides of the alkalis together with ferric sulphate or chloride and a small proportion of free chlorine. From the leach liquor so obtained copper or tin, or both, may be recovered by electrolysis or by precipitation by hydrogen sulphide and the waste liquor regenerated by electrolysis. A. R. POWELL.

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 300,078, 3.4.28).—An alloy of aluminium contains 0.2–1.5% Ni, 0.2–5% Mg, 0.5–2% Cu, 0.2–0.5% Si, 0.7–1.5% Fe, and not more than 0.25% Mn (Ti). C. A. KING.

Manufacture of articles of refractory metal or metallic mixtures. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,675,119, 26.6.28. Appl., 10.4.22).—To avoid fashioning the articles, *e.g.*, X-ray targets, crucibles, at high temperatures, a cream is formed of a refractory metal powder, cryolite, phosphoric acid, and water, and a plaster of Paris mould is filled therewith. After settling, the liquid is poured off and the deposit dried. The article is removed and fired in hydrogen. F. G. CLARKE.

Metal pickling. J. H. GRAVELL and A. DOUTY (U.S.P. 1,678,776, 31.7.28. Appl., 12.11.26).—Stannous chloride is added to prevent pitting, but in insufficient quantity to dissolve the scale removed from the metal. Organic substances, such as glue, formaldehyde, or preferably those which produce a foam (*e.g.*, flour and sulphite-cellulose waste), may be added to trap noxious gases and prevent oxidation of the tin. F. G. CLARKE.

Coating non-conducting substances with metals. A. I. and E. L. GATES-WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (B.P. 299,903, 2.8.27 and 2.4.28).—A solution of a metallic salt, *e.g.*, silver nitrate, and a finely-divided metallic precipitate dispersed in a suitable carrier, *e.g.*, silver, are applied successively or simultaneously to the abraded surface of the substance, *e.g.*, a manufactured protein product, and the metal salt is reduced by formaldehyde or other reducing agent. J. S. G. THOMAS.

Using direct metal from blast furnaces. R. MOLDENKE, Assr. to NEW PROCESS MULTI-CASTINGS Co. (U.S.P. 1,691,401, 13.11.28. Appl., 19.12.24).—See B.P. 267,590; B., 1927, 369.

Solder for aluminium. O. SPENGLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,691,532, 13.11.28. Appl., 13.7.25. Ger., 24.9.24).—See B.P. 240,411; B., 1925, 996.

Production of magnesium. W. KOEHLER (B.P. 300,149, 29.7.27).—See U.S.P. 1,650,893—4; B., 1928, 95, 96.

Apparatus for charging tilting smelting furnaces. DEMAG A.-G. (B.P. 286,224, 25.2.28. Ger., 28.2.27).

Calcining furnace (U.S.P. 1,686,565). **Treatment of foundry sand** (B.P. 298,384).—See I. **Waste iron pickle** (B.P. 299,919).—See VII. **Electric furnace for metal bands** (U.S.P. 1,689,369). **Electrodeposition of metals** (U.S.P. 1,689,597). **Electrodeposition of chromium** (B.P. 300,043).—See XI.

XI.—ELECTROTECHNICS.

Effect of hydrogen-ion concentration on the voltage of the Leclanché dry cell. B. M. THOMPSON (Ind. Eng. Chem., 1928, 20, 1176–1178).—Improved technique in which the hydrogen-ion concentration of a solution is obtained from a Leclanché cell under equilibrium conditions has shown that mixtures of manganese dioxide and graphite give straight lines when the voltage, referred to the normal calomel electrode, is plotted against the p_H value of the solution. Natural manganese ores gave a line with a slope of -0.07 , as compared with the theoretical value -0.059 , the difference being probably attributable to the presence of oxidising ions other than quadri- or ter-valent manganese ions. Graphite acts as an inert electrode without influence on the reaction. Artificial oxides vary greatly in properties, depending on the method of their preparation. C. A. KING.

Wood separators for lead storage batteries. C. A. ROBAK (Ind. Eng. Chem., 1928, 20, 1151–1152).—As a result of long-period trials of accumulators under working traffic conditions the destructive effect of the positive plate on the wood substance cannot be considered an objection to placing a flat side of a wood separator next to the positive plates. After several years' use separators next to the negative plates showed a mechanical strength about 30–45% higher than those in contact with the positive plates, though the values were only about 60% that of the original wood. C. A. KING.

Automatic control. SWAN.—See I. **Reduction of thoria by hydrogen.** SEGALL.—See VII. **Control in beet-sugar manufacture.** BALCH and KEANE.—See XVII. **Purification of water.** ATEN.—See XXIII.

PATENTS.

Electric furnace. F. T. COPE, Assr. to ELECTRIC FURNACE Co. (U.S.P. 1,690,273, 6.11.28. Appl., 12.1.27).—Independently detachable spaced blocks having ledges on the under side of a refractory furnace roof are supported independently of the roof, and the end parts of rigid resistor grids are supported only on the ledges. J. S. G. THOMAS.

Electric furnace. G. SAGRANOSO (U.S.P. 1,690,795, 6.11.28. Appl., 19.5.24. Italy, 28.5.23).—A double-walled jacket, secured to a cross-beam adjustably mounted on standards and surrounding an electrode secured to the beam and movable therewith, is adapted to slide within a double-walled bell supported by the furnace. Means are provided for connecting the bell with the beam in such manner that the electrode, jacket, and beam can be elevated with respect to the furnace. J. S. G. THOMAS.

Electric furnace for glowing metal bands by induction currents. M. TAMA (U.S.P. 1,689,369, 30.10.28. Appl., 29.11.27. Ger., 17.9.27).—Metal strip to be annealed is coiled round a drum and forms one of the coils of a transformer designed to generate the induction current. J. S. G. THOMAS.

Electrical gas-purifying apparatus. METALLBANK & METALLURGISCHE GES. A.-G., and R. LADENBURG (G.P. 452,394, 17.11.22).—Particles in the gases under treatment having an opposite charge to that of the emission electrode, in apparatus comprising an emission electrode with a precipitation electrode around it having a considerably lower current density, have their charge neutralised by adding oppositely-charged ions before the gases enter the electric field. L. A. COLES.

Cleaning gas-purifying apparatus by heating the electrodes. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 452,437, 3.8.24).—A precipitation electrode in the form of an endless band is heated, *e.g.*, by hot rollers as soon as it leaves the electric field, passed through liquid to clean and cool it, and brought in contact with scrapers to remove adhering liquid before it is returned to the field. L. A. COLES.

Gas-filled incandescence cathode discharge tube. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 299,931, 12.8.27).—A gas-filled discharge tube adapted for the emission of ultra-violet radiation comprises an oxide cathode or an incandescence cathode containing oxide, *e.g.*, a tungsten wire wound with nickel wire coated with barium oxide, one or more solid anodes, *e.g.*, of carbon, a gaseous filling of mercury vapour and a rare gas, *e.g.*, argon, and a wall composed of quartz or partly of quartz and partly of glass, the respective coefficients of expansion of the glasses increasing from the quartz to the glass. An auxiliary anode is arranged near the incandescence cathode to start the discharge. J. S. G. THOMAS.

Electron-discharge device. J. E. HARRIS, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,689,338, 30.10.28. Appl., 19.11.21).—A cathode composed of mischmetal is used. J. S. G. THOMAS.

Electron-emitting cathodes. RADIOTECHNIQUE SOC. ANON. (B.P. 281,262, 10.11.27. Fr., 26.11.26).—A wire core, *e.g.*, of tungsten, of fairly large diameter, is covered with an adherent coating of refractory and insulating material, *e.g.*, magnesia, by being heated to about 3200° Abs. in a bed of the powdered refractory, and is then covered with a sheath of electron-emitting material, *e.g.*, thoriated tungsten, wound helically, the respective m.p. of the core and coatings decreasing from the inside outwards. J. S. G. THOMAS.

Producing rapidly-moving electrons and subjecting matter thereto. H. E. POTTS. From H. PLAUSON (B.P. 299,735, 30.5.27).—Cathode rays are subjected to the action of intense electron fields and/or rotating electromagnetic fields in a reaction chamber. J. S. G. THOMAS.

Method of exhausting vacuum devices. H. C. RENTSCHLER, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,689,297, 30.10.28. Appl., 16.8.22).—A plate to which

a piece of aluminium is attached is sealed into a bulb, which is then exhausted to a moderate vacuum. The plate is heated to drive off occluded gases and the heating continued; the aluminium is vaporised and residual gases are eliminated. J. S. G. THOMAS.

Accumulator with negative zinc electrodes. A. BRANDES and P. O. H. RICHTER (G.P. 452,573, 12.5.26).—The positive electrode is constructed of strips of lead covered with a mixture of lampblack etc. with lead monoxide wrapped around with suitable material and connected with a supporting plate, and the electrolyte consists of zinc or an alkali sulphate solution. L. A. COLES.

Production of [accumulator] plates of spongy lead stable in the air. ACCUMULATOREN-FABR. A.-G. (G.P. 451,492, 18.8.26).—A small portion of the charge is withdrawn from the fully-charged plates by short exposure to a strong current before they are washed and dried in the absence of oxygen. L. A. COLES.

[Gas] accumulator. SOC. ANON. LE CARBONE (B.P. 291,420, 15.11.27. Fr., 4.6.27. Addn. to B.P. 230,307; B., 1925, 364).—A metal, *e.g.*, lead, forming with chlorine a chloride which is, preferably, insoluble is incorporated with the active mass of porous impermeable carbon used in the accumulator. J. S. G. THOMAS.

[Porous electrodes for] galvanic cells. P. PÖRSCKE (B.P. 299,591, 8.7.27).—The porous paste produced by adding an acid or base to a mixture of metals and/or oxides is moulded or spread on a carrier. J. S. G. THOMAS.

Means for preventing explosions. H. C. P. WEBER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,686,051, 2.10.28. Appl., 23.3.22).—Natural, sewer, or other explosive gas is burned in a Davy safety lamp provided with an electrically-heated filament comprising a catalyst such as nickel. A thermocouple may be incorporated to indicate when explosive gas is present. F. G. CLARKE.

Preparation of organic articles for electrolytically covering them with a metallic layer. I. AINSTEIN (W. I. EINSTEIN) (B.P. 300,060, 22.2.28).—The organic article is dipped in soluble glass, dried, and its surface sanded. It is then given a thin preparatory metallisation with an alloy of easily fusible metal, *e.g.*, lead, antimony, bismuth, tin, or zinc, by pulverisation with a gun; if desired, this is followed by a similar second metallisation with copper or its alloys. The article is then lightly plated in an electroplating bath at below 10° before being subjected to the main plating process in a hot bath. J. S. G. THOMAS.

Cell for electrodeposition of metals. R. D. PIKE, G. H. WEST, and B. P. LITTLE, Assrs. to R. D. PIKE (U.S.P. 1,689,597, 30.10.28. Appl., 23.9.25).—A cell comprises cathode and anode compartments, separated by porous diaphragms, means for maintaining a constant level of catholyte in the cathode compartment and for removing anolyte from anode compartments, common channels connected with the anode compartments on each side, and means for automatically admitting catholyte in order to maintain the level of the anolyte about constant. J. S. G. THOMAS.

Electrodeposition of chromium. E. V. HAYES-GRATZE (B.P. 300,043, 5.1.28).—When electroplating with chromium the anode is made of pure electrodeposited chromium of the reverse shape of the surface of the article to be plated; e.g., a cast or mould of the article may be plated with chromium and used as an anode, or the article itself may be first used as a mould and the deposit of chromium stripped off for use as the anode.

C. A. KING.

Insulating material. BRIT. THOMSON-HOUSTON CO., LTD., H. W. H. WARREN, R. I. MARTIN, and A. E. SMITH (B.P. 299,906, 3.8.27).—Layers of fabric, paper, or asbestos are held together by a synthetic resin of the phenolic condensation type; the whole is wrapped with a thin sheet of paper or fabric impregnated with shellac and bonded by subjection to heat and pressure.

J. S. G. THOMAS.

Construction of electroplating vats or troughs. K. BARANOVSKI (B.P. 300,038, 21.12.27).

Coke electrodes (B.P. 295,314).—See II. **Electrodeposition of rubber** (B.P. 299,738).—See XIV.

XII.—FATS; OILS; WAXES.

Fat in the residual material remaining after the unwinding of the silk cocoon. J. JELAKOV (Oil Fat Ind. Russia, 1926, No. 10—11, 35).—The unwound cocoon contains a fat (25%) having *d* 0.919, saponif. value 191.0, iodine value 105.2, Reichert-Meissl value 3.24, mol. wt. 283, m.p. 30°. The residue contains protein (60%) and phosphorus. CHEMICAL ABSTRACTS.

Fluorescence of oils and fats. J. F. CARRIÈRE (Chem. Weekblad, 1928, 25, 632—634).—A criticism of the conclusions of van Raalte (B., 1928, 792). The statement that crude materials do not fluoresce, but refined do, is incorrect. The material under examination should be placed below the lamp and viewed from above, since the surface generally shows the strongest fluorescence. The fluorescence of many vegetable oils is actually diminished by refining, but no definite rule can be laid down. The phenomenon is very complicated, but the possibility that vitamins play any part is extremely remote.

S. I. LEVY.

Determination of fats. A. TICHONOV (Oil Fat Ind. Russia, 1926, No. 6, 38—40).—Soxhlet's method of fat extraction, using ether or light petroleum, extracts 97.6% in 1 hr., or all in 5—6 hrs. Cold extraction for 2—3 days extracts nearly all the fat. In Soxhlet's apparatus a separating funnel and a tube are placed between the flask and the extractor so that the solvent can be collected after the final extraction. Fat can be determined by measuring the density of the extracted material, and by weighing the residue after extraction.

CHEMICAL ABSTRACTS.

Hydrogenation of fatty oils. VII. **Formation of isooleic acid by the hydrogenation of soya-bean oil.** VIII. **Hydrogenation of the methyl esters of oleic and linoleic acids.** T. MAZUME (J. Soc. Chem. Ind., Japan, 1928, 31, 467—469, 470—472).—VII. Formation of isooleic acid was less when the temperature of hydrogenation was low, and increased with the amount of catalyst used. When the nickel catalyst contained a

small amount of copper (0.3%) the formation of the acid was very great; large amounts of copper (4%) considerably reduced the amount of acid formed. Nickel catalysts that had, after reduction, been exposed to air at a low temperature produced more isooleic acid than those exposed at high temperatures.

VIII. During the hydrogenation of methyl oleate a large amount of isooleic acid was formed at the beginning of the process, and the acid so formed was gradually transformed into oleic acid by further hydrogenation. In the case of the methyl ester of linoleic acid the transformation was linoleic acid \rightarrow oleic acid \rightarrow isooleic acid \rightarrow stearic acid.

Y. TOMODA.

Polymerisation of tung oil and the ethyl ester of elæostearic acid by heat. T. MAZUME and S. NAGAO (J. Soc. Chem. Ind., Japan, 1928, 31, 473—476).—When tung oil was heated at 200—300° its mol. wt. was doubled, but its iodine value did not much decrease; it is considered, therefore, that the change was one of extramolecular polymerisation. In case of the ethyl ester of elæostearic acid the change was similar, but unlike the oil the ester did not solidify.

Y. TOMODA.

Nutritive value of hardened oils. II. S. UENO, M. YAMASHITA, and Y. OTA (J. Soc. Chem. Ind., Japan, 1928, 31, 393—399).—Tests on animals showed the nutritive value of the low-temperature hardened oils (sardine oil, herring oil, finback whale oil, chrysalis oil) to be superior to that of the original oils.

Y. TOMODA.

Examination of cresol-naphthene soap solutions. G. KOGAN (Pharm. Zentr., 1928, 69, 681—684).—The material is shaken with ether and dilute sulphuric acid saturated with sodium sulphate; the aqueous layer contains the bases. The naphthenic acids are removed from the ether solution by shaking with sodium carbonate solution, and after separation the cresols are removed by shaking the ethereal layer with sodium hydroxide solution, leaving only hydrocarbons in the ether solution.

S. I. LEVY.

Vulcanisation of oils. II. P. STAMBERGER (Rec. trav. chim., 1928, 47, 973—976; cf. B., 1928, 23).—When triolein (5 pts.) is heated with sulphur (1 pt.) at 160° a viscous fluid is obtained which is soluble in benzene, but only partly soluble in acetone. The mol. wt. of the acetone-soluble and insoluble products are 1204 and 2040, respectively. The fluid products are converted into elastic solids (cf. *loc. cit.*) by (a) keeping at room temperature for several months; (b) heating to 180°; (c) irradiation with ultra-violet light; and (d) treatment with some chemically indifferent solvents (acetone or water). Whilst the solids produced are only slightly soluble in benzene, they swell considerably in this liquid, and the amount of benzene absorbed varies with the hardness of the solid. The benzene-soluble product previously described (*loc. cit.*) changes to an elastic solid on keeping for 3—4 months at room temperature. This solid swells in cold benzene, is soluble in hot benzene, and the product from this solution redissolves in the cold solvent. The solvent thus appears to have some action on the gel, probably causing an aggregation or polymerisation of the molecules (cf. Stevens, B., 1928, 203).

H. BURTON.

Prevention of autoxidation of vegetable oils and their fatty acids. V. NOVIKOV (Oil Fat Ind. Russia, 1927, No. 2, 17—20).—The rate of absorption of oxygen by linoleic acid, and the action of various organic substances in retarding the oxidation, were studied. β -Naphthol (1%) prevents the oxidation of linoleic acid in oxygen at 60°. Linseed oil containing 1% of β -naphthol did not change in iodine value or viscosity during 2 years. CHEMICAL ABSTRACTS.

Differences in crude soya-bean oils from various sources. J. F. CARRIÈRE (Chem. Weekblad, 1928, 25, 630—632).—The crude expressed oil from Manchuria has an abnormally low sp. gr. (d_{20}^{20} 0.9219), an abnormally high content of unsaponifiable matter (0.80—1.07%), and a characteristic bluish fluorescence in ultra-violet light. Extracted Manchurian oil on the other hand is normal in all three properties. The expressed oil appears to be contaminated with about 0.4% of mineral or resin oil. S. I. LEVY.

Oil from apricot stones. V. TEMNOV (Oil Fat Ind. Russia, 1926, No. 10—11, 67—68).—The oil (yield 37% out of 47%) had d_4^{20} 0.915, iodine value 101.4, saponif. value 196, acid value 3.11. CHEMICAL ABSTRACTS.

Obtaining average samples of oil cake. F. GOGOLEV (Oil Fat Ind. Russia, 1926, No. 4—5, 46—49).—Variations of the oil content in different parts of the cake are recorded, e.g., 7.03% in the middle, 8.03% at the edges; uppermost layer in the press 0.5% more than others. CHEMICAL ABSTRACTS.

Volumetric analysis of oil cakes for their oil content and moisture. A. LAPTEV (Oil Fat Ind. Russia, 1926, No. 4—5, 49—52).—The finely-divided substance is kept overnight in ether, and then extracted for 3—3.5 hrs.; the bottom of the apparatus contains a filter plug of defatted wadding. The last traces of ether are expelled in an oven in a current of carbon dioxide. CHEMICAL ABSTRACTS.

Determination of the oil content of linseed by the results of mechanical analysis of the purity. J. ORLOV (Oil Fat Ind. Russia, 1927, No. 1, 1—16).—The oil content is $0.355a + 0.05b$, where a is the content (%) of pure linseed and b the content of other oil-bearing seeds. CHEMICAL ABSTRACTS.

Saponification with the Twitchell and Pfeilring reagents. G. PETROV (Oil Fat Ind. Russia, 1926, No. 6, 25—28; No. 7—8, 21—27).—The aromatic thio-fatty acids of anthracene, phenanthrene, benzene, and xylene are less active than those of naphthalene. The Pfeilring cleavage agent is examined. CHEMICAL ABSTRACTS.

Seed oil of *Citrus limomum*. OCCHIPINTI.—See XX.

PATENTS.

Generation of oxygen from washing and bleaching agents containing persalts. G. LESCHNIK (B.P. 273,711, 27.6.27. Ger., 5.7.26).—A catalyst, e.g., a manganous compound, or tyrosinase expressed from fresh potato skins, is added to such washing and bleaching powders, during use, to obtain quantitative development of oxygen. W. G. CAREY.

Production of material of the consistency of ointment from colophony. A. RIEBECK'SCHE MONTANWERKE A.-G. (G.P. 451,180, 23.6.22).—Material suitable for use in the manufacture of soap, textile preparations, etc. is obtained by the catalytic hydrogenation of a mixture of colophony and castor oil at raised temperature and pressure. L. A. COLES.

Soap product and its manufacture [by spraying and drying]. INDUSTRIAL SPRAY-DRYING CORP., ASSEES. OF D. R. LAMONT (B.P. 291,041, 8.8.27. U.S., 25.5.27).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Microscopical examination of lithopones and zinc whites. V. ALLARD (Ann. Falsif., 1928, 21, 445—449).—“Molecular” lithopone, which is obtained by double precipitation, may readily be distinguished by means of the microscope from “mixed” lithopones, obtained from a mixture of zinc sulphate and chloride with barium sulphide such that 60—70% of zinc sulphide is produced, and to which is added white baryta, to reduce the proportion of zinc sulphide to about 30%. The “molecular” product presents under the microscope a homogeneous, amorphous structure, whilst such products as baryta and chalk have a translucent crystalline appearance. The effect is more marked if polarised light is used with interposition of a layer of gypsum to give a red background, when the dark, amorphous, “molecular” lithopone is in marked contrast to the brilliant colours exhibited by the salts of the mixture. D. G. HEWER.

Brewers' pitch. KUTTER.—See XVIII.

PATENTS.

Preparation of pigments. L. A. LEVY and D. W. WEST (B.P. 299,797, 1.7.27).—A very finely divided condition of the zinc sulphide or zinc cadmium sulphide used for phosphorescent and fluorescent paint is ensured by the addition, before firing, of up to 20% of white rare-earth or alkaline-earth oxides, and discoloration in sunlight etc. is prevented by the addition, after firing, of a solution of trisodium phosphate, sodium carbonate, or borax, the solvent being then evaporated. W. G. CAREY.

Manufacture of titanium oxide [pigments]. F. G. C. STEPHENS, L. J. ANDERSON, and W. A. CASH (B.P. 299,835, 2.8.27).—Titanium sulphate solution containing about 16% TiO_2 , preferably prepared as described in B.P. 256,734 (B., 1926, 876), is added slowly with agitation to a solution containing, e.g., 100 pts. of barium chloride per 750 pts. of water at 25—40° (preferably at 30—35°), and the mixture is heated at 100° by steam until all the titanium is thrown out of solution. The precipitate is removed, washed, and suspended in borax or boric acid solution before calcination. L. A. COLES.

Improvement of recent natural acid resins. CHEM. FABR. DR. K. ALBERT G.M.B.H. (G.P. 440,003, 9.8.17).—Natural acid resins are combined with phenol-aldehyde resins, the remaining acidity being reduced by esterification or addition of lime. In this way the less valuable natural resins, such as pine rosin, acquire properties resembling those of the copals. Colophony,

crude cresol, and formaldehyde are heated to give a clear resin, volatile products and water are removed, and warm glycerin is gradually added, reaction being completed at 250–260°; or the preformed cresol-formaldehyde resin may be dissolved in molten colophony. Glycol, arabitol, or other multivalent alcohols may replace glycerin, or lime or calcium carbonate may be used. C. HOLLINS.

Synthetic resin and its manufacture. L. T. RICHARDSON, Assr. to CUTLER-HAMMER MANUF. CO. (U.S.P. 1,682,934, 4.9.28. Appl., 15.5.22).—Furfuraldehyde is treated with an inorganic acid (e.g., 0.2 pt. of concentrated sulphuric acid) to form a dense, hard product which is characterised by brilliant natural superficial finish and lustrous fracture.

R. BRIGHTMAN.

Manufacture of an artificial resin. I. G. FARBENIND A.-G., Assees. of W. PUNGS (G.P. 451,116, 30.8.22).—Oxidised paraffin, freed from hydrocarbons, is chlorinated in carbon tetrachloride at 70°. On removal of solvent, a hard, flexible, yellow resin (55% Cl) is obtained, which may be further hardened by heating at 110°.

C. HOLLINS.

Manufacture of benzene- and spirit-soluble polymerisation products of vinyl acetate, and of lacquers therefrom. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H., Assees. of W. PAEHNEL and W. O. HERRMANN (G.P. 446,562, 16.5.26).—The polymerisation of vinyl acetate by means of ultra-violet light is stopped as soon as the product begins to be insoluble in alcohol. The polymerisation may be performed with the aid of benzoyl peroxide in hot alcohol, and yields thus a viscous 50% alcoholic lacquer directly; a benzene lacquer is similarly prepared.

C. HOLLINS.

Manufacture of condensation products from urea, thiourea, or their derivatives, and an alcohol or a ketone. I. G. FARBENIND. A.-G. (B.P. 280,238, 7.11.27. Ger., 6.11.26. Addn. to B.P. 278,390; B., 1928, 648).—The resinous products of the prior patent are also obtained at 15–60° in presence of strong acid (concentrated sulphuric acid), an excess of alcohol or ketone being used.

C. HOLLINS.

Production of plastic masses from casein. G. JAKOVA-MERTURI (F.P. 623,650, 25.10.26).—Alkaline-earth oxides or carbonates are worked up to a pasty mixture with a solution containing casein, borax, and ammonia previously prepared at 100°, dyes being added if desired. The product is moulded into shape, dipped into magnesium chloride solution, dried, dipped into formaldehyde solution, and again dried, or, when required for the manufacture of gramophone records, sheets of the product are dipped into a solution containing borax and acetic acid.

L. A. COLES.

Insulating material (B.P. 299,906).—See XI. **Material from colophony** (G.P. 451,180).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Effect of repeated reclaiming of rubber. G. W. MILLER (Ind. Eng. Chem., 1928, 20, 1165–1169).—A sample of reclaimed rubber from whole motor covers was vulcanised with sulphur (1.9%) and a small

proportion of organic accelerator and then “reclaimed” by heating with 10% sodium hydroxide solution for 9 hrs. at 184.4°; these operations were then repeated thrice more. With repeated reclaiming the tensile strength after re-vulcanisation was little affected, but the material each time became less extensible and drier; the chloroform extract decreased from 22% to 5%, while the combined sulphur increased from 2.75% to 8.05%. When the successive grades of reclaimed material were incorporated in a vulcanisable mixture containing new rubber and carbon black as the other chief ingredients, the tensile strength decreased each time. The repeated reclaiming appeared to have little influence on the behaviour of this mixture towards accelerated ageing.

D. F. TWISS.

Comparison of scorching qualities of accelerators [for vulcanisation of rubber]. II. R. THIES (Ind. Eng. Chem., 1928, 20, 1223–1227).—The relative tendency of various accelerators to induce pre-vulcanisation is measured by comparing the opacity of the dispersions obtained when a sample disc, 3 mm. thick and 13 mm. diam., after storage at a definite temperature, is shaken with a circular motion with 50 c.c. of gasoline (*d* 0.70) for 1 min., giving 105 shakes. The storage temperatures recommended for the test are 99.5° representing milling temperatures, 66.5° representing a high storage temperature for milled stock, and the ordinary room temperature.

D. F. TWISS.

Heat transfer in the vulcanisation of rubber. T. K. SHERWOOD (Ind. Eng. Chem., 1928, 20, 1181–1185).—On account of the low thermal conductivity of rubber, non-uniformity of vulcanisation arising from “temperature lag” occurs with all but the thinnest articles. Curves are given illustrating the effect of increase in the thickness of the rubber layer on the observed course of rise of temperature in a motor cover during vulcanisation. From a knowledge of the temperature at different times during the heating process it is possible to calculate the relative vulcanisation effect at different points in a massive rubber article. Results are given demonstrating the advantages, in time consumption and in uniformity, gained by vulcanising pneumatic tyres on an inflated former filled with circulating superheated water instead of with air.

D. F. TWISS.

The accelerator [of vulcanisation] “Tuads” and its substitutes. W. ESCH (Kautschuk, 1928, 4, 255–258).—A discussion of the application of tetramethylthiuram disulphide (“Tuads”) as an accelerator of vulcanisation, including some reference to the relative efficacy of its ethyl and monosulphide analogues.

D. F. TWISS.

Ultra-accelerators [of vulcanisation of rubber]. A. D. CUMMINGS and H. E. SIMMONS (Ind. Eng. Chem., 1928, 20, 1173–1176).—Tetramethyl-, tetraethyl-, dimethyl-, diphenyldimethyl-, and dimethyldiethylthiuram disulphides were prepared by the action of alcoholic iodine (1 mol.) on an alcoholic solution of carbon disulphide (1 mol.) and the necessary amine (2 mols.); dimethyldiethylthiuram disulphide has m.p. 72°. The inactivity of dimethylthiuram disulphide

as an accelerator is attributed to its instability and rapid decomposition under the conditions of vulcanisation. The vulcanising power of tetramethylthiuram disulphide is substantially identical with that of a mixture of the monosulphide and sulphur in the equivalent proportions, thus indicating that the same accelerating substance, presumably zinc diethyl-dithiocarbamate, is produced by both. Diethylamine diethylmonothiocarbamate, obtained by the interaction of carbonyl sulphide and diethylamine, reacted with iodine giving an oily disulphide, and with zinc acetate yielding zinc diethylthiocarbamate; the latter has only feeble accelerating power. Comparative vulcanisation experiments with the thiuram disulphides and dithiocarbamates derived from dimethylamine and diethylamine, respectively, indicated that the ethyl compounds had somewhat greater acceleration activity.

D. F. TWISS.

Determination of sulphur in rubber by the perchloric acid method. E. WOLESENSKY (Ind. Eng. Chem., 1928, 20, 1234—1238; cf. Kahane, B., 1927, 532).—Kahane's method is improved in accuracy, experimental examples being given. The finely-divided sample (1 g.) in an 800-c.c. Kjeldahl flask is heated on a water-bath with 10 c.c. of 41% nitric acid until reaction subsides; concentrated nitric acid (10 c.c.) is then added, and heating continued for 15 min. until the rubber is dissolved. After adding 5 c.c. of a 60% solution of perchloric acid the inclined flask is heated until dense white fumes appear and the liquid is colourless. If free carbon is present a further addition of 3—5 c.c. of perchloric acid may be made or the carbon may be removed by filtration. After cooling, concentrated hydrochloric acid (5 c.c.) is slowly added and heating repeated until white fumes appear. Water (200 c.c.) is then added and the sulphate is precipitated hot with barium chloride. Any insoluble mineral matter may be removed before treatment with barium chloride and examined separately.

D. F. TWISS.

Determination of iodine value of raw rubber. A. GORGAS (Kautschuk, 1928, 4, 253—254).—The iodine value of rubber can be conveniently determined by using iodine bromide at the ordinary temperature; the reagent is prepared by dissolving 10 g. of iodine monobromide in 500 c.c. of carbon tetrachloride, and is stored in glass-stoppered bottles. The comminuted rubber (0.15 g.) is allowed to swell in carbon tetrachloride (50 c.c.). When sufficiently swollen (e.g., after 24 hrs.), 50 c.c. of the reagent are added with shaking. After 15 min. in the dark, 30 c.c. of 10% solution of potassium iodide are introduced, together with 100 c.c. of water, and the mixture is titrated with 0.1N-thiosulphate. Two independent tests are made concurrently, together with a blank test. D. F. TWISS.

Inaccuracies in determination of acidity of raw rubber by water extraction. A. D. CUMMINGS and H. E. SIMMONS (Ind. Eng. Chem., 1928, 20, 1213—1214).—Soxhlet extraction with water yields high results for the water-soluble acids present in rubber; the prolonged treatment probably leads to hydrolysis of esters in the rubber. It is better to treat the acetone-extracted

rubber with water on a water-bath until the aqueous extract shows no further increase in acidity.

D. F. TWISS.

Physical properties of rubber. M. RUHEMANN and F. SIMON (Z. physikal. Chem., 1928, 138, 1—20).—Determinations have been made of the sp. heats, thermal expansions, and X-ray interference figures of a series of samples of rubber at temperatures between -100° and 40° . Abrupt increases in sp. heat occur with smoked rubber sheet near -70° and near 0° , and with synthetic rubber near 0° . Less marked anomalies were found in the thermal expansion curves of some specimens of chilled rubber and of patent gums. These anomalies are attributed in part to a hardening process in one phase of the rubber, and in part to a change of state, probably from the solid to the liquid, of another phase.

F. G. TRYHORN.

Solubility of sulphur in rubber. H. LOEWEN (Kautschuk, 1928, 4, 243—249).—The phenomena of dissolution and crystallisation of sulphur in rubber are reviewed, and reproductions of microscopical enlargements are given in illustration of the appearance of the separated sulphur under different conditions.

D. F. TWISS.

Crystallised and light-sensitive gutta-percha. F. KIRCHHOFF (Kautschuk, 1928, 4, 254—255).—A solution of deresinised gutta-percha in carbon tetrachloride on evaporation yields a turbid film which under the microscope reveals distinct crystalline appearance; the turbidity disappears at $43-44^{\circ}$ and reappears on cooling, probably on account of the fusing point of crystals lying near this temperature. Crystals of gutta-percha can also be formed by slow evaporation of a solution in acetic acid. Such colourless gutta-percha on exposure to ultra-violet light becomes more opaque and brittle, although still completely soluble in benzene. As this change takes place equally well in an atmosphere of hydrogen it is presumably due to more complete transformation into the crystalline form.

D. F. TWISS.

PATENTS.

Preservation of india-rubber. RUBBER GROWERS' Assoc., Inc., G. MARTIN, and W. DAVEY (B.P. 299,585, 26.9.27).—A mixture of evaporated serum from rubber latex and alkali such as sodium hydroxide or carbonate (whether added before evaporation or subsequently), e.g., in the proportion of 3—5 pts. and $\frac{1}{2}$ —1 pt., respectively, to 100 pts. of rubber, has an anti-oxidant effect superior to that of serum solids alone and, unlike other anti-oxidants, does not lead to subsequent reduction in extensibility of the rubber.

D. F. TWISS.

Stabilising natural or artificial types of caoutchouc. SILESIA VER. CHEM. FABR. (B.P. 291,431, 30.3.28. Ger., 2.6.27).—Ageing of rubber, vulcanised or not, is retarded by incorporating reaction products of amines, e.g., aniline, with polyhydroxy-aldehydes such as dextrose.

D. F. TWISS.

Electrodeposition of rubber or homologous substances. W. A. WILLIAMS (B.P. 299,738, 30.6.27. Cf. B.P. 289,965; B., 1928, 494).—There is added to rubber latex, compounded or otherwise, an electrolyte, e.g., ammonium chloride, which will not readily cause

coagulation, but will enter into chemical action with the anode of two inserted electrodes and also act as the excitant of an internal electric current. The electrophoretic action of this current is assisted by applying to the electrodes an additional external source of current of voltage insufficient to decompose the added electrolyte, and such that the total electric potential shall be insufficient to effect electrolysis of water while causing deposition of rubber on the anode. D. F. TWISS.

Manufacture of articles from rubber or similar materials. DUNLOP RUBBER CO., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 299,974—5, 5.10.27).—(A) For the production of bulbous rubber articles, a former consisting of a rod bearing a bulbous enlargement of rubber with a roughened surface is dipped as many times as necessary in concentrated and/or compounded latex; after drying and vulcanising, the rod is removed, leaving the bulbous enlargement as part of the finished article. Between individual dippings in the latex the coated former may be immersed in a dehydrating and setting medium. (B) Articles with bulbous enlargements are made by dipping as in (A), but using smooth collapsible formers which are subsequently withdrawn from the formed articles. D. F. TWISS.

Production of shaped objects from aqueous dispersions of organic substances. DUNLOP RUBBER CO., LTD. From P. KLEIN and F. GABOR (B.P. 299,737, 28.6.27).—In the production of articles from dispersions such as rubber latex by dipping, formers are used which serve to produce the agglomerated deposit as well as to shape it. The formers, for instance, may be wholly or externally of alum, citric acid, acetic acid solidified with collodion, or of swollen or pasty masses containing coagulant substances. An aluminium former also may be used with alkaline latex, or a zinc former with latex containing ammonium sulphide. Such dipping formers may be provided with an outer semipermeable coat of inactive material. D. F. TWISS.

Rubber vulcanisation accelerator. D. H. POWERS, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,674,122, 19.6.28. Appl., 28.7.26).—The thioanhydrides, $(RO\cdot CS)_2S$, obtained by the action of cyanogen bromide or chloride on sodium or potassium alkylxanthates, are used, especially in presence of ammonia or amines (aniline), as vulcanisation accelerators. The butyl and ethyl compounds are specifically claimed. C. HOLLINS.

Rubber and resin compound. S. P. MILLER, ASSR. to BARRETT Co. (U.S.P. 1,682,397, 28.8.28. Appl., 25.11.24).—From 10% of crude unvulcanised rubber is dissolved in "cumar" resin, m.p. 125—128°, giving a waxy product, m.p. about 123°. R. BRIGHTMAN.

Treatment of gut or other strings or strands. DUNLOP RUBBER CO., LTD., H. WILLSHAW, S. N. GOODHALL, and C. FOLLISS (B.P. 299,535, 4.8.27).—In vulcanising strings or strands the heating fluid which is forced into the apparatus exerts a pressure which actuates devices maintaining a continual longitudinal pull on the strands. D. F. TWISS.

Securing the adhesion of bodies of dense, hard, or soft caoutchouc to other articles. H. BECKMANN

(B.P. 300,008, 8.11.27).—The rubber surface to be attached is first coated with a thin film of porous rubber which is vulcanised in position; this film is then caused to adhere to the desired articles by means of adhesives such as glue. Porous rubber of the type described in B.P. 240,430 (B., 1925, 1000) is very suitable.

D. F. TWISS.

Treatment of subdivided materials (B.P. 298,172).—See I.

XV.—LEATHER; GLUE.

PATENTS.

Production of leather. A. EHRENREICH (B.P. 299,599, 11.10.27).—The great intestine and the wall of the stomach of chondropterygii, plagiostomi, and other similar fishes are prepared, cleaned, and treated (a) for 2—4 days with a lime liquor containing 0.1—0.2% of carbolates and/or their derivatives, then with a pickle, and tanned with basic chrome liquors or with vegetable or other tanning materials to produce leather, or (b) felled with fish oil and sea salt to produce drum skins.

D. WOODROFFE.

Treatment of leather. A. O. T. BEARDMORE (B.P. 300,077, 3.4.28).—Vegetable-tanned sole leather hides, particularly those with grain blemishes, or other tanned skins are moistened so as to contain 20—25% of water, placed in a closed room at ordinary temperature and free from air currents, left to condition, sprayed on the surface with a mixture of mineral and vegetable oils at 60°, rough rolled, and finally compressed while still moist at 27—45° and under 12,000 lb./in.²

D. WOODROFFE.

Production of solid glue and gelatin in drop- or lens-shape. C. GREINER (B.P. 287,866, 17.12.27. Ger., 28.3.27).—Drops of liquid gelatin or glue are arranged to fall on to a conveyor, preferably an endless band of metal gauze, through which drying air may be passed, and conveyed through freezing and thawing zones, arranged alternately, then scraped off the band, conveyed to a drying chamber, and dried with hot air.

D. WOODROFFE.

Filler for acetylene containers (B.P. 299,681).—See II.

XVI.—AGRICULTURE.

Suggested nomenclature and classification of Dutch soils. D. J. HISSINK (Mem. Com. Internat. de Pedologie Helsingfors, 1924; Proc. Internat. Soc. Soil Sci., 1928, 3, 289—290).—The general classification of mineral soils into clays, loams, sands, and combinations of these is amplified by considerations of mechanical analysis, p_H values, and the contents of chalk and exchangeable calcium. Peat soils are classified primarily as high-moor, low-moor, and transition soils, and are further characterised by the contents of organic matter and lime as well as of the nitrogen and phosphorus of the organic material. Mixed soils are classified according to their content of clay, sand, humus, chalk, exchangeable calcium, and p_H value. A. G. POLLARD.

Soil acidity. C. R. RUNK (Delaware Agric. Exp. Sta. Bull., 1927, No. 152, 6—7).—Delaware soils have a wide range of acidity. A p_H value of 5.4—6.5 is optimal for lucerne. Experiments on lime requirement and

the effect of continued fertiliser treatment on reaction are described.

CHEMICAL ABSTRACTS.

Significance of the determination of soil acidity in agricultural practice. D. J. HISSINK (Rev. Internat. Rens. Agric. N.S., 1924, 2, 827—830; Proc. Internat. Soc. Soil Sci., 1928, 3, 274).—Comber's method for determining soil acidity gives results in general accord with direct measurements of hydrogen-ion concentration, and if the colour measurements are previously standardised by means of the hydrogen electrode the method is suitable for general use among farmers.

A. G. POLLARD.

Important physical quality of a heavy soil in natural deposits. KÖTTGEN and KLITSCH (Forstwiss. Cent., 1927, 705—720; Proc. Internat. Soc. Soil Sci., 1928, 3, 252).—Mechanical analysis of soils fails to give a true representation of the true state of aggregation of soils and subsoils. Measurements of water percolation are indicative of soil pore-space and afford a single-value determination of soil structure. Changes in structure resulting from alterations in soil reaction are of practical importance, and are not detectable by the customary sedimentation processes.

A. G. POLLARD.

Influence of various forest types on nitrate content and formation in forest soils. A NĚMEC and K. KVAPIL (Z. f. Forst- u. Jagdwesen, 1927, 322—412; Bied. Zentr., 1928, 57, 487—489).—The nitrate content of different soil horizons under various types of forest was studied, and nitrifying ability correlated with the type of vegetation. Nitrifying ability was determined from the rate of accumulation of nitrate in samples of soil. Nitrate content varied with the depth of the horizon, surface litter and mould being generally richest. In both coniferous and deciduous forests free from undergrowth there was little or no nitrification at any horizon, and even a tendency to denitrification when the surface layer was highly acid. The existence of an undergrowth was accompanied by considerable nitrifying power in the humus layers; certain plants were suggested as indicators of nitrifying ability in forest soils. In clearings there was a striking increase in nitrifying ability, attributed to the accelerating action of direct sunlight on the decomposition of litter material.

H. L. RICHARDSON.

Utilisation of phosphates by rye seedlings in different soils. S. GERICKE (Illustr. Landw. Ztg., 1927, 47, 396; Proc. Internat. Soc. Soil Sci., 1928, 3, 276—277).—In general, the smallest proportional utilisation of phosphate occurred in soils of low phosphorus content, the mean values for marsh soils being 4.1%, sandy soils 7.5%, and humus sands 14.0%.

A. G. POLLARD.

Influence of various fertilisers on the manurial action of "insoluble" phosphate. A. RINDELL (Nordisk Jordbruksforskning, 1921—2, 3—4, 1; Bied. Zentr., 1928, 57, 491—492).—In pot experiments other salts increased or decreased the manurial action of difficultly soluble phosphate in accordance with their effect on solubility, as predicted by physico-chemical considerations. In the field there was no positive or negative effect attributable to solubility, but some agreement between the manurial action of phosphates and the

changes in reaction of the soil solution produced by long-continued application of physiologically acid or basic fertilisers.

H. L. RICHARDSON.

Determination of phosphoric acid requirements of field soils. G. HAHN (Botan. Arch., 1927, 20, 223; Bied. Zentr., 1928, 57, 490—491).—The phosphoric acid requirements of 30 soils were determined by Lemmermann's (relative solubility) and Neubauer's (seedling) methods and by treatment with carbonic acid. Neubauer's method gave results that did not agree with those obtained by the other two. The author concludes that no method gives trustworthy numerical values, and that, until the seedling method is further improved, Lemmermann's is a simpler and quicker way of getting an idea of the phosphoric acid requirement of a soil.

H. L. RICHARDSON.

Determination of phosphates [in soil extracts] by the phosphomolybdate method. O. ARRHENIUS (Arch. Suikerind. Ned. Indië, 1927, 2, 903; Proc. Internat. Soc. Soil Sci., 1928, 3, 247).—10 c.c. of a 2% citric acid extract of soil are diluted to about 80 c.c. To this are added 1 c.c. of concentrated sulphuric acid, 5 c.c. of ammonium molybdate solution (25 g. of ammonium molybdate dissolved in 300 c.c. of water to which are added 25 c.c. of concentrated sulphuric acid diluted with water to 200 c.c.), 1 c.c. of sodium sulphite solution (20 g. of sodium sulphite in 80 c.c. of water), and 1 c.c. of quinol solution (0.5 g. per 100 c.c., plus 1 drop of sulphuric acid). The whole is diluted to 100 c.c. and after 24 hrs. the colour is compared with a series of standards containing 0.03—0.003 mg. P_2O_5 per 100 c.c.

A. G. POLLARD.

Action of alkali chloride on plants and soils. C. DUPONT (Ann. Sci. Agron. franç. et étrang., 1924, 41, 369; Bied. Zentr., 1928, 57, 492—493).—A discussion of the action of added chlorides on alkali soil with especial reference to the use of sylvinite. The interaction of the potassium chloride in the sylvinite with soil calcium is said to lessen the harmful action of the sodium chloride it contains, but harm may result if the soil is poor in lime and is treated with an excessive amount of sylvinite. Certain precautions necessary in its application are mentioned.

H. L. RICHARDSON.

Sodium salts, used in conjunction with potash, as a plant food. H. HEINRICH (Z. Pflanz. Düng., 12A, 252; cf. B., 1928, 343).—A correction.

A. G. POLLARD.

Influence on the development of plants of zinc and lead precipitated on the soil from factory fumes. H. LUNGEGÅRDH (Aveldein. f. Landbruksbotanik, 1927, No. 42; Proc. Internat. Soc. Soil Sci., 1928, 3, 272).—No toxicity resulted from the presence of zinc in soils at lower concentrations than 0.027%. Germination tests with oats, wheat, and clover in nutrient solutions containing zinc sulphate at concentrations between $N/300,000$ and $N/60,000$ showed in some instances a weak stimulation and in others a slight checking of germination. Oats germinated normally in $N/3000$ zinc sulphate solutions. Growth of plants in culture solutions showed slight injury at concentrations of $N/500,000$ for zinc and $N/5,000,000$ for lead. These metals are assumed to form nearly insoluble compounds in soil.

A. G. POLLARD.

Influence of the factor "soil" in cultural and manurial experiments. R. STORP (Diss. Danzig, 1928; Proc. Internat. Soc. Soil Sci., 1928, 3, 286).—The value of examining soil profiles in field experiments is emphasised. Conditions favourable to plant growth are: high humus content and considerable depth of the A-horizon; compact formation in the B- and C-horizons; ample humus, subsoil water, and capillary water in B- and C-horizons. A. G. POLLARD.

Effect of manurial constituents on the quality of sugar-cane juice and gur. P. B. SANYAL (Agric. J. India, 1928, 23, 277—286).—Juices and gurs of the highest purity and sucrose content were obtained (in the case of cane variety Co. 213) when superphosphate and potassium sulphate had been used as manure; organic nitrogen (as mustard cake) gave intermediate results, whilst cyanamide and ammonium sulphate gave the lowest results of all. A superior quality of gur was produced from juices clarified by Horne's "super-defecation" process (B., 1924, 685). J. P. OGILVIE.

Nitrogen question in the sugar industry of Java. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, No. 3; Proc. Internat. Soc. Soil Sci., 1928, 3, 284—285).—From sand-culture experiments it is calculated that the nitrogen requirement of sugar cane is 620—800 lb. per acre. Nitrogen assimilation increases with the nitrogen concentration of the nutrient solution. It is probable that the cane can utilise ammoniacal nitrogen. Cane soils in Java contain little or no nitrite even when waterlogged. The p_H value of these soils has no influence on nitrate production. A. G. POLLARD.

Influence of concentrations of chlorine on development of the sugar cane. O. ARRHENIUS (Arch. Suikerind. Ned. Indië, 1928, 1, 90; Proc. Internat. Soc. Soil Sci., 1928, 3, 284).—Sugar-cane plants were grown in sand culture with nutrient solutions containing chloride. Injury was observed with concentrations of more than 0.060% of chlorine. Remedial measures consist essentially of leaching. A. G. POLLARD.

Relation between different properties of sugar-cane soils. O. ARRHENIUS (Arch. Suikerind. Ned. Indië, 1927, 2, 1197; Proc. Internat. Soc. Soil Sci., 1928, 3, 285).—A relationship between soil acidity and phosphate content is attributed to the different solubilities of mono-, di-, and tri-phosphates. The phosphate content of the soil influences nitrification. A. G. POLLARD.

Soil acidity and sugar cane. O. ARRHENIUS (Arch. Suikerind. Ned. Indië, 1927, 2, 1027; Proc. Internat. Soc. Soil Sci., 1928, 3, 285).—Optimum yields of cane are produced on neutral soils. A. G. POLLARD.

Noxious effect of molasses on soil. O. ARRHENIUS (Arch. Suikerind. Ned. Indië, 1927, 2, 791; Proc. Internat. Soc. Soil Sci., 1928, 3, 284).—Injury to *Amarantus tricolor*, L., soon after the application of molasses to the soil was observed. The period of injury was shorter in sandy soils than in clays, and is attributed to the withdrawal of nitrate from the soil by organisms fermenting the molasses. A. G. POLLARD.

Red- and sheep's fescue. K. BÄR (J. Landw., 1928, 76, 255—284).—The dependence on external conditions of the growth, form, colour, and general botanical char-

acteristics of the fescues is examined. The digestibility of the protein of the stems of the grasses is less than that of the panicles, but in all cases is independent of the total protein content. A. G. POLLARD.

Vitamin content of silo juices. F. DUSCHEK (J. Landw., 1928, 76, 197—216).—The vitamin content of the juice from Serradella silage was demonstrated by the live weight increases and improved conditions of health of rats previously fed on a diet deficient in vitamins. With the freshly-pressed juice from maize and red clover silage under similar conditions no beneficial effects were observed in growth and development, but health conditions were improved. Neither effect was apparent when juices from cold-pressed silos were used. Losses of valuable food material in electrical ensilage practice can be avoided by the incorporation of the juices with absorbent material which can subsequently be fed to cattle. A. G. POLLARD.

Insecticides. PEET.—See XXIII.

PATENTS.

Polyhydroxy-[organo]-mercury compound. H. GÜNZLER and O. NEUBERT, Asssts. to WINTHROP CHEM. CO., INC. (U.S.P. 1,677,823, 17.7.28. Appl., 5.5.27. Ger., 20.1.26).—Colourless, odourless, stable products having insecticidal and fungicidal properties are obtained by dissolving in water a mixture of a polyhydroxybenzene (resorcinol, 2:6-dihydroxytoluene, pyrogallol) and mercuric chloride. C. HOLLINS.

Mixed crystals containing potassium and ammonium (B.P. 299,984). **Fertiliser** (U.S.P. 1,686,873).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Frothing of [sugar] juice during carbonatation in the beet sugar factory. V. STANĚK and J. VONDRÁK (Z. Zuckerind. Czechoslov., 1928, 53, 81—98).—Various vegetable and mineral oils were found to exert a varying effect on the abatement of froth in a miniature carbonatation apparatus designed closely to imitate the conditions of practice. It was shown that the optimum effect is obtained with such substances in extremely small amount, viz., only 0.2 mg. per litre of juice. Rape oil in the form of emulsion (0.1—5%) gave the same result as its direct application. J. P. OGILVIE.

Final saturation of [sugar] thin-juice. O. SPENGLER and C. BRENDL (Z. Ver. deut. Zuckerind., 1928, 637—643).—To obtain juice low in calcium the second carbonatation is carried out so as to saturate the natural alkalinity, after which follow successively heating, filtration, sulphitation almost to neutrality at high temperature, and a second filtration. At this stage sodium carbonate is added in amount corresponding to the content in calcium present, the juice being lastly boiled and filtered. If at the syrup stage the alkalinity is too high it can be reduced by sulphiting. J. P. OGILVIE.

Automatic control of the carbonatation process of beet-sugar manufacture. R. T. BALCH and J. C. KEANE (Ind. Eng. Chem., 1928, 20, 1148—1151).—In automatically controlling second carbonatation, use was made of (1) a standard saturated calomel half-cell, contact with the juice being made through a porous

cup filled with potassium chloride, and (2) a bare tungsten wire electrode. The electrodes were suitably arranged in a glass flow-chamber through which a continuous sample of the juice was passed. A temperature compensator, the purpose of which was to correct automatically for the changes in p_H caused by temperature fluctuations, was also inserted in the flow-chamber. A potentiometer was in combination with a device for controlling the reversing motor operating the gas valve of the tank, this being gradually opened when the reaction of the juice was above the selected p_H and gradually closed when falling below. Given means for delivering sulphur dioxide to a tank under as constant conditions as in the carbonation system, sulphitation could be subjected to a similar automatic control.

J. P. OGILVIE.

Incrustations in the carbon dioxide piping [of beet sugar factories]. O. SPENGLER and C. BRENDSEL (Z. Ver. deut. Zuckerind., 1928, 644—645).—Analysis of a greyish incrustation taken from the valves of the carbon dioxide pumps in two beet factories gave, respectively, organic matter 14.5, 24.4; sand and insoluble 0.2, 0.6; iron 2.5, 1.1; copper 0.6, none; potassium and sodium 35.0, 30.9; chlorine 38.3, 33.3; and SO_4 5.1, 6.3%.

J. P. OGILVIE.

Determination of solubility of sucrose in beet-house syrups. R. J. BROWN, J. E. SHARP, and H. W. DAHLBERG (Ind. Eng. Chem., 1928, 20, 1230—1232).—Using a low-purity beet molasses of known composition, various solutions of known impurity-water ratios were prepared, these being saturated with sucrose at the proper temperature without any loss of water. Then the purity and sucrose contents were calculated from their dry substance values, and the results plotted. A table is given showing the concentration of saturated beet syrups from 40° to 80°, and with impurity-water ratios from 0 to 1.378.

J. P. OGILVIE.

Cane sugar manufacture in Java. Centrifugal treatment of syrups. P. HONG and W. F. ALEWYN (Arch. Suikerind. Neder-Indië, 1928, 36, 330—370).—Experiments were made to ascertain at which stage centrifugal clarification could be applied, a De Laval machine (No. 300), 16.5 cm., being used. It was concluded not to be practicable to work with the juice, raw or clarified, as later, owing to evaporation, a further precipitate is formed. Good results, however, were obtained at the syrup stage, solids to the amount of 15 g./litre being separated. Molasses was also treated to advantage, and also the wash-liquor from the white-sugar centrifugals.

J. P. OGILVIE.

Application of clarification after instead of before evaporation. B. J. VAN SANTEN (Arch. Suikerind. Neder-Indië, 1928, 36, 579—581).—Unclassified sugar juice was evaporated to syrup (62° Brix) and clarified in this concentrated state. If generally practicable on the large scale, this procedure would be preferable to the present procedure, since working would be more rapid, scaling in the evaporators would probably be avoided, and a better product by crystallisation from a clearer syrup might result.

J. P. OGILVIE.

p_H value of sugar media and their power of

inversion. H. COLIN and A. CHAUDUN (Bull. Assoc. Chim. Sucr., 1928, 45, 626—642).—A lecture. The chief methods of determining the p_H of media are mentioned and its importance in sugar manufacture is discussed. Although the values form a useful guide, simple determination of p_H does not supply a solution to the difficulties of the manufacturer since the complex phenomena forming the basis of sugar technique do not depend exclusively on hydrogen-ion concentration. The optimum values in various stages of manufacture vary from week to week since the different batches of raw material (sugar beet) are not uniform. The quantities of reducing sugars formed during the heating of solutions are unimportant so long as the p_H does not fall below 6.5; after this they increase rapidly.

H. INGLESON.

Inversion of sucrose by weak acids. V. ZATFOUK (Z. Zuckerind. Czechoslov., 1928, 53, 33—42).—Under like conditions regarding molar concentration, temperature (70°), and hydrogen-ion concentration, aspartic acid hydrolyses sucrose more rapidly than does succinic acid. Glutamic acid, on the other hand, has the same rate as succinic acid at p_H 4.5, but this increases with rising p_H , until at p_H 5.1 it is the same as in the case of aspartic acid. At a still higher p_H , glutamic acid inverts more rapidly than aspartic acid. At p_H 5.5 succinic acid inverts insignificantly, and at p_H 5.7 aspartic acid also practically ceases to hydrolyse.

J. P. OGILVIE.

Chemical composition and physical properties of beet molasses. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1928, 53, 1—11).—Average figures for beet molasses of the 1925—6 campaign in Czechoslovakia, France, and Germany, respectively, are: dry substance (refractometric) 78.5, 78.3, 76.9; direct polarisation 51.26, 50.45, 50.40; apparent quotient of purity 65.35, 64.50, 65.50; ash 10.14, 8.69, 8.27; total non-sugars 30.19, 27.85, 28.14; and nitrogen 1.64, 1.63, 1.44%.

J. P. OGILVIE.

Testing decolorising media. LINSBAUER and VAŠÁTKO.—See II. **Effect of manures on sugar cane.** SANYAL. **Sugar industry of Java. Development of sugar cane.** **Sugar-cane soils.** ARRHENIUS.—See XVI.

PATENTS.

Oxidation of aldoses. CHEM. FABR. VORM. SANDOZ (B.P. 293,322, 2.7.28. Ger., 2.7.27. Addn. to B.P. 289,280; B., 1928, 461).—The cooling necessary in the process of the prior patent is avoided by treating dextrose in aqueous solution containing a little sodium carbonate and sodium bromide at 50—55° with chlorine, calcium carbonate being added from time to time to maintain alkalinity; calcium glutaconate crystallises out. Lactose similarly gives calcium lactobionate.

C. HOLLINS.

Automatic apparatus for the continuous production of extracts, particularly of sugar contained in sugar plants. SOC. ANON. DES ÉTABL. A. OLIER (B.P. 286,668, 29.2.28. Fr., 8.3.27).

Extraction of solid materials (B.P. 298,501).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Brewers' pitch [for lining casks]. F. KUTTER (Woch. Brau., 1928, 45, 473—478, 483—487, 493—497, 503—506, 517—521).—The Klinger-Lampe and Hoffmann-Herbst methods, and that depending on observing the fall of a ball through a disc of the heated material, all yield reproducible results for softening and melting points, but do not agree among themselves. A simplification of the Hoffmann-Herbst method, in which the rate of sinking of a brass rod through the heated material is measured, is recommended. The viscosity should increase slowly and uniformly as the liquid cools, and solidification should be gradual. A high coefficient of expansion favours the formation of cracks. In these respects rosin pitch is superior to paraffin. Compositions were intermediate in behaviour and showed no appreciable difference from rosin pitch when roughly tested as to their behaviour under impact. Rosin oil lowers the flash point, and both it and paraffin increase the proportion of neutral substances extractable by ether from an alkaline emulsion. The "oxidation value," (c.c. of *N*-permanganate reduced by 1 g. of the material) is best determined under standardised conditions on a solution in glacial acetic acid, the precipitated manganese oxides being dissolved by hydrogen peroxide, the excess of which is titrated with *N*-permanganate. In applying the evaporation test (cf. Brand, B., 1900, 761) the loss, though greater at first, soon becomes proportional to the time, and 5 hrs.' heating gives a sufficient indication of the changes which occur. The loss of weight and rise of m.p. are generally greater and the increase of viscosity is less in rosin pitches than in compositions containing a reasonable proportion of paraffin. An aeration test is described in which 400 g. of the material are placed in a covered nickel beaker, 8 cm. wide and 10 cm. high, which slides into a similar but slightly wider beaker heated in a paraffin bath. The cover carries a thermometer and an air inlet tube, and is provided with a hole of 1 cm.² area as an outlet for air and vapour. The tube is 6 mm. in diam., and slightly tapered at the end, which is placed 1 cm. from the bottom of the beaker. Air is passed continuously during heating, at 9—10 litres/hr. It is claimed that the changes undergone by a composition during this treatment are a good indication of those occurring in practice, whilst those which take place during heating without aeration have no comparative value.

F. E. DAY.

Defective attenuation of the mash. E. LÜHDER and B. LAMPE (Z. Spiritusind., 1928, 51, 335—336).—Many worts from mashes made with malt and potatoes show a low final percentage of attenuation at the conclusion of the fermentation. This is chiefly due to a deficiency in the content of amylase in the malt, and can be alleviated by the addition to the mash of a relatively greater quantity of malt. The deficiency in attenuative properties may also be brought about if the mash is made of too high a concentration, whilst, among other contributory causes, is the use of a yeast infected with bacteria. The presence of the bacteria results in the excessive production of acidity, which tends to inhibit the action of the amylase and to weaken the yeast.

C. RANKEN.

Conditions for the aeration of fermenting vats in the yeast industry or for the aeration of liquids in general. E. G. STICH (Chem.-Ztg., 1928, 52, 865—866).—The efficient aeration of liquids is discussed. Important factors are the ratio of the total area of the air passages in the aeration apparatus to that of the vat floor, and also of the total surface area of the bubbles to the required air content of the liquid.

F. R. ENNOS.

Clarification of natural sweet wines by centrifuging. J. DUBAQUIÉ (Ann. Falsif., 1928, 21, 460—463).—Although rapid and satisfactory clarification of Sauterne wines by centrifuging was obtained the effect was not perfect, and at the end of a year control samples were equally clear. The violent mechanical action appears to affect the colloidal condition of the product, certain chemical precipitations are accentuated, particularly that of cream of tartar, and the degree of flocculation of various ingredients is altered. The use of the centrifuge, at least for the more delicate wines, is not recommended.

D. G. HEWER.

Determination of lævulose and dextrose in sweet wines. F. LUCIUS (Pharm. Zentr., 1928, 69, 725—727).—Comparison is made of results obtained by the usual methods applied to various wines with those obtained by calculation from the rotations before and after inversion.

S. I. LEVY.

Effect of successive generations of yeast on the alcoholic fermentation of cider. S. C. VANDECAVEYE (J. Agric. Res., 1928, 37, 43—54).—The problem of preparing unfermentable fruit juices which can be sold as non-alcoholic beverages is one of some moment in America. By coagulating freshly pressed cider at 45° and growing three successive generations of yeast in the liquid after removing the coagulum it is possible to obtain a temporarily unfermentable liquor possessing the unchanged taste and palatability of cider and containing 0.53% of alcohol. The inhibition of fermentation is due to the removal of the nitrogen and phosphorus.

H. J. G. HINES.

Sauerkraut. PREUSS and others.—See XIX.
Pharmaceutical preparations. PLACERES.—See XX.

PATENT.

Manufacture of yeast. A. J. C. OLSEN, and INTERNATIONAL YEAST Co., LTD. (B.P. 299,336, 21.6.27).—In the process three or more fermenters communicating in series are used. In each of the fermenters following the first which are traversed by the incompletely treated or unripe yeast-containing liquid, the environment of the yeast is controlled by the continuous addition of yeast nutrients, with or without added seed yeast, so as to maintain constancy of quality of the final yeast, which is withdrawn from the final fermenter at a rate sufficient to prevent the intermediate fermenters from becoming overfilled. Spare fermenters may be used to short-circuit any of the series of fermenters for the purpose of cleansing.

C. RANKEN.

XIX.—FOODS.

Determination of fat in cocoa products. A. HEIDUSCHKA and F. MUTH (Chem.-Ztg., 1928, 52, 879).—An apparatus suitable for very rapid, accurate determinations at laboratory temperature consists of a Schott

glass filter-crucible which, after weighing with the material, is secured by means of a rubber band in an adapter carrying a ground-glass joint-piece, by which it is fitted to a flask which has a side tube for attachment to a pump during the extraction and to a condenser during removal of the solvent. S. I. LEVY.

Gas production in the making of sauerkraut. L. M. PREUSS, W. H. PETERSON, and E. B. FRED (Ind. Eng. Chem., 1928, 20, 1187—1190).—The gas evolved during the formation of sauerkraut is almost 100% carbon dioxide, and most of the gas formed is given off within 40—160 hrs. after the cabbage is packed into the container. At higher temperatures gas and acid production increase together with a rapid increase in the number of bacteria, and the conclusion is reached that the gas production is due to bacterial activity and not to yeast growth or plant-cell respiration. Washing the raw cabbage before cutting and packing evidently results in a better flavoured sauerkraut. E. H. SHARPLES.

Iron and manganese content of foodstuffs. J. T. SKINNER and W. H. PETERSON (J. Biol. Chem., 1928, 79, 679—687).—Figures are given for the iron and manganese content of a number of (cattle) foodstuffs; in general, the content of iron is about three times that of manganese. C. R. HARTINGTON.

Nutritive value of hardened oils. UENO and others.—See XII. **Vitamin content of silo juices.** DUSCHEK. **Red- and sheep's fescue.** BÄR.—See XVI.

PATENTS.

Recovery of alcohol [from the vapours from bake-house ovens etc.]. N. NAVROTZKY (F.P. 672,889, 22.1.27).—The gases and vapours from ovens containing fermented dough etc. are passed through cooling coils arranged to condense alcohol and water separately, the water being returned to the ovens. The withdrawal of the gases and vapours from the ovens and the supply of water to the coolers are regulated automatically. L. A. COLES.

Preventing the blackening of tinned foods. K. OSHIMA (U.S.P. 1,686,393, 2.10.28. Appl., 19.2.27. Jap., 1.3.26).—An acidity up to p_H 4 is maintained by adding a mixture of a weak organic acid and salt thereof, e.g., a mixture of acetic acid and sodium acetate, to the food before canning. Sodium chloride is also preferably added. F. G. CLARKE.

Margarine composition. J. C. SHERMAN, ASST. to BROWN Co. (U.S.P. 1,691,087, 13.11.28. Appl., 9.12.22).—See Can.P. 259,391; B., 1927, 265.

Drying machines (B.P. 299,134).—See I.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Preparation and stability of tincture of iodine. P. BOHRISCH (Apoth.-Ztg., 1928, 43, 436—440; Chem. Zentr., 1928, i, 2514—2515).—The directions of the German pharmacopœia (6th ed.) for the determination of potassium iodide and iodine are criticised. The stability of the tincture is unaffected by the substitution of potassium iodide by bromide, or by the use of 70% alcohol. A. A. ELDRIDGE.

Mercuric iodide determination in tablets. H. O. MORAW (J. Amer. Pharm. Assoc., 1928, 17, 1084—1086).

—A weighed quantity of the finely-powdered sample equivalent to 0.25—0.40 g. of mercuric iodide is well mixed with about 0.2 g. of lactose, and the mixture made into a smooth paste with 2—5 c.c. of 10% sodium hydroxide solution and then an additional quantity is added to make a total of 30 c.c. About 40 c.c. of water are added, and after digesting gently over a low flame for 30—50 min. the mixture is filtered and the residue washed. 5 c.c. of 10% silver nitrate solution are added to the filtrate and then 25 c.c. of concentrated nitric acid. The mixture is gently boiled until the liquid becomes clear and the silver iodide is collected on a filter and weighed. Intimate mixing with the lactose is necessary to ensure complete reduction of the mercuric iodide and to prevent losses due to occlusion and incomplete peptisation. The limits of accuracy of the method are within 1%. E. H. SHARPLES.

Determination of chloral in syrup of chloral. P. ANDRON (J. Pharm. Chim., 1928, [viii], 8, 453—455).—A reply to the statements of François (B., 1928, 243), which are shown to be incorrect. E. H. SHARPLES.

Detection of lead and copper in citric acid, tartaric acid, and extracts. G. FRERICHS (Apoth.-Ztg., 1928, 43, 513—514; Chem. Zentr., 1928, i, 2850—2851).—The methods of the German pharmacopœia, 6th ed., are criticised. A. A. ELDRIDGE.

Pharmaceutical preparations containing lactic acid organisms. J. PLACERES (Anal. oficina quim. prov. La Plata, 1927, 1, 71—89).—When incubated at 37° for 48 hrs. with 100 c.c. of 3% lactose milk medium, the fluid preparations (1 c.c.) produced larger amounts (11.70—20.61 g./litre) of lactic acid than the solid preparations (0.5 g.). Formic acid (0.014—0.081%) and acetic acid (0.123—0.945%) were also formed, but butyric acid, aldehydes, and acetone were not observed. CHEMICAL ABSTRACTS.

Sodium phenylethylbarbiturate. H. SANDQVIST and T. II. LINDSTRÖM (Arch. Pharm., 1928, 266, 613—616).—Commercial samples of sodium phenylethylbarbiturate have been found to contain water and as much as 5% of alcohol, which is removed only by long heating at 140—150°. The pure substance gives a red colour with phenolphthalein but not with thymolphthalein, although the latter is coloured blue by many commercial products. The organic matter in this compound is satisfactorily determined by acidification, evaporation to dryness, and extraction with ether. The results obtained by extracting an acidified, aqueous solution are not reliable. S. COFFEY.

Chemical and pharmacological examination of the leaves of *Atropa belladonna*, L., and of the extract prepared from them. T. EXLER (Pharm. Weekblad, 1928, 65, 1152—1177).—Examination of the leaves by the methods of the German, United States, Swiss, and Dutch Pharmacopœias gave for alkaloid content the values 0.86, 1.13; 0.64, 0.62; 0.54, 0.56; and 0.52, 0.52%, respectively. Using the same methods, but with increased amounts of solvent for extraction, the German method gave 0.92, 0.74%; the others gave values very close to those obtained previously. Errors in the German method are pointed out. The worst error arises from the method of distilling the

ether solution before titrating, in which the solution is evaporated to one third of its bulk; ammonia and other volatile alkaline substances remain behind. If the solution is evaporated to dryness and the residue twice damped and heated, concordant and accurate results are obtained. The American method is also criticised. The methods of preparing the extract are discussed, and the differences in pharmacological properties observed are traced to racemisation and hydrolysis of the alkaloids during extraction. Gravimetric determination of the alkaloids by the silicotungstate method gives results always lower than the titration method. Pharmacological tests were carried out quantitatively by determining the amount of alkaloid necessary to counterbalance the contraction caused by pilocarpine in the intestine of the cat and rabbit. Tropine has no effect; 2 pts. of hyosecyamine have an effect greater than that of 3 pts. but less than that of 4 pts. of atropine. Hydrolysis of the alkaloids, which produces tropine, therefore lowers the pharmacological activity as well as the rotatory power.

S. I. LEVY.

Fluid extracts. II. Fluid extract of *Hydrastis canadensis*. A. ALBANESE and A. PEDRONI (Annali Chim. Appl., 1928, 18, 429—447).—The qualitative reactions, identification of the active principles, and the quantitative examination of liquid extract of *Hydrastis canadensis* are discussed, and modifications in the details of certain analytical procedures suggested. (Cf. Belloni, B., 1919, 438 A.)

T. H. POPE.

Stability of homatropine hydrobromide solution. P. S. PITTENGER and J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1928, 17, 1081—1084).—Solutions of homatropine hydrobromide of varying concentrations have been examined physically and physiologically after storage under different conditions and exposure to ultra-violet light. Sterilisation or exposure to ultra-violet radiation has no apparent deleterious effect on the mydriatic action of homatropine hydrobromide either in distilled water or in sodium chloride solution isotonic with the tear. Similar solutions preserved in ampules for 9 months showed no loss in mydriatic action.

E. H. SHARPLES.

Technical isolation of scopolamine. F. CHEMNIERUS (J. pr. Chem., 1928, [ii], 120, 221—224).—The isolation, on the technical scale, of scopolamine from the roots of various *Solanaceae* by hydrolysis with 10% sodium hydroxide solution, extraction with ether from which the base is removed with acetic acid, isolation with potassium carbonate, and purification through its hydrobromide, is described.

J. W. BAKER.

Keeping properties of opium powder. A. JERMSTAD (Pharm. Zentr., 1928, 69, 693—694).—Samples of opium powder preserved in dry, well-closed bottles for 10 years showed diminutions of morphine content of only 0.32—0.38% of the actual content. S. I. LEVY.

Denicotinised tobacco. E. M. BAILEY, O. L. NOLAN, and W. T. MATHIS (Conn. Agric. Exp. Sta. Bull., 1928, No. 295, 338—351).—“Denicotinised” tobacco varies considerably in nicotine content. Some denicotinised tobaccos contained as much nicotine as is likely to be present in ordinary tobacco. The smallest nicotine content was 0.75%; ordinary tobacco may contain

as little as 1%. Nitrate- and ammonia-nitrogen are present to the same extent in both cases.

CHEMICAL ABSTRACTS.

Seed oil of *Citrus limomum*. F. OCCHIPINTI (Atti II Cong. Naz. Chim. Pura Appl., 1926, 945—950; Chem. Zentr., 1928, i, 2884).—The seed oil of *Citrus limomum* has d_{4}^{15} 0.922, f.p. —6°, n_D^{20} 70.8, Tortelli value 83°, acid value 21.1, saponif. value 194.6, ester value 173.5, iodine value 103.5, Hehner value 94.5, Reichert-Meissl value 0.5, unsaponifiable matter 0.5, drying value (Livache) 5%. The fatty acids (25% solid) have m.p. 41.5°, acid value 202.0, acetyl value 33.0, iodine value 114.0, mol. wt. 277.7. The solid acids consist of stearic (30%) and palmitic (70%), and the liquid acids of oleic and linoleic acids.

A. A. ELDRIDGE.

Oil of *Salvia sclarea*. E. KOPP (Pharm. Zentr., 1928, 69, 677—680).—The oil should be prepared from the peduncles, the leaves containing very little; peduncles from Rumanian plants yielded 0.15% of oil. Tables of the physical and chemical properties of oils from various sources are given, and show very wide variations. The Rumanian oil contains 41.8—59.2% of linalyl acetate and 13.8—26.5% of the free alcohol.

S. I. LEVY.

Vibration theory of odour. G. M. DYSON (Perf. Ess. Oil Rec., 1928, 19, 456—459).—The relationship between vapour pressure and odour is discussed and the following theory for the sensation of odour is propounded. The odorous molecule forms a loose absorption system with the protein molecules of the osmic cells and absorbs radiant heat energy. This causes displacement of one or more electrons from their normal orbit. When the electrons revert to their normal positions the energy released as wave motion of a definite frequency is absorbed by the sensory apparatus and leaves the system as a nervous impulse. A scale of frequencies may be associated with osmic perception, each frequency being associated with a definite osmic sensation. An odour may be simple and depend on the emission of a wave motion of definite frequency, or it may be complex and arise from a series of waves of different frequencies analogous to simple and compound colours and pure tones and chords. The improvement in odour of certain unpleasant substances on extreme dilution is probably due to the “critical threshold concentration,” or the limiting concentration at which osmic frequency can be physiologically detected, differing with the frequency, and the “pleasant” frequency is still observable at a dilution where the “unpleasant” frequency has ceased to be detectable.

E. H. SHARPLES.

Cresol-naphthene soap solutions. KOGAN.—See XII.

PATENTS.

Manufacture of chloral. SOC. D'ÉLECTROCHIM., D'ÉLECTROMÉTALLURGIE, ET DES ACIÉRIES ÉLECTRIQUES D'UGINE (F.P. 612,396, 27.6.25).—Chlorine is passed briskly into a mixture, cooled at first, of 97% alcohol and acetaldehyde, which is eventually boiled. Chloral alkoxide is formed by way of trichloroacetal. After 30 hrs. aqueous acetaldehyde is added, and a slow stream of chlorine is led into the warm mixture. The product is almost entirely chloral hydrate. A ferric chloride catalyst may be used.

C. HOLLINS.

Preparation of *N*-monosubstituted ethylenediamines. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assecs. of H. SCHOTTE and H. PRIEWE (G.P. 446,547, 1.7.25).—*N*-Alkyl- or *N*-aryl-*N*-(β -hydroxyethyl)guanidines are heated with concentrated hydrochloric acid at 160°. Creatinol is thus converted, by way of 1-methyl-2-ketotetrahydroglyoxaline, into *N*-methylethylenediamine. *N*-Ethyl (b.p. about 130°) and *N*-phenyl (b.p. 261—262°; *picrate*, m.p. 162°) derivatives are similarly prepared (cf. Schotte and Prieue, A., 1926, 717). The products have therapeutic value. C. HOLLINS.

Manufacture of 3 : 5-di-iodo-2-[hydr]oxypyridine. SCHERING-KAHLBAUM A.-G. (B.P. 288,133, 24.2.28. Ger., 31.3.27).—2-Pyridone is treated with iodine monochloride in dilute hydrochloric acid. C. HOLLINS.

Emetine derivatives for therapeutical purposes. E. ROTHLIN and F. MÜLLER, Assrs. to CHEM. FABR. VORM. SANDOZ (U.S.P. 1,686,930, 9.10.28. Appl., 22.12.27. Switz., 12.1.27).—Salts of bile acids with emetine, or other cephaline alkyl ethers, have strong antiparasitic action and are soluble in alcohol and nearly insoluble in water. F. G. CLARKE.

Extraction of lupanine. E. VON AMMON and K. SZOMBATHY (B.P. 288,637, 12.4.28. Ger., 14.4.27).—Rough-ground lupin seeds are treated with a solution of a salt (especially calcium chloride) of such concentration that the conglutin remains undissolved. The liquid is treated with sulphuric acid to remove proteins, lupinine, and lupinidine, and then rendered alkaline and the lupanine extracted with benzene, whence it is recovered by crystallisation or treatment with dilute hydrochloric acid, giving the hydrochloride. The pure lupanine thus obtained has m.p. 99°. The hydrochloride is separated by treatment with chloroform into its *d*-form (which dissolves) and its *l*-form (insoluble). It is used in the treatment of intestinal diseases. The residue left after extraction of the alkaloids from the seeds as above is re-extracted, and washed with water at 78—82°. The total loss of protein is 2—4%. B. FULLMAN.

Cleansing nicotine vapours. R. G. MEWBORNE (U.S.P. 1,684,740, 18.9.28. Appl., 19.2.24).—The vapours are scrubbed with a hot, circulating solution of caustic alkali, and the cleaned moist vapour is passed to a condenser to recover free nicotine, or is absorbed in sulphuric acid. The liquid containing free nicotine may be concentrated in a vacuum evaporator and the escaping vapour absorbed in sulphuric acid. F. G. CLARKE.

Preparation of basic phenol ethers. I. G. FARBERIND. A.-G., Assecs. of H. HAHN (G.P. 446,606, 8.10.25. Addn. to G.P. 433,182).—The process of the prior patent is extended to alkylated amino-derivatives of higher alkyl halides. The products have strong therapeutic action. 6-Allylguaicol is heated with alcoholic sodium ethoxide and $\beta\gamma$ -bisdiethylaminopropyl chloride, b.p. 87—89°/5 mm. (from the alcohol and thionyl chloride), to give the bisdiethylaminopropyl ether, b.p. 184°/12 mm. Similarly 8-hydroxy-7-allyl-5-methylquinoline reacts with α -chloro- γ -dimethylamino- β -methylbutane, b.p. 52—54°/18 mm., to form an ether, b.p. 215—220°/10 mm., and 8-hydroxy-7-allylquinoline with $\beta\beta'$ -bisdimethylaminoisopropyl chloride, b.p. 83—84° 20 mm., an ether, b.p. 210—214°/10 mm. C. HOLLINS

Manufacture of musk-scented lactones. HAARMANN & REIMER CHEM. FABR. ZU HOLZMINDEN G.M.B.H., Assecs. of M. KERSCHBAUM (G.P. 449,217, 6.3.26. Cf. Kerschbaum, A., 1927, 541).—A suitable halogenated aliphatic acid is treated with silver oxide, or a metal salt is heated in an inert solvent or distilled in a vacuum to give a lactone having at least C₁₂ and preferably C₁₅—16 in the ring. The ω -hydroxy-acids, especially, separated from vegetable musk extracts, are converted first into ω -halides and thence into lactones having an intense musk odour. Juniperic acid [ω -hydroxy-hexadecic acid], m.p. 93—94°, thus yields a lactone, b.p. 170—180°/12 mm., by way of ω -iodohexadecic acid, m.p. 76°; the lactone, b.p. 160—180°/11 mm., of ω -hydroxypentadecic acid, m.p. 83—84°, is similarly obtained from the ω -bromo-acid, m.p. 65°. C. HOLLINS.

Manufacture of water-soluble, complex organic bismuth compounds. B. HEPNER (F.P. 617,063, 4.6.26).—Freshly precipitated bismuth hydroxide, with or without addition of glycerol, is heated with 3 : 3'-diamino-4 : 4'-dihydroxyarsenobenzene and mannitol; alcohol precipitates an oily complex bismuth compound, which quickly sets. The formaldehydesulphoxylate of the arsenobenzene behaves similarly, as does 4 : 4'-dihydroxyarsenobenzene. C. HOLLINS.

Production of chyle and manufacture of a medication therefrom. TORII & Co., LTD., and H. MORI (B.P. 299,530, 2.8.27).—Fatty substances are given to an animal *per os* or by a stomachal probe, and a fistula is made in its ductus thoracicus. The lymph is discharged after 2 or 3 hrs., changes to chyle, which is filtered through silk and paper, respectively, and centrifuged. The liquid obtained is sealed in glass ampoules, and kept at 58—60° for 30 min., this process being intermittently repeated for a few days. The milky-white product contains fatty substances (neutral fats and fatty acids), lipoids, and small quantities of albumin, carbohydrates, and internal secretions. It is injected for therapeutic purposes. B. FULLMAN.

Naphthacridine-*ms*-carboxylic acids (G.P. 446,543).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Negative developers for contrast. J. SOUTHWORTH (Brit. J. Phot., 1928, 75, 689—691, 706—708).—The fog produced by development may be divided into two parts, viz., intrinsic fog, which is latent in the emulsion before development and is formed during either the ripening process or the subsequent ageing, and extrinsic fog, which is caused solely by the action of the developer. The latter, in absence of bromide, tends to increase more rapidly as development is prolonged. With metol-quinol-carbonate developer, and at high values of gamma, potassium bromide depresses extrinsic fog more than intrinsic fog or the latent image. Small traces of soluble iodide assist the action of potassium bromide, and are probably the cause of the decreased fogging tendencies of used developers. The yellow or reddish stain of colloidal silver produced by contrast developers is due to alternate dissolution and reduction of the silver halide. Bad stain may be obtained with quinol-caustic soda developers if the

negative is not thoroughly rinsed before insertion in the fixing bath. The stain is readily soluble in ferricyanide and thiosulphate reducer. J. W. GLASSETT.

PATENTS.

Photographic developers. B. MEREJKOVSKY (B.P. 299,751, 29.6.27. Cf. B.P. 255,925; B., 1926, 805).—Addition of 0.03—0.05% of a secondary amine (piperidine, methyl-*p*-aminophenol) or a primary diamine to the developers of the prior patent counterbalances the retarding effect of the hydroxylaminesulphonic acids whilst retaining the automatic correction of over- or under-exposure. C. HOLLINS.

Manufacture of photographic developers [hydroxyethylated *p*-aminophenols]. I. G. FARBENIND. A.-G. (B.P. 280,873, 17.10.27. Ger., 19.11.26).—*p*-Aminophenol is heated with ethylene chlorohydrin and sodium acetate to give *di*-(β -hydroxyethyl)-*p*-aminophenol, m.p. 140°, or is mixed with less chlorohydrin and alcoholic alkali in much alcohol in the cold to give β -hydroxyethyl-*p*-aminophenol, m.p. 86—97°. Both may be used as photographic developers, and are stable in alkaline solution. C. HOLLINS.

Production of coloured photographs. W. F. FOX, ASSR. to NATURAL COLOR PICTURES CO. (U.S.P. 1,685,281, 25.9.28. Appl., 22.6.20).—A positive and a negative image of the object are printed in alignment on a single layer of sensitive emulsion coated on a suitable base, with unaffected emulsion separating the images, the positive being undermost. Both images are toned with the same colour and the emulsion is then hardened in proportion to the depth of tone. Finally the film is treated with a second dye adapted to act most vigorously on the soft portions of the film. J. W. GLASSETT.

Colour cinematography. R. GSCHÖPF (B.P. 283,560 and Addn. B.P. 289,827, 20.7.27. Austr., [A] 15.1.27, [B] 3.5.27).—(A) Two negative films, one sensitised to red and the other to green, are exposed respectively through a yellow filter alternating with a blue-transmitting red filter and a yellow filter alternating with a blue-transmitting green filter. Positives are printed from these on to either side of a doubly-coated film, the image from the yellow and red filter negative being dyed bluish-green and that from the yellow and green filter negative bluish-red. The dyed images are reduced by washing until cleared of colour in the half tones and are then redyed with yellow. (B) To facilitate the reducing process the positive silver images are converted into a mordant, such as vanadyl ferrocyanide, which holds the dyes very loosely. J. W. GLASSETT.

Non-static and anti-static photographic films and film bases therefore. P. C. SEEL (U.S.P. 1,687,401—2, 9.10.28. Appl., 12.8.27).—The photographic film is coated on the rear side with an anti-static layer containing (A) *p*-toluenesulphonamide-formaldehyde resin, or (B) an ester of abietic acid. J. W. GLASSETT.

Printing with vat dyes (B.P. 279,864).—See VI.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Blasting explosive. R. A. LONG, ASSR. to ATLAS POWDER CO. (U.S.P. 1,689,674, 30.10.28. Appl., 13.5.25).—The explosive contains ammonium nitrate, trinitro-

toluene, a non-explosive combustible, and not more than 8% of nitroglycerin. The total content of nitroglycerin and trinitrotoluene does not exceed 12%, and the proportion of nitroglycerin always exceeds that of the trinitrotoluene. S. BINNING.

Gelatinised explosive composition. K. R. BROWN, ASSR. to ATLAS POWDER CO. (U.S.P. 1,686,952, 9.10.28. Appl., 10.9.26).—In order to produce a stable, readily gelatinisable explosive containing nitrated sugar as well as nitroglycerin and nitrocellulose, a nitrated glycol, e.g., ethylene glycol dinitrate, is added. An explosive containing a high percentage of nitrated sugar is obtained by nitrating a saturated solution of sugar in a glycerin-glycol mixture. F. G. CLARKE.

Storing explosive gases (B.P. 281,718).—See II.

XXIII.—SANITATION; WATER PURIFICATION.

Evaluation of insecticides. C. H. PEET (Ind. Eng. Chem., 1928, 20, 1164—1165).—In order to obtain concordant results in the biological testing of insecticides, temperature, time, humidity, and concentration of toxic mist in the testing chamber must be predetermined. Tests with flies were carried out in a large chamber with spray ports on four sides and a fan for mixing. Non-flying insects were sprayed with a known amount of insecticide in wire cages. Plant pests were tested by spraying an infested plant set in the stopper of a bottle of water. C. IRWIN.

Hydrogen-ion control in water-softening. J. R. BAYLIS (Ind. Eng. Chem., 1928, 20, 1191—1194).—If carbon dioxide is added to water containing calcium hydroxide the minimum hardness of 13 p.p.m. is reached at p_H 9.4. On the other hand, hardness due to magnesium increases with addition of carbon dioxide from zero value at p_H 10.6. The solubility relation of both carbonates to p_H values at 23° was redetermined, and similar curves at other temperatures up to 98° were also obtained. In both cases increase of temperature produces a decrease of solubility. Calcium carbonate shows some increase in solubility in natural waters containing magnesia. A water containing both carbonates can be reduced to minimum hardness by precipitating magnesia with lime at p_H 10.6 and recarbonating to p_H 9.4. In practice, a final figure of p_H 8.7 is better to prevent the incrustation of water-pipes. The best results are obtained by carbonating in stages, but the first stage should be to about p_H 9.0 to avoid incrustation of the sand of filter-beds. C. IRWIN.

Electro-osmotic purification of water. II. A. H. W. ATEN (Chem. Weekblad, 1928, 25, 646—648; cf. B., 1928, 626).—The extent to which water may be freed from dissolved salts by electrolysis in a cell containing two diaphragms of different permeability is mathematically examined. With an E.M.F. of 40 volts, purification of 50 litres requires about 1 kw.-hr. S. I. LEVY.

Protective gas masks. E. DRAGER (B.P. 289,808, 30.4.28. Ger., 2.5.27).

Sand filters (B.P. 298,835). **Air filters** (B.P. 275,257).—See I. **Preventing explosions** (U.S.P. 1,686,051).—See XI. **Polyhydroxy-compound [insecticide]** (U.S.P. 1,677,823).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 18, 1929.

I.—GENERAL; PLANT; MACHINERY.

Utilisable natural energy. H. BARJOT (Compt. rend., 1928, 187, 1048—1050).—It is suggested that the water at almost 0° beneath thick layers of ice in regions where the air temperature is of the order of -40° might be pumped to the surface and its latent heat used to boil a liquefied gas (*e.g.*, ammonia) or, preferably, to remove by boiling a volatile non-aqueous hydrocarbon (*e.g.*, propane) from an intimate mixture with water. The fluid could then be used to work a turbine, frozen sea-water or brine providing an efficient and convenient condenser, since the hydrocarbon would be recoverable from the saline liquor produced by fusion. If the mechanical efficiency of the installation is assumed to be 4%, 1 m.³ of water would supply energy equal to that produced by the fall of the same quantity of water through 1360 m.

J. GRANT.

Technique and economics of mechanical dispersing processes. F. HEBLER (Kolloid-Z., 1928, 46, 225—227).—An investigation into the efficiency of various types of colloid mill. The type selected for a given process must vary with the particular conditions.

E. S. HEDGES.

The Cottrell-Moeller process [for precipitation of dust and mists from gases]. P. VER EECHE (Bull. Féd. Ind. Chim. Belg., 1928, 7, 389—402; cf. B., 1928, 877).—In a particular case the dust deposited by a Cottrell plant working on pyrites burner gases in a sulphuric acid plant amounted to 550 kg./24 hrs., or 1.33% of the pyrites charged, with a current consumption of 0.2 kw. The high temperature (560—570°) in such plants formerly created difficulty owing to the destruction of the insulation, but this has been overcome. At a Norwegian works dusts other than arsenic are precipitated at above 300°, and the arsenic is then separated by treatment at 220°. In plants working in conjunction with contact acid plants, the precipitation of arsenic is so complete that the Marsh test gives a negative result. Amongst other acid mists successfully treated by the Cottrell process are those from acid copper sulphate solution, ammonium nitrate solution, and phosphoric acid. The exit gases from bleaching powder chambers have been treated by injection of lime and subsequent electrical precipitation, and nitric acid is recovered from the waste gases from cellulose nitration. Sulphur dioxide for use in the paper industry is first freed from iron which reduces the catalytic formation of sulphur trioxide, and then freed from the latter by a further Cottrell precipitation with cooling and injection of water. Experiments have been made with the electrodeposition of hydrochloric acid. The removal of dust from blast-furnace gases is a particularly desirable application where these dusts

are rich in alkalis. Carbon dust is successfully precipitated in German lignite briquetting plants, the risk of explosion being guarded against by automatic cut-outs. The electrodeposition of tar is particularly suitable for lignite tar, producer tar, and the like, as trouble with emulsification is avoided. The Cottrell process is capable of removing smoke particles from the gases from industrial furnaces, but in this case no economic return is received.

C. IRWIN.

See also A., Dec., 1345, **Calculation of analyses (LIESCHE)**. 1348, **Tungsten and zirconium oxide furnace (COHN)**. **Thermostat (CUPP)**. **Thermostat for polarimetric work (RAMBERG and HEUBERGER)**.

PATENTS.

[Preventing heat loss from] annealing and other furnaces, muffles, etc. BIRMINGHAM ELECTRIC FURNACES, LTD., and A. G. LOBLEY (B.P. 300,293, 6.7.27).—Heat-retaining flaps or shutters arranged within the charging and/or discharging doors, and/or between preheating and cooling chambers, are displaced by the working charges during movement through or from the furnace.

J. S. G. THOMAS.

Drying or smouldering of loose material. O. DOBBELSTEIN (U.S.P. 1,690,444, 6.11.28. Appl., 26.11.26. Ger., 30.11.25).—The apparatus comprises a vertical, rotary, cylindrical drum operating within a stationary jacket and sealed therefrom at the lower end by means of a liquid. The drum is provided with annular heating chambers arranged one above another; the material to be treated is fed to the spaces between the heating chambers, and means are provided for heating the chambers and for automatically charging the dryer.

A. R. POWELL.

Apparatus for mixing dry and liquid materials. L. TRUE (U.S.P. 1,691,535, 13.11.28. Appl., 23.2.28).—Dry, powdered material is mixed in a chamber provided with delivery nozzles for discharging liquid into different parts of the chamber from points above the material. The liquid is drawn from a tank provided with means for heating the liquid and is forced to the nozzles by a pump.

W. J. BOYD.

Treatment of solid materials with liquid reagents. J. E. EGLESON, Assr. to GEN. CHEM. CO. (U.S.P. 1,690,363, 6.11.28. Appl., 7.3.27).—The solid material is digested with the liquid reagent and the solid residue allowed to settle. The supernatant liquid is withdrawn on completion of settling, and the sludge is washed with a counter-current of water under such conditions that the sludge settles continuously and is continuously withdrawn.

W. J. BOYD.

Apparatus for the concentration of liquids. ZELLSTOFF-FABR. WALDHOF, and A. SCHNEIDER (G.P. 449,685, 6.5.25).—The hot gases after passing around the heating tubes in a tubular evaporator are led over the tops of the Field tubes, these being open. The liquid is conveyed into the Field tubes through rotatable inlet tubes provided with scrapers to remove deposits from the inner walls of the Field tubes. Scrapers are also provided for cleaning the surface of the heating tubes in contact with the hot gases. L. A. COLES.

Performing [exothermic] chemical reactions [between gases]. C. W. MORTIMER (U.S.P. 1,691,903, 13.11.28. Appl., 17.4.23).—The vapour of a liquid kept at the boil to regulate the temperature of gases reacting in the presence of a catalyst is separated into two portions, one being used to heat part of the gases entering the reaction chamber and the other being condensed separately. L. A. COLES.

Friction material, particularly for use for brake liners. BRIT. THOMSON-HOUSTON CO., LTD., H. W. II. WARREN, and R. NEWBOUND (B.P. 300,309, 12.8.27).—Layers of asbestos cloth and brass wire are coated with a solution of the condensation product of phthalic anhydride and glycerin modified with a plasticising agent, *e.g.*, oleic acid; after drying, the layers are heated and moulded under pressure. W. G. CAREY.

Manufacture of friction elements. I. J. NOVAK, Assr. to RAYBESTOS CO. (U.S.P. 1,692,136, 20.11.28. Appl., 26.2.23. Renewed 22.12.27).—The element has an asbestos base containing as binder the solid residue from waste sulphite-pulp liquor. II. ROYAL-DAWSON.

Production of fire-extinguishing foam. R. SCHNABEL, and EXCELSIOR FEUERLÖSCHGERÄTE A.-G. (B.P. 299,097, 22.7.27).—Foam-producing substances, such as saponin solution, are supersaturated with carbon dioxide, or other suitable gas under pressure, in closed vessels; the foam is generated by releasing the pressure. Soluble or insoluble substances which assist the absorption or liberation of the gas (either chemically or catalytically) may be added to the liquid.

P. E. L. FARINA.

Producer or shaft furnace. J. W. REBER, Assr. to WOODALL-DUCKHAM (1920), LTD. (U.S.P. 1,692,572, 20.11.28. Appl., 25.1.27. U.K., 7.7.26).—See B.P. 262,668; B., 1927, 128.

Ultra-filter membrane. J. DUCLAUX (U.S.P. 1,693,890, 4.12.28. Appl., 30.8.23. Fr., 11.9.22).—See B.P. 203,714; B., 1924, 657.

Mechanism for stirring the contents of vessels. G. W. CUSSONS and G. CUSSONS, LTD. (B.P. 301,159, 8.10.27).

II.—FUEL; GAS; TAR; MINERAL OILS.

Air-drying of Canadian lignites, and the re-absorption of moisture by the same. J. H. H. NICOLLS (Canada Dept. Mines Fuel Invest., 1926, 51—60).—When samples of air-dried lignite were completely dried and then re-exposed to the air they never regained the original moisture content. This may have been caused by oxidation during drying. Even when the samples were wetted and exposed to air

they did not retain as much moisture as the original samples. Water was reabsorbed less rapidly and to a smaller extent the more severe and prolonged were the drying conditions. The gain in weight of a dried sample exposed to air was not wholly accounted for by moisture. The moisture content of finely-divided lignites in air depended on its humidity. Improvements in the standard air-drying apparatus (Rep. Sci. Ind. Res. Council of Alberta, 1923, 39) are described. A. KEY.

Pyritic oxidation with special reference to the Ravine seam. II. MACPHERSON, N. SIMPKIN, and S. V. WILD (Safety in Mines Res. Bd., 1928, Paper No. 47, 24 pp.; cf. B., 1926, 1001).—Analyses were undertaken to determine the behaviour on oxidation, by exposure to air, of pyrites in sections of coal from the Ravine seam. Underground observations of corresponding sections in the pit were made. The Ravine seam, consisting mainly of clarain, is covered by a low-grade material, the "Chitters." Examination of the latter and of a coal from a freshly exposed section of the seam showed that the larger the amount of pyritic iron present the greater is the proportion of pyritic oxidation. The base of the "Chitters" and the upper part of the top coal showed the greatest pyritic oxidation. These parts of the seam are also those chiefly suspected of causing heating. Microscopical examination shows the distribution of pyrites as being irregular throughout the seam and occurring chiefly at the base of the "Chitters" and the upper part of the top coal. The content of oxidisable pyrites is small. Its oxidation causes disintegration of the coal and some self-heating. Analyses show the absence of any material likely to cause catalytic oxidation at any point. Photographs showing incrustations and atmospheric oxidation are appended. C. B. MARSON.

Nature of sulphur in Canadian coal and coke. J. H. H. NICOLLS (Canada Dept. Mines Fuel Invest., 1926, 34—50).—Typical Western Canadian coals were analysed for sulphate, pyritic, and organic sulphur. The coking coals from the Mountain Park and the two Crowsnest Pass areas and also the partly coking coals from the Nanaimo area contained very little pyritic sulphur, but the two Saskatchewan coals analysed contained a considerable percentage of both pyritic and sulphate sulphur. Cokes made from the Maritime Provinces' coals were analysed for sulphide, sulphate, free, and solid-solution sulphur. The coke made from the Springhill coal was the only one with a low enough sulphur content to be classed as a good metallurgical coke. The proportion of pyritic sulphur present in Maritime Provinces' coals in a finely-divided condition was determined by means of "float-and-sink" separations, as this part of the sulphur may influence the liability to undergo spontaneous combustion.

W. H. BLACKBURN.

Determination of total sulphur in coal. O. HACKL (Chem.-Ztg., 1928, 52, 933—934).—In view of the low results given by the Eschka method, especially in the case of coals very rich in sulphur, due to (a) loss of sulphur during the heating up process and (b) incomplete oxidation, the following method is suggested. 1 g. of finely-powdered coal, after drying at 100°, is mixed in an iron crucible with 8 g. of a mixture of equal

parts of sodium carbonate and potassium permanganate, and the whole covered with a further 2–3 g. of the mixture. The crucible is very carefully heated to redness and maintained there for $\frac{1}{2}$ hr. After cooling, the crucible contents are extracted with 100–150 c.c. of hot water, containing a small amount of sodium carbonate, until free from sulphate. The filtrate is then precipitated with barium chloride after making slightly acid with hydrochloric acid. In the case of coals very rich in sulphur only $\frac{1}{2}$ g. of coal is taken. F. J. DENT.

Laboratory methods of testing the ash of solid fuels with regard to its chemical activity towards refractories. N. G. PACUKOV (Izvestia Thermo-Tech. Inst., 1928, No. 5, 18–28).—A discussion of recent work on this subject, mainly by the U.S. Bureau of Mines.

T. H. POPE.

Relative inflammability and explosibility of coal dusts. T. N. MASON and R. V. WHEELER (Safety in Mines Res. Bd., 1928, Paper No. 48, 13 pp.).—The results of experiments on a larger scale than those described in Paper No. 33 (B., 1927, 434) are given. Explosions were produced in a gallery 7½ ft. diam. and 720 ft. long. The relative “inflammability” was measured by the mean speed of the flames over a given distance, and the relative “explosibility” by the maximum pressure developed. It is shown that the orders of inflammability and explosibility are approximately the same and in close agreement with the order of inflammability as determined for the same coals from the proportions of incombustible dust required to suppress inflammation, as previously described (*loc. cit.*). The inflammability of a coal dust is directly proportional to its volatile matter content. Exceptions to this proportionality are coals containing a large amount of resinous matter, dusts of which are more inflammable than is indicated by their volatile matter content. In general, volatile matter determinations serve as a useful guide to the degree of inflammability of a coal dust. C. B. MARSON.

Ignition of firedamp by the heat of impact of rocks. M. J. BURGESS and R. V. WHEELER (Safety in Mines Res. Bd., 1928, Paper No. 46, 25 pp.).—Mixtures of methane and air could readily be ignited by the sparks or the heat produced when the edge of a block of rock was pressed against a revolving wheel of the same material. The heated area on the stationary rock appeared to be the direct cause of the ignition. The expenditure of energy required for ignition was comparatively small. The conditions most likely to cause ignition in the mine would appear to be those occurring either when a large mass of rock falls some distance and glides along the sharp edge of another piece, or when a mass of falling roof causes two surfaces to grind against one another under great pressure. No marked differences were observed in the ease of ignition with different sandstones, but the quartzitic types appeared to be the more dangerous. A. B. MANNING.

Low-temperature carbonisation of Brazilian coals. F. W. FREISE (Brennstoff-Chem., 1928, 9, 385–387).—Results of carbonisation tests at 450° are recorded. A. WILSON.

Low-temperature carbonisation tests on Canadian bituminous coals. R. A. STRONG (Canada Dept. Mines Fuel Invest., 1926, 12–33).—The products of distillation of ten bituminous coals of varying volatile matter and ash content were collected and examined, carbonisation being carried out by heating 2 kg. of the crushed coal in an annular retort immersed in a lead bath at 600°. The apparatus and method were described in a previous report (B., 1926, 697). For the coals examined, the yield of tar (average = 13.5 gals./ton) and gas (3000–4000 cub. ft./ton, calorific value 618–724 B.Th.U./cub. ft.) was not sufficiently high to warrant the adoption of low-temperature, in place of high-temperature, carbonisation in view of the poorer handling properties of the cokes. The crude tar oils contained 10–24% of tar acids, and approx. 50% of the remainder was unsaturated and soluble in concentrated sulphuric acid. 1.1–2.7 gals./ton of light oil were obtained from the gas, and an average of 7 lb. of ammonium sulphate from the liquor. F. J. DENT.

Production of illuminating gas from lignite. K. SCHMIDT (Gas- u. Wasserfach, 1928, 71, 1153–1161; cf. B., 1927, 691).—Retorts designed for carbonising lignite are discussed. The gas so obtained, after removing carbon dioxide and hydrogen sulphide, cannot be used directly for town supply because of its high calorific value and density. Methods to overcome this difficulty are: (a) by dilution with hydrogen, (b) by gasifying tar in steam in a separate apparatus and mixing the gas obtained, after removing the carbon dioxide, with the original carbonisation gas, or (c) by the carbonisation of lignite in superheated steam. Processes based on the last-named idea are described. To avoid the initial outlay in adopting the above processes, experiments were made on the carbonisation of briquetted lignite in normal gas-works' retorts. Lignite carbonised more rapidly than bituminous coal, but, especially if the retorts were steamed, the tar escaped without decomposition, yielding a gas of low calorific value. Thus a mode of operation is suggested wherein a setting of retorts is divided into three groups; steam, superheated in the first, is passed through the lignite, being carbonised in the second, and the gas produced is passed through hot coke in the third group to crack the tar vapours and diminish the carbon dioxide content.

F. J. DENT.

Production of illuminating gas from lignite. A. SANDER (Gas- u. Wasserfach, 1928, 71, 1181–1185; cf. preceding abstract).—The carbon dioxide and hydrogen sulphide contents of the gas from lignite, carbonised by the low-temperature process of the Kohlenveredlung A.-G., are reduced from 34 to 6.7% and from 3.9 to 0.08%, respectively, by washing with water under 13 atm. pressure at 15°, while the net calorific value is increased from 5000 to 6200 kg.-cal./m.³ and a yield of 65% by vol. of the crude gas is obtained. The cost of the purification process could be reduced by the formation of sulphur as a by-product from the waste hydrogen sulphide. The hydrogen content of the purified gas could be raised and that of the methane and heavy hydrocarbons diminished, thereby facilitating its combustion in normal burners and decreasing its density

and calorific value, by the addition of (a) ordinary coal gas or (b) electrolytic hydrogen, or by subjecting part of the gas to cracking by passage over coke at 900–1000°, but dilution with water-gas is unsatisfactory. The high-pressure distribution over long distances of a town's gas thus produced is discussed. A. WILSON.

Formation of products rich in sulphur by the action of sulphur dioxide on carbon. F. FISCHER and A. PRANSCHKE (Brennstoff-Chem., 1928, 9, 361–363).—By the passage of sulphur dioxide over semi-coke or over carbon (from carbon monoxide; cf. B., 1928, 146) at 500° or by carbonising filter paper or wood in a current of the gas at the same temperature, products were obtained containing up to 37.5% S, about 60% C, and <1% H. At the same time some free sulphur was deposited on the cooler parts of the tube. Although no means could be found of proving that the sulphur had combined chemically with the carbon, yet it could not be separated from the latter by extraction with solvents, so that the assumption of physical adsorption, at least of the usual type, also failed to account for the formation of these products. A. B. MANNING.

Peat bitumen. G. STADNIKOV and N. TITOV (Brennstoff-Chem., 1928, 9, 357–358).—Russian peats contain up to 24% of bitumen, extractable by means of a mixture of benzene and alcohol. The bitumen consists of a mixture of free acids, saponifiable esters, saturated and unsaturated compounds. By treatment with solvents it can be separated into two fractions, (a) yellow or brown crystalline waxes, soluble in light petroleum, and (b) black, non-crystalline resins, soluble in benzene but not in light petroleum. On heating the bitumen at 180° some polymerisation of the resins takes place with the formation of substances which are insoluble even in carbon disulphide. Comparison of the results of the low-temperature carbonisation of the original peat, the bitumen, and the peat freed from bitumen shows that in carbonising the peat the decomposition of the bitumen and that of the residue proceed independently of each other. A. B. MANNING.

Dehydration of bitumen emulsion from Alberta bituminous sands. P. V. ROSEWARNE and G. P. CONNELL (Canada Dept. Mines Fuel Invest., 1926, 96–103).—Small and large laboratory-scale experiments made on a barrel sample of "separated" bitumen, containing 28% by wt. of water in the form of a stubborn emulsion and 4.3% by wt. of mineral matter, have been found satisfactory. The method in each case consisted in causing the emulsion to flow on to a hot surface in an enclosed space sufficiently large to allow the bitumen to foam without overflowing. Initially, the hot surface was provided by the bottom of a heated pyrex glass flask in the small-scale experiments and by molten lead in the work on a larger scale, but after dehydration the liquid bitumen itself constituted the hot surface for the incoming wet material. The apparatus employed for the intermittently operated small-scale process and also for the large-scale work, either intermittent or continuous in operation, is described, and data are given from typical runs when these processes were employed. The method of proced-

ure should prove suitable for the dehydration of oil-water emulsions in general. A. WILSON.

Determination of the penetration of asphalts. E. GRAEFE (Petroleum, 1928, 24, 1599–1603).—The design and application of a number of penetrometers are described, and results obtained for asphalts of different classes are tabulated together with figures for other physical and chemical properties.

R. H. GRIFFITH.

Theory of the formation of petroleum. III. Composition of the low-temperature tar from Chachareiski boghead coal. G. STADNIKOV and N. PROSKURNINA (Brennstoff-Chem., 1928, 9, 358–361; cf. B., 1928, 700, 735, 882).—The tar was steam-distilled, and from the non-volatile portion the "asphaltenes" (0.31% of the tar) and the paraffin wax (13.3%) were separated. Both portions were then freed from their acidic and basic constituents and were fractionated. The fractions, which contained 3.5–6% of oxygen and consisted largely of unsaturated compounds, yielded, on oxidation by potassium permanganate, products which consisted principally of monobasic acids of the aliphatic series. The hypothesis previously put forward, that the boghead coals consist of the polymerised fats, waxes, and fatty acids of algæ, is thus further confirmed.

A. B. MANNING.

Animal and vegetable fats as parent substances of the inactive constituents of petroleum. M. A. RAKUSIN (Petroleum, 1928, 24, 1519–1520).—The work of Zelinski and Lavrovski on the formation of paraffin hydrocarbons by the action of aluminium chloride on oleic, palmitic, and stearic acids is summarised (cf. A., 1928, 731).

A. B. MANNING.

Canadian shale oil, and bitumen from bituminous sands, as sources of gasoline and fuel oil by pressure cracking. R. E. GILMORE, P. V. ROSEWARNE, and A. A. SWINNERTON (Canada Dept. Mines Fuel Invest., 1926, 121–132).—Shale oil (d 0.896, b.p. range 65–362°), obtained by the destructive distillation of New Brunswick oil shale, was submitted, after "topping" by ordinary distillation to remove the "blending naphtha," to cracking tests at different pressures by the Dubbs residuum and non-residuum processes. Lower gasoline yields with higher yields of better quality residue oils were given by the former process, whilst the results from the latter were in close agreement with those obtained when the same crude shale oil underwent pressure-cracking by the Cross process. Bitumen, obtained from Alberta bituminous sands and containing 28.8% of water and 4% of mineral matter by wt., was dehydrated and "topped" and the product (d 1.061) was distilled in a fire still. The resulting gas oil (equivalent to 67.3% of the dehydrated and "topped" bitumen) was then cracked at 468° and under 700 lb. pressure by the Cross process. The yields of gasoline and fuel oil, from both "topping" and cracking, amounted to over 80% by vol. of the crude shale oil and to about 60% by vol. of the bitumen (water-free basis), whilst gasoline yields of 60–65% of the shale oil, approx. equivalent to 18–20 Imp. gals./ton (2000 lb.) of shale retorted, and of nearly 40% of the bitumen were obtained.

Other products were a good quality coke and an uncondensed gas of high calorific value. After refining, the gasoline, from both shale oil and bitumen, was of good quality; that from bitumen also possessed excellent anti-detonating properties. A. WILSON.

Pritchard process for distillation of oil shale. R. E. GILMORE and A. A. SWINNERTON (Canada Dept. Mines Fuel Invest., 1926, 106—120).—This process, primarily designed for the distillation of wood (cf. B.P. 18,576 of 1914; B., 1915, 949), employs recirculation of uncondensed gases through the charge to facilitate the removal of decomposition products. Laboratory distillation of New Brunswick shale gave an oil yield of 35 gals./ton (2000 lb.). In the large-scale tests on charges of 1500 lb., oil commenced to distil at 400°, and the temperature exceeded 430° only towards the end of the process. The yield of oil in the first two tests was 21 gals./ton, the low value being ascribed to cracking of tar fog reintroduced into the retort, producing gaseous hydrocarbons. The oil yield in the third test was increased to 29 gals./ton by reducing the rate of circulation, by fitting more efficient condensers and scrubbers, and preventing the ingoing gas from coming into contact with the sides of the retort. The quality of the oil was not superior to the product of ordinary distillation processes. The quantity of heat required to preheat the circulating gases was excessive. A. KEY.

Detonation of motor fuels. N. A. BUTKOV (Izvestia Thermo-Tech. Inst., 1928, No. 1, 9—10).—The oxidisability of *n*-heptane, toluene, and Grozny aviation petrol was determined by heating 1 g. of the liquid in a 500 c.c. bomb charged with oxygen under 3 atm. pressure in a bath at 230° for a definite time (1, 2, or 3 hrs.), the bomb being afterwards allowed to cool and the percentages of carbon monoxide and dioxide in the gas determined. The results obtained show that the tendency of a motor fuel to knocking is conditioned by its slight resistance to oxidation. The oxidisability of *n*-heptane is retarded by addition of 2% of β -naphthylamine and, to a greater extent, by 2% of aniline, and aviation petrol containing 30% of toluene is considerably less oxidisable than the petrol alone. The above test is suggested as a means of obtaining an indication of the knocking properties of motor fuels. T. II. POPE.

Capillarity of lubricating oils. N. A. BUTKOV (Izvestia Thermo-Tech. Inst., 1928, No. 5, 29—31).—The theory of the action of lubricating oils is discussed, and the values of the limiting angle for various oils are calculated from measurements of the surface tension and the force of adhesion. Purification of "nigrol" and of a transformer oil by treatment with sulphuric acid removes valuable lubricating substances. The value of the limiting angle for medicinal oil is appreciably diminished by small admixtures of vegetable oils.

T. H. POPE.

Utilisation of marine-animal oils in [internal-combustion] motors. H. MARCELET (Chim. et Ind., 1928, 20, 829—836).—The following constants were determined for a large number of whale, seal, and fish oils: calorific value, temperature of inflammability and combustibility, *d*, and viscosities at various temperatures. Considerable variations occurred in the calorific values

found, some oils, especially those containing squalene, approaching gas oil in value. The lowest ignition point observed was 175°, the majority being above 200°. Viscosities at 25° were very variable, but at 50° were usually below 4° Engler. It is considered that on the whole the figures indicate greater suitability for Diesel engines than with vegetable oils. Trials in Diesel motors showed only minor difficulties due to the rather high viscosities of the oils. It is concluded that there is a prospect of these oils being economically used for such purposes in certain French colonies. C. IRWIN.

[Mechanism of] combustion in internal-combustion motors. A. GREBEL (Chim. et Ind., 1928, 20, 813—818).—It is pointed out that the combustion of hydrocarbons is essentially a chemical problem, and physical calculations based on the assumption that combustion is complete to carbon dioxide and water vapour are inaccurate. The gas mixture in the cylinder is not homogeneous, and its composition does not correspond with chemical equilibrium. The evidence of combustion by stages (to aldehydes, acids, carbon monoxide, etc.) in the presence of insufficient oxygen is summarised. The beneficial effects of the presence of gases of combustion (carbon dioxide and water vapour) in promoting ignition or rather precombustion are explained by hypothetical equations. Methyl alcohol, unlike hydrocarbons, cannot undergo precombustion, being decomposed under cylinder conditions directly into carbon monoxide and hydrogen. The speed of revolution of the engine is necessarily limited by that of flame propagation. A carburettor is referred to in which the heat of exhaust gases is used to effect precombustion with primary air and catalysts. The simple composition of the resultant explosive mixture is an advantage in engine design. C. IRWIN.

See also A., Dec., 1317, Charcoal as an adsorbent (DUBININ). Adsorptive power of certain adsorbents (ALEKSEEVSKI). 1331, Gaseous explosion rates at constant pressure (STEVENS). Propagation of flame in gaseous mixtures (PAYMAN). Gaseous explosions (HUNN and BROWN). 1336, Poisoning of iron catalyst in decomposition of carbon monoxide (WATANABE). 1341, Crystalline carbon with high adsorptive power (HOFMANN).

Utilisable natural energy. BARJOT.—See I. **Purification of phenols.** BRÜCKNER. **Naphthene-sulphonic acids.** VON PILAT and DAVIDSON.—See III.

PATENTS.

Treating and forming artificial fuel. H. E. WETHERBEE, Assr. to R. F. GRANT and H. M. HANNA (U.S.P. 1,687,815, 16.10.28. Appl., 13.4.26).—The finely-divided fuel, before being coalesced, is subjected, in a moistened condition, to the action of a filming carrier and a binding material, in such a way that the latter is evenly applied to the fuel particles.

A. B. MANNING.

Treatment [de-watering] of peat. F. GINSBACH, Assr. to H. HORST (U.S.P. 1,686,807, 9.10.28. Appl., 23.4.27. Luxembourg, 30.4.26).—Raw peat is de-watered in stages by mixing and compressing it with

peat partially dehydrated by compression, and then mixing the product with dry peat and again compressing.

W. G. CAREY.

Distillation of solid fuels. M. KULZINSKI (B.P. 278,740, 7.10.27. Esthonia, 7.10.26).—The fuel is conveyed in a layer through a furnace wherein a gaseous heating medium is circulated in such a way that it passes transversely through the layer of fuel, and then, together with the products of distillation, is reheated by any suitable means and recirculated. At the same time a general movement of the vapours and gases is maintained along the furnace, and at a suitable point they are drawn off and passed to a condensing system.

A. B. MANNING.

Dry distillation of shales, brown coal, etc. PATENTAKTIEBOLAGET GRONDAL-RAMEN (B.P. 278,694, 24.9.27. Swed., 7.10.26).—An apparatus for the distillation of highly bituminous shales etc. consists of a tunnel oven containing a series of travelling perforated grates which can be charged with a layer of the material. Below the grates lie the heating elements formed of vertical tubes heated by the combustion gases from suitably placed fireplaces. The oven is divided into several chambers by partitions which end a little above the upper edge of the grate and a little below the lower edge of the heating elements; at the top of these chambers are fans which draw the distillation gases through the material on the grate, pass them down to the heating elements through channels in the oven walls, and so recirculate the heated gases through the material. In order to maintain sufficient steam within the oven to prevent sticking of the material, part of the gas escaping from the distilling chamber may be returned thereto after the removal of the bulk of its oil, but none of its steam, by cooling it to 100°.

A. B. MANNING.

Heating of finely-pulverised coal. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 291,062, 12.9.27. U.S., 25.10.26).—Coal, sufficiently finely pulverised that it flows like a liquid, is preheated at 300–350° and passed into a carbonising retort in which it travels down through narrow vertical pipes, up between them, down again through an annular space surrounding the pipes, and finally up to an outlet in the side of the retort. Under these conditions the exothermic reaction which is initiated by the preheating produces sufficient heat to complete the carbonisation of the powdered coal. The volatile products of carbonisation are discharged through outlets near the top of the retort.

A. B. MANNING.

Retorts for the distillation of solid carbonaceous substances. LOW-TEMPERATURE CARBONISATION, LTD., and C. H. PARKER (B.P. 299,795, 27.4.27).—A retort setting, specially applicable to vertical retorts of oblong cross-section, comprises two parallel series of retorts and combustion chambers with a series of recuperator chambers lying between them. The heating gases pass from the combustion chambers, down through the corresponding recuperator chambers, and thence to the chimney. The recuperators, which are used for preheating the air for combustion, are formed by sets of vertical pipes connected in series.

A. B. MANNING.

Apparatus for distilling combustible materials.

O. HUBMANN, Assr. to METALLBANK U. METALLURGISCHE GES. A.-G. (U.S.P. 1,690,934—5, 6.11.28. Appl., [A] 12.7.26, [B] 7.12.26. Ger., [A] 23.10.24, [B] 18.12.25).—(A) The material is fed into a shaft-like still, in which it is distilled in a current of hot gases introduced centrally about mid-way down the shaft. Below the inlet for the heating gases is a second inlet for cooling gases. Gas outlets are provided near the top of the shaft and also in the side walls at about the level of the cooling gas inlets. (B) High-ash, solid, carbonaceous material is passed continuously down through a retort in which it passes successively through a distillation zone, a reaction zone, and a residue-cooling zone. Gases free from oxygen are introduced into the cooling zone, and gases containing oxygen into the reaction zone. The hot gases issuing from both these zones are mixed and passed through the distillation zone, thereby effecting carbonisation of the charge.

A. B. MANNING.

Regenerative channel oven. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,687,774, 16.10.28. Appl., 6.7.21. Renewed 25.6.27. Ger., 13.9.18).—The heating channels, which lie parallel to one another, are divided by a partition, and the oven is so arranged that the preheated fuel enters one channel at the outlet end and flows counter-current to the material being heated; the burning fuel then flows round the partition and passes along the second channel in the direction of movement of the material being heated therein, finally passing from the outlet of that channel to the outflowing regenerators.

A. B. MANNING.

Regenerative coke oven. G. O. WOLTERS (U.S.P. 1,690,805, 6.11.28. Appl., 10.9.26. Ger., 14.3.25).—A coke oven with vertical flues communicating with each other at their upper ends is provided with primary air passages connecting the regenerators beneath the coking chambers with the bottoms of the flues, and also has secondary air inlet passages passing vertically up between the regenerators and through alternate partitions separating the flues, with outlets to the combustion chambers at the upper ends of the partitions.

A. B. MANNING.

Production of coke. CHEM. TECHN. GES. M.B.H., Assees. of S. STERNBERG (B.P. 288,264, 19.3.28. Ger., 6.4.27).—A semi-coke is produced by the low-temperature carbonisation of a fuel to which has been added a flux which, although not interacting with the ash of the fuel during carbonisation, does so at the higher temperatures reached during the combustion of the coke, producing an ash or clinker of a desired character. The flux added is preferably one which also favourably affects the yield and composition of the tar.

A. B. MANNING.

Coke and its production. URBANA COKE CORP., Assees. of S. W. PARR and T. E. LAYNG (B.P. 290,575, 14.5.28. U.S., 12.5.27. Cf. Chapman, B., 1926, 905).—The crushed coal is passed from a hopper into an inclined rotating drum, mounted within a heating chamber, its temperature being thereby raised to a point (about 300°) just below that at which it becomes plastic. The coal undergoes a preliminary drying in the hopper, in which are pipes traversed by hot waste gases. The preheated coal is discharged from the rotating drum into a travelling car which, in turn, feeds a battery of vertical retorts.

The latter, each of which is constructed of a single piece of metal, are circular in cross-section and slightly tapered; they are heated by gas flames and are maintained at 700–800°. The exothermic reactions which commence immediately after the coal is dropped into the retorts produce a rapid rise of temperature throughout the charge, the coking process being completed in a few hours. The coke produced is as strong as by-product coke, but possesses smaller pores and a much lower ignition temperature; its volatile content is approx. 4%, and it forms a smokeless fuel suitable for domestic and other purposes. If desired, the preheating apparatus may be used in conjunction with an ordinary by-product oven in which the carbonisation is completed.

A. B. MANNING.

Activating or revivifying carbonaceous material. L. WICKENDEN and S. A. W. OKELL (B.P. 300,146, 5.7.27).—The material is fed continuously into an electric furnace in which it is heated at 800–900°. The upper electrode of the furnace is adjustable in position, whilst the lower, which is rotatable, is hollow and perforated, and so designed that a current of gas or steam can be passed through it into the furnace. The treated material is carried over into a separator and passes thence to a rotary electric furnace, in which it is subjected to further treatment at about 400° in a current of air or other oxidising gas.

A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 300,703, 11.8.27. Cf. B.P. 276,001; B., 1929, 8).—A catalyst for use in the hydrogenation of carbonaceous materials at elevated temperatures and under high pressures contains up to 10% of a mixture of copper and zinc or cadmium, the remainder consisting of boron, aluminium, silicon, vanadium, etc., or their compounds. The hydrogenating gas is used in large excess and preferably in a circulatory system.

A. B. MANNING.

Manufacture of liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of carbonaceous materials. I. G. FARBERIND. A.-G. (B.P. 275,663, 8.8.27. Ger., 7.8.26).—The process is carried out at elevated temperatures and under pressures of at least 50 atm., in the presence of catalysts composed of metals of the fifth periodic group, or their compounds, together with magnesium or its compounds.

A. B. MANNING.

Treatment of bituminous materials [from the berginisation of coal]. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 623,184, 15.10.26. Ger., 8.5. and 11.6.26).—The crude viscous distillate is treated with benzene or light petroleum and with a small quantity of a mineral acid, whereby the resinous matter is coagulated and may be separated by filtration. The remaining oils are then separated by fractional distillation or by chemical treatment.

A. R. POWELL.

Treatment of fuel-air mixtures for internal-combustion engines. J. GRIVINS (B.P. 281,702, 2.12.27. Latvia, 4.12.26).—The fuel-air mixtures are passed through a centrifugal separator which throws the insufficiently atomised fuel particles into an annular chamber heated by the exhaust gases. After evapora-

tion therein the fuel is re-mixed with the remainder of the fuel-air mixture. The whole is then mixed thoroughly with cold secondary air, cooled in this way until the fuel begins to condense, and then admitted to the cylinders.

A. B. MANNING.

Gas-producing apparatus. W. L. SHEPARD, Assr. to E. A. BEALS (U.S.P. 1,689,159, 23.10.28. Appl., 11.6.25).—A gas-generating chamber, arranged for the partial combustion of fuel therein, is fed from a hopper immediately above it. A hollow, vertical shaft to which worm-gearing is attached can be rotated within the hopper, the lower end of which can be closed or opened by means of a valve attached to and operated by a stem passing down through the hollow shaft.

A. B. MANNING.

Manufacture of fuel gas. A. H. WHITE (U.S.P. 1,689,940, 30.10.28. Appl., 14.1.20).—A gas of calorific value above 200 B.Th.U./cub. ft. is made by blowing a charge of solid fuel in a generator with a gaseous mixture containing steam and oxygen in such proportions that the fuel bed is continuously maintained at a suitable temperature; at the same time enriching materials are introduced into the hot gases and are allowed to interact with them, out of contact with metallic surfaces, at temperatures which fall slowly, so that the separation of free carbon from the enriching hydrocarbon is avoided.

A. B. MANNING.

Manufacture of gas of high calorific value. R. DRAWE (F.P. 622,305, 30.9.26).—Bituminous coal is partly coked in a container above the ordinary gas producer, and the coke is then treated with air and steam in the producer proper.

A. R. POWELL.

Recovery of benzene and hydrocarbons from coke-oven gas. SOC. ANON. MÉTALLURG. DE SAMBRE ET DE MOSELLE, and GES. F. LINDE'S EISMASCHINEN A.-G. (F.P. 623,568, 19.10.26. Ger., 4.8.26).—The gas is compressed and cooled to remove benzene, water, and naphthalene, and the purified gas is then cooled further on the counter-current principle by means of expanded gas which has already been purified, whereby the remaining easily condensable hydrocarbons are deposited.

A. R. POWELL.

Production of mixtures of hydrogen and carbon monoxide. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 300,328, 24.8.27).—Vaporised hydrocarbons, or gases containing hydrocarbons (e.g., coal gas), are burnt with just sufficient oxygen at temperatures above 1000° (preferably 1250–1500°) to convert all the carbon into carbon monoxide. The gas is first given an eddying motion by tangential passage into the lower part of a chamber, whence it expands through an annular passage in to the combustion chamber where the oxygen, as such or in admixture with air, is introduced under pressure through a series of water-cooled nozzles. The hydrocarbons are all converted into carbon monoxide and a small quantity of carbon dioxide; no carbon is deposited. The admission of oxygen is regulated so as to produce the required temperature of combustion; water vapour should be excluded.

P. E. L. FARINA.

Isolation of pure hydrogen from gaseous mixtures. J. Y. JOHNSON. From I. G. FARBERIND. A.-G.

(B.P. 299,167, 29.9.27).—The gaseous mixtures are washed with benzines under pressure and at a temperature below -60° but above the f.p. of the solvent. Greater efficiency is obtained by atomising the solvent in a scrubber into which the gas is introduced under pressure. Methane and its higher homologues are the chief impurities removed, other compounds being potential catalytic poisons such as ether, carbon monoxide, hydrogen sulphide, carbon disulphide, etc.

P. E. L. FARINA.

Dehydration of tar. A. W. WARNER (U.S.P. 1,689,309, 30.10.28. Appl., 12.6.24).—A comparatively dehydrated tar is circulated through a heater, a separator, and a pump, and a limited amount of the wet tar is added to the main current as it passes from the separator, so that the temperature of the mixture is reduced below that at which frothing occurs in the pump. The temperature of the tar is raised in the heater to a point at which the water is freely vaporised, so that water vapour and dry tar can be drawn off from the separator at a rate corresponding with that of addition of the wet tar.

A. B. MANNING.

Working-up low-temperature tar. F. C. BUNGE and H. MACURA (B.P. 285,000, 4.5.27. Ger., 8.2.27).—The tar or any of its fractions is treated with a halogen compound, *e.g.*, phosgene, phosphorus tri- or pentachloride, tin tetrachloride, etc., which combines with hydroxyl or thiol groups with the elimination of hydrogen halide. The tar may be neutralised and dried before treatment. The products are fractionated under reduced pressure and yield phenol esters, lubricants, substances which on nitration produce explosives, etc.

A. B. MANNING.

Separation of the neutral constituents from low-temperature tar and its fractions. F. C. BUNGE (B.P. 287,471, 3.5.27. Ger., 21.3.27).—The acid constituents of low-temperature tar, or its fractions, are removed by extraction with phosphoric acid, either at the ordinary or at higher temperatures. The tar may be diluted before treatment with some of the neutral oil already separated, or other suitable diluent.

A. B. MANNING.

Production of aqueous emulsions of bitumen. CHEM. FABR. BUCKAU (B.P. 298,842, 22.2.28. Ger., 26.1.28).—Melted bituminous material is mixed with 0.03–2% of an alkaline solution of boric acid as emulsion carrier.

W. G. CAREY.

Production of bituminous emulsions. J. A. MONTGOMERIE (B.P. 300,414–5, 19.12.27. Cf. B.P. 226,032; B., 1925, 91).—Aqueous bituminous emulsions are prepared by pouring molten bitumen of the type artificially produced from petroleum, *e.g.*, Mexican asphalt, with stirring, into a solution of (A) normal sodium phosphate, or (B) an oxidising alkali, *e.g.*, sodium peroxide, both ingredients being at 100° , with or without the addition of other reagents or assistants.

H. S. GARLICK.

Production of bituminous emulsions. J. A. MONTGOMERIE (B.P. 300,821, 24.12.27).—Molten asphalt is emulsified with a hot dilute alkali cresoxide solution with the addition, if desired, of stabilising agents, *e.g.*, sodium laurate.

L. A. COLES.

Apparatus for controlling cracking stills. W. C. PARRISH, Assr. to TEXAS Co. (U.S.P. 1,690,243, 6.11.28. Appl., 16.4.24).—The oil is maintained at the cracking temperature and is agitated mechanically, the power necessary to operate the stirrer being recorded and giving a measure of the production of carbon within the cracking chamber. The process can then be stopped when the carbon production exceeds a predetermined amount.

A. B. MANNING.

Apparatus for treating oils. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,690,997, 6.11.28. Appl., 9.7.17).—An oil-cracking still consists of a number of vertical pipes, situated in the heating flues of a furnace, and through which the oil is pumped.

C. O. HARVEY.

Treatment of mineral oils. T. G. DELBRIDGE and H. F. DURE, Assrs. to ATLANTIC REFINING Co. (U.S.P. 1,690,772, 6.11.28. Appl., 21.5.25).—Unrefined hydrocarbon oil is decolorised by a process involving acid treatment, separation from the acid sludge, air-blowing the heated unneutralised oil, adding finely-divided clay, and finally subjecting the hot mixture to a treatment with air at below 100° .

C. O. HARVEY.

Distillation [of hydrocarbon oil]. G. W. WATTS, Assr. to STANDARD OIL Co. (U.S.P. 1,689,606, 30.10.28. Appl., 3.4.25).—Superheated steam is allowed to expand into a chamber wherein it is mixed with hydrocarbon oil. The mixture passes along an externally unheated conduit until equilibrium between oil and steam is established, when the vapours and unvaporised residue are separated and drawn off.

C. O. HARVEY.

Desulphurising hydrocarbon oils. R. C. MORAN, Assr. to VACUUM OIL Co. (U.S.P. 1,692,756, 20.11.28. Appl., 18.1.27).—The oil is subjected to the action of mercury, which is simultaneously treated with an alkali sulphide solution.

H. ROYAL-DAWSON.

Treatment of hydrocarbon residues. C. ARNOLD. From STANDARD DEVELOPMENT Co. (B.P. 299,925, 9.8.27).—Heavy hydrocarbon oil residues derived from cracking processes are treated with "acid oil" at an elevated temperature, if desired, in order to bring about a rapid precipitation of the suspended solid matter in such a way as to permit a ready separation of the clarified oil and the sediment. The "acid oil" is obtained in the process of recovering acid which has been used in petroleum refining.

A. B. MANNING.

Fuel for use in internal-combustion engines. ASIATIC PETROLEUM Co. LTD., A. C. EGERTON, and C. H. BARTON (B.P. 300,156, 6.8.27).—0.5–1.5% of a compound of an alkali metal, *e.g.*, oleate, naphthenate, or sulphonate, is added to a liquid hydrocarbon fuel, such as petrol, in which it is soluble, to reduce the tendency to pink or knock.

H. S. GARLICK.

Apparatus for producing hard, homogeneous fuel or similar objects from peat, peat moss, lignite, etc. B. JIROTKA, Assr. to DR. O. SPRENGER PATENTVERWERTUNG JIROTKA M.B.H. (U.S.P. 1,691,196, 13.11.28. Appl., 11.2.27. Ger., 13.8.21).—See B.P. 276,471; B., 1927, 867.

Gas producer. F. L. DUFFIELD (U.S.P. 1,692,724, 20.11.28. Appl., 10.1.24. U.K., 19.3.23).—See B.P. 217,645; B., 1924, 703.

Apparatus for continuous preliminary treatment of crude benzols. E. A. BARBET (U.S.P. 1,693,774, 4.12.28. Appl., 1.12.25. Fr., 5.12.24).—See B.P. 244,130; B., 1927, 182.

Coal dressing apparatus. K. WOLINSKI (B.P. 277,011, 1.9.27. Ger., 1.9.26).

III.—ORGANIC INTERMEDIATES.

Hydrolysis of phenolsulphonic acids and purification of phenols by separation of their sulphonic acids. H. BRÜCKNER (Z. anal. Chem., 1928, 75, 289—292).—The temperatures at which hydrolysis in dilute sulphuric acid solution occurs of phenol-, cresol-, and xylol-sulphonic acids have been determined. The results are applied in a method of separation of mixtures of these phenols by sulphonation of such a mixture, dilution, and fractional hydrolysis of the sulphonic acids.

A. DAVIDSON.

Naphthenesulphonic acids. S. VON PILAT and E. DAVIDSON (Petroleum, 1928, 24, 1559—1561).—Technical naphthenesulphonic acids, obtained by sulphonation and separated during refining of petroleum with caustic alkali, consisted of a thick, almost black liquid which contained 19% of water and 6—8% of ash; the crude material dissolves in water and in various organic solvents, and has an acid value of 24.6. Attempts to separate the acids from hydrocarbon oils by treatment with acetone or dilute alcohol proved unsuccessful, but by extracting a slightly alkaline solution with limited amounts of ether the oily impurities were removed. The raw material (10 g.) was treated with 20 g. of water, made alkaline with caustic soda, and shaken ten times with 10 c.c. portions of ether. The residual acid was freed from the sodium salt solution, washed, and dried at reduced pressure over phosphorus pentoxide, when it formed a dark, gummy, hygroscopic mass. The mol. wt., determined by the b.p. method, was 400—401, and by titration 410, showing that the acids are monobasic. Two series of salts have been isolated, known as α - and β -compounds; the latter are soluble in ether, but their relationship to, or differences from, each other are not yet defined.

R. H. GRIFFITH.

See also A., Dec., 1317, **Adsorption of organic acids on animal charcoal** (GRIFFIN and others). 1338, **Electrolytic oxidation of formaldehyde** (MÜLLER and TAKEGAMI). 1353, **Electrolytic oxidation of alcohols** (KOIDZUMI). 1366, **Reduction of aromatic nitro-compounds** (BRAND and MODERSOHN). 1369, **Nitration of phenol** (BEAUCOURT and HÄMMERLE). 1370, **Catalytic production of polynuclear compounds** (CLEMO and SPENCE). 1371, **Electrolytic oxidation of alcohols** (KOIDZUMI). 1372, **Electrolytic reduction of aldehydes** (SHIMA). 1373, **4-Chloroanthranilic acid** (HELLER and HESSEL). 1376, **Perylene and its derivatives** (PONGRATZ).

Succinchloroimide for treatment of water. WOOD.—See XXIII.

PATENTS.

Catalytic production of methyl alcohol. COMMERCIAL SOLVENTS CORP., Assees. of J. C. WOODRUFF and G. BLOOMFIELD (B.P. 272,864, 20.4.27. U.S., 21.6.26).—As catalyst for the combination of hydrogen with carbon oxides under more than 50 atm. pressure a mixture of non-reducible metal oxides and a metal halide is used; examples are zinc, magnesium, or strontium oxide with chromium, manganese, or vanadium oxide, and zinc, magnesium, or chromium chloride. C. HOLLINS.

Separation in the anhydrous state of fatty acids contained in dilute aqueous solutions. DISTILLERIES DES DEUX-SÈVRES (B.P. 273,744 and 290,992, [A] 1.7.27, [B] 11.10.27. Belg., [A] 1.7.26, [B] 23.5.27).—(A) Anhydrous fatty acids such as acetic or butyric acid may be separated from their dilute aqueous solutions by adding an acetic acid ester and distilling. The whole of the water is removed in the form of an azeotropic mixture with the ester, leaving an easily separable acid-ester mixture. (B) Since the foregoing process becomes uneconomic or even impracticable at higher concentrations of the aqueous fatty acid, it is proposed to add a hydrocarbon solvent, as benzene, toluene, or xylene, to the ester used in (A), the amount of hydrocarbon varying with the concentration of the solution. Thus a 20% solution of acetic acid may satisfactorily be treated with a mixture of 75% of ethyl acetate and 25% of benzene. E. HOLMES.

Substitution of alkyl groups in the aromatic nucleus [nuclear alkylation of aromatic compounds]. A. VERLEY (B.P. 288,122, 16.6.27. Fr., 31.3.27).—Potassium pyrosulphate is dissolved in 3 pts. of concentrated sulphuric acid and an alcohol is added to give a solution of alkyl hydrogen sulphate, which reacts with aromatic compounds at 60—70° to yield nuclear alkyl derivatives, e.g., *o*-cresol-5-sulphonic acid from phenol-*p*-sulphonic acid, thymol from *m*-cresol, *p*-cymene from toluene, *m*-toluic acid from benzoic acid.

C. HOLLINS.

Manufacture of sulphonated aminoanthraquinones. IMPERIAL CHEM. INDUSTRIES, LTD., A. DAVIDSON, W. W. TATUM, and G. E. WATTS (B.P. 299,279, 30.5.28).—1-Aminoanthraquinone-2-sulphonic acid is obtained by baking α -aminoanthraquinone sulphate (1.0—1.5 mol. H_2SO_4) at 220—230°; if pure α -aminoanthraquinone is the starting material the temperature may be 240—245°. C. HOLLINS.

Carrying-out catalytic oxidations in the gaseous or vapour state. J. BRODE and A. JOHANNSEN, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,693,915, 4.12.28. Appl., 25.4.27. Ger., 14.6.26).—See B.P. 272,557; B., 1928, 440.

Production of metaldehyde. E. LÜSCHER and H. STEIGER, Assrs. to ELEKTRIZITÄTWERK LONZA (U.S.P. 1,693,204, 27.11.28. Appl., 22.7.26. Ger., 23.6.24).—See B.P. 235,867; B., 1925, 738.

Manufacture of crotonaldehyde from acetaldehyde and aldol. E. LÜSCHER, Assr. to ELEKTRIZITÄTWERK LONZA (U.S.P. 1,693,907, 4.12.28. Appl., 25.4.27. Switz., 4.5.26).—See B.P. 270,764; B., 1928, 515.

Manufacture of aliphatic compounds [acetic acid and acetic anhydride]. W. BADER, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,693,331, 27.11.28. Appl., 6.11.24. U.K., 24.1.24).—See B.P. 237,302; B., 1925, 827.

Manufacture of cyanides [nitriles] of the perylene series. A. PONGRATZ, Assr. to F. BENSA (U.S.P. 1,692,417, 20.11.28. Appl., 18.6.26. Austr., 26.6.25).—See B.P. 254,310; B., 1927, 29.

Manufacture of 1:3-diamino-2-hydroxyanthraquinone from 4'-hydroxy-o-benzoylbenzoic acid, and of the intermediate products. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN (B.P. 301,119, 29.8.27).—See U.S.P. 1,659,358—60; B., 1928, 316.

Bleaching of fatty acids (B.P. 299,149).—See XII. **Nitration of alcohols** (B.P. 284,700).—See XXII.

IV.—DYESTUFFS.

Photographic applications of diazo compounds. SPENCER.—See XXI.

See A., Dec., 1368, **Substantive dyes from 2:7-diaminofluorene** (NOVELLI and RUIZ). 1371, **Bathochromic action of the methylthiol group in azo-dyes** (BLUMENSTOCK-HALWARD and others). 1385, **Polymethine dyes** (KÖNIG and others). 1403, **Reactions of basic dyes with cyclic derivatives of an acid character** (HOLMES and HANN).

PATENTS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 298,696, 18.7.27).—A halogenated benzamidoalkoxyanthraquinone is condensed with an aminobenzamidoanthraquinone (which may carry an alkoxyl group), or corresponding hydroxy-compounds are condensed and alkylated. From 5-chloro-1-benzamido-4-hydroxyanthraquinone and 4-amino-1-benzamidoanthraquinone an alkali-sensitive bordeaux vat dye is produced, which on methylation yields an alkali-fast brown vat dye. 5-Chloro-1-benzamido-4-methoxyanthraquinone with 5-amino-1-benzamidoanthraquinone gives an intense yellowish-brown, whilst with 5-amino-1-benzamido-4-methoxyanthraquinone it yields a clear red.

C. HOLLINS.

Manufacture of azo dyes insoluble in water. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 298,697, 18.7.27).—An unsulphonated aminofluorenone is diazotised and coupled in substance or on the fibre with β -naphthol, 2:3-hydroxynaphthoic arylamides, etc. Examples are 2-aminofluorenone with Naphthol AS (red), β -naphthol (red), bis-(2:3-hydroxynaphthoyl)-dianisidine (red-brown); 2:7-diaminofluorenone with Naphthol AS (violet) or with hydroxy- α -naphthacarbazole (from 1:7-naphthylaminesulphonic acid and phenylhydrazine, with subsequent alkaline fusion; black).

C. HOLLINS.

Manufacture of polyazo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 298,518, 6.7.27).—Fast green tetrakisazo dyes are obtained by coupling a diazotised aminodisazo dye of the type: arylamine-sulphonic acid \rightarrow α -naphthylamine or Cleve acid \rightarrow a substituted α -naphthylamine, with a J-acid derivative

containing an external amino-group, rediazotising, and coupling with an end-component. As first components aniline-2:4- or -2:5-disulphonic acid, metanilic acid, *o*-anisidine-4-sulphonic acid, *m*-chloroaniline-6-sulphonic acid, *p*-nitroaniline-2:5-disulphonic acid, 5-sulphoanthranilic acid, and β -naphthylamine-4:8-disulphonic acid are mentioned. Suitable third components are 2-ethoxy- or 2-methoxy-Cleve acid, 1:2-aminonaphthyl methyl ether, 1-amino-2:7-dimethoxynaphthalene, 1:5-aminonaphthol, 1:5:7-, 1:5:6-, and 1:8:6-aminonaphtholsulphonic acid (all aminonaphthols are best coupled in the form of their *O*-acetates, with subsequent hydrolysis), β -(1-amino-2-naphthoxy)-propionic acid, 2- β -sulphatoethoxy-1-naphthylamine, 2:7-biscarboxymethyl- α -naphthylamine; the three compounds last mentioned are obtained by nitration and reduction of β -2-naphthoxypropionic acid, the condensation product of 2:7-dihydroxynaphthalene with chloroacetic acid, and β -naphthyl β -sulphatoethyl ether, respectively. For the fourth component *m*- or *p*-aminobenzoyl-J-acid or their 1-sulpho- or 1-chloro-derivatives, *m*- or *p*-aminobenzamidobenzoyl-J-acid, 8-hydroxy-2-*m*-aminophenyl- α -naphthiminazole-6-sulphonic acid ("J-acid iminazole"), the corresponding naphththiazole, 5-amino-*N*-(5-hydroxy-7-sulpho- β -naphthyl)benziminazole, 5-amino-*N*-(5-hydroxy-7-sulpho- β -naphthyl)benztriazole, and *s-m*- or -*p*-aminophenyl-5-hydroxy-7-sulpho- β -naphthylcarbamide (from J-acid by action of *m*- or *p*-nitrophenyl-carbamide or -carbimide, with subsequent reduction) are recommended. The end-component may be 2-methylindole or its sulphonic acid, salicylic or *o*-cresotic acid, 2:4-dihydroxyquinoline, indan-1:3-

dione, benzsulphazone ($\text{C}_6\text{H}_4 \begin{smallmatrix} \text{SO}_2 \cdot \text{CH}_2 \\ | \\ \text{NH} - \text{CO} \end{smallmatrix}$), acetoacetic

anilide, *o*-toluidide, tolidide, *p*-carboxyanilide, etc., or pyrazolones; pyrazolones from *o*- and *m*-aminobenzoic acids, benzidine, dehydrothiotoluidinesulphonic acid, 5-aminosalicylic acid, 3-amino-5-sulphosalicylic acid, β -naphthylamine-6-sulphonic acid, J-acid, and 4"-amino-4:4'-dihydroxy-5:5'-dicarboxy-3:3'-dimethyltriphenylmethane are mentioned.

C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Transmission of ultra-violet radiation through various fabrics. W. W. COBLENTZ, R. STAIR, and C. W. SCHOFFSTALL (U.S. Bur. Stand. J. Res., 1928, 1, 105—124).—The transmission of ultra-violet and of visible radiation through cotton, silk, nitrocellulose, cuprammonium, viscose, and cellulose acetate silks, linen, and wool has been measured. By examining black and white samples of the same material, the effect of radiation transmitted through the openings in the fabric was eliminated. Comparing materials having the same weight, there is practically no difference in the amount of ultra-violet radiation transmitted through bleached samples of cotton, linen, viscose silk, and cellulose acetate silk. Fresh, white, natural silk is almost as transparent as bleached cotton, whilst wool is only half as transparent. In all cases when the fabric is dyed, or slightly yellow with age, the ultra-violet transmission through the thread is greatly decreased. Hence, in

comparing dyed fabrics, the one with the largest openings between the threads transmits the most ultra-violet radiation.

W. E. DOWNEY.

Viscosity determinations with artificial silk pulp. E. OEMAN (Papier-Fabr., 1928, 26, 770—778).—Methods for the preparation of viscose for viscosity determinations are examined, and the effect of variations in technique on the results obtained are recorded and discussed. A slightly modified Ostwald viscosimeter is recommended and arranged to give minimum deviation of pressure during the flow of liquid. Xanthation is most satisfactorily carried out with carbon disulphide vapour in a closed vessel. Pretreatment of the pulp tending to increase the hydration of the pulp (*e.g.*, low-temperature drying, soaking in water, heavy beating, etc.) increases the viscosity of the product. Increased amounts of carbon disulphide used result in higher viscosities. A burette fitted for accurately delivering small quantities of carbon disulphide is described. The increased viscosity associated with smaller amounts of residual soda after pressing out the mercerising solution is considered not as a function of the soda content but as the result of the more rapid and intimate contact with carbon disulphide under these conditions.

A. G. POLLARD.

Paper pulp and cellulose from the Eucalypts by the sulphite process. L. R. BENJAMIN and J. L. SOMERVILLE (Comm. Australia Council for Sci. Ind. Res., Bull. No. 37, 1928, 84 pp.; cf. B., 1927, 871).—Details of cooking technique for newsprint pulp from several species of Eucalypts are examined. The best pulp was produced by cooking for $5\frac{1}{2}$ –6 hrs. at 140° with liquor containing 1.6% of combined, and 3% of excess, sulphur dioxide. Liquor was used at the rate of 1000 Imp. gals. per 2000 lb. of chips (oven-dry basis). *E. regnans* and *obliqua* were most satisfactory for yield and strength of newsprint. *E. delegatensis* (gum top) was rather less satisfactory for newsprint, but not markedly inferior for other purposes. The shorter cooking period, compared with spruce, fir, etc., is attributed to the lower lignin contents of the Eucalypts and to the fact that the lignin is more accessible to the liquor, since the ultimate cells are shorter, finer, and thinner-walled. Well prepared pulp for artificial silk had an α -cellulose content of 88%. Details of tests with *Pinus insignis* and *Acacia dealbata* (silver wattle) are recorded, and photomicrographs of the various pulped fibres are given.

A. G. POLLARD.

Pulping flax straw. V. Production of pulp by the chlorine process. E. R. SCHAFER and C. E. PETERSON (Pulp and Paper Mag., 1928, 26, 1588—1595).—Semi-commercial trials based on the results of laboratory experiments (*cf.* B., 1927, 327) indicate that chlorinated flax-straw pulps might possibly be utilised in the manufacture of high-grade papers if they were admixed with, *e.g.*, soda wood pulp to increase the opacity and softness of the sheet. Chlorinated flax-straw pulps, however, are at best greyish-white in colour and hydrate very easily, so that the beating operation must take place after the alkaline digestion and before chlorination. For the alkaline digestion the sulphate process appears to give somewhat better

results than the soda process, and it is on the degree of pulping accomplished at this stage that the physical properties of the chlorinated pulps largely depend; the lower the degree of cooking the greater are the chlorine consumption to complete the pulping action and the tendency of the finished pulp to hydrate.

D. J. NORMAN.

Quality of pine wood, with special reference to wood for grinding and papermaking. E. MORK (Papier-Fabr., 1928, 26, 741—747).—The total width of the annual growth-rings has been compared with the width of the autumn-wood sections, and measurements of fibre length and thickness in both ring zones have been made for wood from different parts of the trunk. The terms "autumn" and "spring" wood are defined. The view that narrow-ringed conifer wood is the more valuable because, independent of the width of the growth-rings, the autumn-wood zones always appear to be constant, cannot always be upheld. Under otherwise equal conditions, narrow growth-rings have generally a greater percentage of autumn wood and longer fibres than wide rings, and wood of higher value has a greater amount of autumn wood and longer fibres than wood of average value. Measurement of such factors is recommended in order to find what localities and conditions of growth give wood of the best quality for the cellulose industries.

B. P. RIDGE.

Theory of sizing [of paper]. H. SCHWALBE (Papier-Fabr., 1928, 26, 738—740).—The nature of sizing is determined by three chief factors, (1) the quality of the rosin, the rosin size, and the rosin milk, (2) precipitation and fixation in the hollander, and (3) fritting and layer formation. Of these, the second is of less importance. The influence of these and of other factors such as temperature, emulsification, etc. is discussed. In drying, the temperature attained by the paper depends on the surface temperature of the cylinders, the speed of the machine, the clearance between cylinders, the motion of the layer of air between them, and the condition of the cylinder surfaces. By increasing the drying temperature the sizing strength of the paper may be increased ten- to twelve-fold. The mechanism of sizing is considered to be a bridging over of the pores between fibres by a layer of rosin, and not a covering of the fibres themselves by an extremely fine layer.

B. P. RIDGE.

See also A., Dec., 1334, **Velocity of acid hydrolysis of cotton cellulose** (HUNTER). 1360, **Cellulose** (HESS and others). 1361, **Substances accompanying cellulose** (HESS and others). **True lignin** (FUCHS and HORN). 1362, **Degradation of the lignin and cellulose of wood** (FALCK and COORDT). 1387, **Structure of silk fibroin** (ABDERHALDEN and MAHN). 1402, **Transformation of cellulose into dextrose by bacteria** (WOODMAN and STEWART).

Bleaching of wood for matches. DATTA and BASU.—See XXII.

PATENTS.

Manufacture of artificial textile fibres. W. SEVER, JUN., and J. B. SPEAKMAN (B.P. 300,221, 9.7.27).—Artificial fibres exhibiting wave or curl similar to certain natural fibres are made by imparting a swirling

or eddying motion to the liquid or gaseous coagulating medium near the spinneret, or by the creation of eddy currents therein. F. R. ENNOS.

Improvement of vegetable fibrous material. L. LILIENTFELD (B.P. 300,663, 11.5.27).—Vegetable textile material (yarn or fabric) is treated with caustic alkali solution containing more than 12% of caustic alkali (calc. as NaOH) and carbon disulphide to form a superficial layer of cellulose xanthate, which latter is then partly removed by the action of a solution which is a solvent for cellulose xanthate, *e.g.*, caustic alkali solution. The fabric is then treated, optionally after washing, with, *e.g.*, 10% sulphuric acid to precipitate the viscose. If the fabric is stretched during the process a silky lustre is obtained without materially affecting the feel; if shrinkage is allowed the fabric acquires a wool-like character. Caustic alkali of less than 12% concentration may be used if the operation is conducted at temperatures below 0°. D. J. NORMAN.

Treatment of [fabric] material to render it gas-tight. B. G. TEXTILWERKE GES.M.B.H., and H. STROBL (B.P. 284,584, 30.1.28. Ger., 29.1.27).—The material is treated with several coatings of a mixture of viscose, gelatin, and glycerin, the proportions of which are varied in the different layers; the first and last layers contain a preponderance of gelatin, whilst the intermediate ones are richer in viscose. F. R. ENNOS.

Production of cellulose. WOLFF & Co. KOMMANDITGES. AUF AKTIEN, and R. WEINGAND (B.P. 288,997, 20.9.27. Ger., 19.4.27).—After removal of the hemicelluloses with alkali, the soda-cellulose is treated with a solution of a suitable salt, *e.g.*, sodium chloride, or an aliphatic alcohol to remove the bulk of the alkali, and is finally washed with water, whereby an after-formation of hemicellulose is avoided. F. R. ENNOS.

Cooking wood chips by the alkaline process of pulp manufacture. R. WOODHEAD, ASST. to V. D. SIMONS (U.S.P. 1,687,076, 9.10.28. Appl., 12.10.27).—The chips are heated in the digester first with black liquor (the alkaline solution used in a previous digesting operation) containing a little fresh digesting liquor; this is then drawn off and replaced by fresh digesting liquor for the final heating. F. R. ENNOS.

Separating or extracting cellulose or paper pulp from raw materials. (SIR) G. C. GODFREY, E. C. BENTHALL, E. S. TARLTON, H. F. WHEELER (BIRD & Co.), G. L. SCOTT, and E. SPENCER (B.P. 283,911, 8.7.27. India, 20.1.27).—On completion of an alkaline digestion of, *e.g.*, bamboo, grasses, etc. under pressure, steam is relieved from the top of the digester and injected at the bottom of another digester which is being loaded with fresh material, so that the steam is utilised to heat and, by intermittent or continuous vomiting, to rinse down the material as it is charged into the digester. [Stat. ref.] D. J. NORMAN.

Preparation of sulphite-cellulose. E. L. RINMAN (F.P. 627,884, 21.1.27).—Wood chips are heated at 130–140° with a solution of sodium bisulphite containing at least 1.2% Na₂O (preferably 2.5–6%, depending on the resin content of the wood). The sulphite liquor is made to circulate continuously through

another vessel, where the resin is removed by extraction with turpentine oil. F. R. ENNOS.

Production of cellulose from flax straw, hemp, jute waste, straw, etc. I. G. FARBENIND. A.-G. (F.P. 626,388, 17.12.26. Ger., 21.1.26).—The raw material (1000 pts.), mixed with 3500 pts. of a solution containing 180 pts. of sodium bisulphite, 54 pts. of sodium sulphite, and 10 pts. of magnesium chloride, is heated for 1 hr. at 110° and then for 10 hrs. at 145–150°. After removal of the liquor, the product is washed several times with water and worked up in the usual manner; it is very resistant and bleaches a pure white. F. R. ENNOS.

Treatment of waste sulphite-cellulose liquor. E. L. RINMAN (F.P. 627,752, 17.1.27).—The separated liquor is evaporated, and the organic constituents are burnt off with the addition of combustible materials to form sodium sulphide or a mixture of sodium sulphide and carbonate. After conversion of the sodium sulphide into hydroxide by means of metallic oxides, the precipitate of metallic sulphides is separated, and sulphur dioxide passed into the solution to regenerate sodium sulphite. F. R. ENNOS.

Recovering sulphur dioxide from waste [pulp-digestion] gases. G. A. RICHTER, ASST. to BROWN Co. (U.S.P. 1,685,754, 25.9.28. Appl., 14.7.23).—The contents of the sulphite digester while still hot are discharged into a blowpit and the hot sulphur dioxide and vapours liberated are passed in direct contact and in counter-current flow to cold water in a tower filled with inert material. The amount of water is so regulated that the bulk of the sulphur dioxide, which is cooled without being absorbed, can be passed on for absorption where required, whilst the small quantity of gas in the water is recovered by evacuation. F. R. ENNOS.

Manufacture of artificial threads and filaments by the dry-spinning method. G. B. ELLIS. From SOC. FABR. DE LA SOIE "RHODIASETA" (B.P. 300,672, 10.8.27).—Stable threads showing no scintillating effects may be spun at speeds of 400 m./min. or over with a filament denier of 1 or even less by combining the processes of B.P. 236,393 and 248,696 (B., 1926, 945), *viz.*, by heating the spinning solution before it reaches the spinning jet and introducing a cold drying atmosphere, *e.g.*, that of the spinning room at 20°, into the spinning cell in the vicinity of the dies. The lower part of the cell is heated to remove the last traces of solvent. D. J. NORMAN.

Manufacture of cellulose derivatives. I. G. FARBENIND. A.-G. (B.P. 275,660, 8.8.27. Ger., 6.8.26).—Alkali-cellulose in the pressed condition or in pulp, sheet, or paper form is treated with the esterifying or etherifying agent, which may be a suitable gas, *e.g.*, ethyl chloride, under pressure. F. R. ENNOS.

[Softener for use with] cellulose ester [coating] compositions. NOBEL INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 300,157, 5.5.27).—A mixture of glycerol, coconut oil, a lower fatty acid, *e.g.*, glacial acetic acid, and a little sulphuric acid is heated under a reflux condenser at a temperature at which the acetic acid boils vigorously, after which water and excess acetic acid are removed by distillation and

sulphuric acid by washing with sodium chloride solution. The product is heated at about 100° with animal charcoal and filtered, yielding a non-volatile oil, f.p. —5° to —13°, suitable for use instead of castor oil as a softener in the manufacture of artificial leather etc. L. A. COLES.

Manufacture of soluble cellulose esters of higher fatty acids. I. G. FARBERIND. A.-G. (B.P. 297,766, 31.3.28. Ger., 27.9.27. Addn. to B.P. 283,181; B., 1928, 637).—Untreated cellulose is esterified below 100° with the chloride of a higher fatty acid in the presence of a base such as pyridine, quinoline, etc., the temperature being subsequently raised above 100° until the desired solubility of the cellulose ester is attained.

F. R. ENNOS.

Preparation of paper and other similar surfaces. H. C. MITCHELL (B.P. 300,303, 10.8.27).—Paper, cardboard, etc. is coated with a layer of plain or coloured paraffin wax or shellac which is subsequently "fixed" by heating, so that portions may be removed by writing or scraping with a style etc. L. A. COLES.

Treatment of paper. D. GENESE, Assr. to W. H. OREM (U.S.P. 1,690,631, 6.11.28. Appl., 31.8.27).—The elasticity and shrinkage of paper are increased by moistening it with a concentrated solution of boric acid, and drying it under heat and pressure.

F. G. CLARKE.

Degreasing raw wool. R. BRAUCKMEYER (U.S.P. 1,693,106, 27.11.28. Appl., 14.1.26. Ger., 25.5.25).—See G.P. 445,792; B., 1928, 400.

Production of artificial yarns or threads. G. LARDY, Assr. to SOC. FABR. DE LA SOIE "RHODIASETA" (U.S.P. 1,693,759, 4.12.28. Appl., 6.4.26. Fr., 28.4.25).—See B.P. 251,580; B., 1926, 975.

Apparatus for boiling cellulose. C. HANGLEITER and A. SCHNEIDER, Assrs. to ZELLSTOFF-FABR. WALDHOF (U.S.P. 1,693,983, 4.12.28. Appl., 10.8.25. Ger., 26.8.24).—See B.P. 239,191; B., 1926, 268.

Filling boilers for cellulose with sulphite liquors. H. CLEMM, Assr. to ZELLSTOFF-FABR. WALDHOF (U.S.P. 1,693,999, 4.12.28. Appl., 12.1.21. Ger., 4.9.20).—See B.P. 168,866; B., 1922, 855 A.

Friction element (U.S.P. 1,692,136).—See I.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

See A., Dec., 1318, Absorption phenomena (RATELADE and TSCHETVERGOV). 1363, Aminofibroin (MOREL and SISLEY). 1393, Thiazine dyes as biological stains (HAYNES).

PATENTS.

Printing and discharging. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 299,210, 25.11.27).—The product of the action of alkali on iron carbonyl (or a mixture of iron carbonyl and alkali) is used as reducing agent in vat-dye printing pastes, or in discharge pastes for vat or direct dyes. The goods are subsequently passed through an oxalic acid bath to remove iron oxide.

C. HOLLINS.

Dyeing or colouring the hair of a pelt. R. R. BALUT (B.P. 300,352, 23.9.27).—Hair-covered pelts are coloured in imitation of spotted pelts such as those of leopards by applying at the proper positions pale and

deep shades of dyes so that the darker shades blend into the lighter shades; the application of these dyes is effected while the hair is displaced from its normal position on the pelt. A. J. HALL.

Dyeing, printing, or stencilling of materials made with or containing cellulose derivatives [esters or ethers]. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 298,993, 19.7.27).—Unsulphonated azo dyes having an *o*- or *peri*-aminonaphthol as end-component dye acetate silk, immunised cotton, etc. Examples are: 1:8-aminonaphthol coupled with diazotised aniline (red), aniline and *p*-nitroaniline (blue), or *p*-nitroaniline, reduced, and developed on the fibre with 2:3-hydroxynaphthoic acid (black), or with tetrastotised dianisidine (alkaline, blue-grey; acid, dark brown); and 2:3-aminonaphthol coupled with diazotised *p*-xylylene (red). Certain couplings are disclaimed. C. HOLLINS.

Protecting animal fibres. P. ONNERTZ and J. B. SCHWÄRZEL, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,692,950, 27.11.28. Appl., 22.6.25. Ger., 21.8.24).—See B.P. 238,857; B., 1925, 879.

Protection of animal fibres against effects of alkaline or acid media. R. S. HORSFALL and L. G. LAWRIE, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,693,926, 4.12.28. Appl., 31.10.27. U.K., 16.11.26).—See B.P. 285,554; B., 1928, 363.

Process and apparatus for mercerising textile fibres [in the form of sliver]. S. S. LORD, and TOOTAL BROADHURST LEE CO., LTD. (B.P. 300,186, 8.8.27).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Efficiency of different contact substances for the sulphuric acid contact process. II. B. NEUMANN and E. GOEBEL (Z. Elektrochem., 1928, 34, 734—740; cf. B., 1928, 891).—The work of previous investigators on the activity of iron oxide as a catalyst for the sulphuric acid contact process is reviewed, and a systematic study has been made, by means of the method described previously (*loc. cit.*), of the activity of a number of catalysts containing iron oxide. Using a gas mixture containing 7% SO₂, 10% O₂, and 83% N₂, the maximum yields obtained with the following catalysts were: iron oxide alone, 69.5% (at 625° and gas velocity of 150 c.c./min.) and 63.5% (650°; 300 c.c./min.); iron oxide containing 4% of bismuth oxide, 72.2% (625°; 150 c.c./min.); iron oxide with about 40% of potassium hydroxide, 52.1% (700°; 150 c.c./min.); iron oxide-strontium oxide, 71.2% (600°; 150 c.c./min.); iron oxide-tin oxide, 76.2% (600°; 150 c.c./min.); chromic oxide-tin oxide, 96.3% (475°; 150 c.c./min.) and 93.4% (500°; 300 c.c./min.); tin oxide, 35.2% (not maximum) (750°; 150 c.c./min.); titanium dioxide 49.0% (700°; 150 c.c./min.). The curves representing the results are all similar in form, and consist of an initial steeply rising portion leading to a sharply-defined maximum, followed by a gradual fall, the latter portion of the curves running parallel to the platinum curve (*loc. cit.*). The results with iron oxide alone are in agreement with those of Keppeler (B., 1908, 402). L. L. BIRCUMSHAW.

The Poupin process [of extraction of sodium nitrate]. DELCOURT, B. PINILLA, J. LATORRE, and J. BANCELIN (Caliche, 1928, 10, 299—311).—A series of official reports on a small works, extracting sodium nitrate from caliche, which has changed from the usual counter-current or Shanks system of extraction to the Poupin process. The necessary alterations in the plant are easily carried out, and the yield of sodium nitrate is increased from 50% with the old to 80% with the new process in favourable circumstances, the cost of labour, fuel, and power per unit of raw material remaining unchanged. The initial cost of the plant is estimated to be less than for the Shanks process. The new process is suitable for small-scale working, since a single boiling tank may be used. The boiling tank contains a perforated false bottom at a height of 1 m., and steam pipes run horizontally below this. The caliche is spread on the false bottom, the first layer only having been screened, and the lower part of the tank is filled with sodium nitrate liquor from a previous run ("preparative liquor"). Steam is then passed and the charge heated and moistened. From an automatic cistern above the centre of the tank hot liquor is distributed intermittently over the charge by perforated pipes and percolates through the caliche. At first mother-liquors are fed on to the charge, then washings of diminishing concentration, and finally fresh water. In the meantime liquor is run off from the bottom of the tank in portions—three portions of saturated solution, which are run into settling tanks and then into crystallising tanks, followed by "preparative liquor" and washings, which are used in another run as already described. Various devices for heat-interchange are employed, *e.g.*, in preheating the liquor fed to the boiling tank. The principal factors in lowering the cost of extraction are the decreased consumption of fuel due to the smaller quantity of water evaporated and the more complete exhaustion of the caliche. R. K. CALLOW.

Use of electrometric analysis in the preparation of bleach liquors. E. MÜLLER (Chem. Fabr., 1928, 677—678).—The chlorination of sodium hydroxide solutions to obtain sodium hypochlorite may be controlled by means of the apparatus described in G.P. 422,159 (B., 1926, 256), using a control solution of calcium hypochlorite rendered alkaline by addition of lime-water (cf. Müller, B., 1925, 628 A). Instead of using platinum electrodes an apparatus has been constructed in which two concentric graphite cylinders are used, the internal comparison electrode dipping into a small porous pot containing the calcium hypochlorite solution. A. R. POWELL.

Electrochemical manufacture of hydrogen peroxide. L. LÖWENSTEIN (Z. Elektrochem., 1928, 34, 784—786).—Various electrochemical methods for the production of hydrogen peroxide are described, with special consideration of the Weissenstein process (persulphuric acid formed at the anode) and the Pietzsch and Adolph process (ammonium persulphate formed at the anode). The relative merits of the two processes are discussed. Although the distillation yields are equal, the electrolytic yield is greater in the Pietzsch and Adolph process. On the other hand, the Weissenstein process has the advantage of greater simplicity. L. L. BIRCUMSHAW.

See also A., Dec., 1311, Desiccation of magnesium nitrate (LE BOUCHER). 1317, Adsorption of gases by sodium chloride and its aqueous solution (DURAN). Adsorptive power of certain adsorbents (ALEKSEEVSKI). 1318, Adsorption of iron from ferric hydroxide sols and ferric chloride solutions by kaolin and talc (FODOR and ROSENBERG). 1324, High-temperature fixation of nitrogen (KRASE and MACKAY). 1337, Preparation of potassium ferri-cyanide electrolytically (PAWECK and HIRSCH). 1341, Purification of helium (COOK). 1345, Volumetric determination of sulphuric acid (TESTONI). 1399, Toxicology of lead and its compounds (DANCKWORT and JÜRGENS).

Cottrell-Moeller process for mists. VER ECKE.—See I. Phosphoric acid solution for match sticks. DATTA and BASU.—See XXII. Use of chlorinated copperas. HEDGEPEETH and others.—See XXIII.

PATENTS.

Concentration of nitric acid. H. FRISCHER (B.P. 281,691, 1.12.27. Ger., 6.12.26).—Dilute nitric acid and a water absorbent, *e.g.*, sulphuric acid, are heated in a column having superposed sections which are indirectly heated, singly or in groups, at different temperatures, increasing in a downward direction as the concentration of the acid increases. W. G. CAREY.

Production of hydrocyanic acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,369, 13.10.27).—A mixture of carbon monoxide with 10—12% of ammonia is passed over catalysts composed of iron (cobalt or nickel) carbide; the catalyst may also contain molybdenum, manganese, tungsten, cerium, titanium, or copper, in a free or combined state, whereby the efficiency is improved. The carbides are obtained by melting the metals with finely-divided carbon. The reaction is carried out at below 600° (*e.g.*, 450°), so that the ammonia is converted with negligible loss; internal or external heating may be used. The hydrocyanic acid is removed either by condensation as ammonium cyanide or by absorption in caustic alkali solution, the residual gases being returned to the process. P. E. L. FARINA.

Synthetic production of ammonia from its elements. F. UIDE (B.P. 273,735, 30.6.27. Ger., 2.7.26).—Efficient catalysts of long life are obtained when they contain, in addition to aluminium, carbon, and iron, compounds of nitrogen (*e.g.*, ferro- or ferri-cyanides) and an alkali metal (preferably potassium), or its compounds. Thus aluminium chloride or sulphate (1 equiv.) and potassium ferrocyanide (1.2 equiv.) solutions are mixed, and evaporated *in vacuo* with stirring and exclusion of air till dry; the pulverised product is poured into a catalyst furnace, heated at 300—450°, with hydrogen and/or nitrogen (preferably under pressure), and used direct, water and gases containing oxygen being excluded. The catalyst is used at 350—450°, and may be revived by disintegration and pulverisation. [Stat. ref.] P. E. L. FARINA.

Manufacture of hydrogen peroxide. P. ASKENASY (B.P. 294,265, 21.7.28. Fr., 21.7.27).—Carbon dioxide under pressure acts on an aqueous suspension of barium peroxide, with the addition of hydrochloric acid or barium chloride to facilitate the filtration of the barium

carbonate and to act as preservative to the hydrogen peroxide. W. G. CAREY.

Manufacture of ferrous hydroxide. E. M. LOFLAND (U.S.P. 1,689,951, 30.10.28. Appl., 14.8.26).—Wet fragments of iron are exposed to the action of oxygen. The ferrous hydroxide coating is removed before it is converted into the ferric state, and may be preserved by adding an acid solution of an iron salt. Suitable apparatus is described. F. G. CLARKE.

Manufacture of finely-divided iron oxide. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 298,926, 14.7.27).—Finely-divided iron oxides suitable for pigments are obtained by heating ferric hydroxide at 110–150° and under 0.5–2.8 atm. for 2–3 hrs. with water, with or without the addition of alkaline hydroxides: a mixture of a ferric salt and a basic substance, such as sodium carbonate, lime, or chalk, may be used, the product being washed and dried. According to the conditions and duration of the treatment colours ranging from yellowish-red to dark violet are obtained. P. E. L. FARINA.

Preparation of an iron compound from solutions of iron [waste pickle liquors etc.] and its conversion into iron oxide. O. S. NEILL (B.P. 300,233, 9.8.27).—A product consisting principally of anhydrous ferrous sulphate is obtained in the form of hollow shells by bringing the atomised liquor in contact with a rapidly rotating element and simultaneously subjecting it to the action of a drying medium. The product can be calcined to yield ferric oxide, and the size of the particles of this can be reduced by subsequent crushing and washing with water. (Cf. B.P. 230,774 and 251,310; B., 1925, 880, 1926, 584.) L. A. COLES.

Magnesia product and its manufacture. T. S. CURTIS, Assr. to PACIFIC-SOUTHWEST TRUST & SAVINGS BANK (U.S.P. 1,690,771, 6.11.28. Appl., 7.4.25).—Magnesia is heated sufficiently to convert it without fusion into periclase. H. ROYAL-DAWSON.

Manufacture of potassium sulphate and ammonium chloride. S. LAMM (F.P. 627,299, 8.1.27. Belg., 11.2.26).—A solution of potassium chloride is treated with ammonia, sulphur dioxide, and an oxidising agent in the presence of a basic substance. A. R. POWELL.

Production of potassium magnesium sulphate and technical potassium sulphate. KALIWERKE ASCHERSLEBEN, and A. WITTE (G.P. 448,737, 13.7.24).—Carnallite containing a high proportion of kieserite is agitated with a cold magnesium chloride solution to obtain a residue of rock salt and a suspension of finely-divided kieserite, potassium chloride, and sodium chloride in a saturated magnesium chloride solution. This suspension is filtered and the residue treated with a solution of potassium chloride relatively free from sodium chloride, whereby the greater part of the sodium chloride is dissolved. Sufficient water is then added to the residual kieserite to hydrate it completely; the potassium chloride present then reacts with the hydrated kieserite forming magnesium chloride and the double salt, potassium magnesium sulphate, which crystallises out. Addition of more potassium chloride converts the double salt into potassium sulphate and

magnesium chloride, which are readily separated by crystallisation. A. R. POWELL.

Manufacture of trisodium phosphate. J. N. CAROTHERS, Assr. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,689,547, 30.10.28. Appl., 8.8.25).—Sodium carbonate is treated with phosphoric acid containing 50% H_3PO_4 or above, and the disodium phosphate formed is converted into trisodium phosphate by the addition of caustic soda. W. G. CAREY.

Manufacture of saline trisodium phosphate. C. F. BOOTH, A. B. GERBER, and P. LOGUE (U.S.P. 1,688,112, 16.10.28. Appl., 25.6.28).—A solution of phosphoric acid containing 4% of sodium chloride is neutralised (to methyl-orange) with sodium carbonate and, after expulsion of carbon dioxide by heating, the theoretical quantity of sodium hydroxide to produce trisodium phosphate is added. On cooling, a double salt of the composition $5(\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}) \cdot \text{NaCl}$ crystallises out, which is more stable and less caustic than the ordinary tribasic phosphate. A. R. POWELL.

Production of potassium salts and by-products. R. D. PIKE and R. CUMMINGS (U.S.P. 1,686,835, 9.10.28. Appl., 25.5.27).—Material (e.g., wyomingite) containing potassium is finely ground and is heated under pressure with a solution of common salt containing not more than 1% of sodium bicarbonate. W. G. CAREY.

Manufacture of solid calcium cyanide and double compounds thereof with ammonia. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,348, 19.9.27 and 2.5.28).—Calcium oxide or hydroxide or a calcium salt soluble in water is treated dry or suspended or dissolved in water and/or an organic liquid with hydrogen cyanide and ammonia to yield "calcium cyanide diammoniate." The product is removed by filtration or centrifuging, and is completely dried in a current of ammonia or by washing with a hygroscopic organic liquid, e.g., alcohol, followed by heating at about 130° *in vacuo*. The product is used direct or is decomposed into calcium cyanide and ammonia by heating at 200–300° under reduced pressure. L. A. COLES.

Production of high-percent. calcium cyanamide or mixtures containing it. N. CARO and A. R. FRANK (B.P. 279,811, 30.9.27. Ger., 27.10.26).—Calcium carbonate or material containing it, e.g., dolomite, with or without the addition of alkali carbonates, calcium oxide or fluoride, etc., is treated at the ordinary pressure with ammonia at a temperature not much exceeding, and not more than 200° below the dissociation temperature of the carbonate, i.e., at 700–900° for calcium carbonate or at 550–700° for dolomite. The apparatus is constructed of material free from catalysts which accelerate the decomposition of ammonia, e.g., of ceramic material or of quartz. L. A. COLES.

Production of the cyanamides of calcium and magnesium. N. CARO and A. R. FRANK, Asses. of STRICKSTOFFWERKE GES.M.B.H. (B.P. 279,812, 30.9.27. Ger., 28.10.26).—Calcium oxide, prepared by heating the carbonate in a current of neutral gas, is heated at 750–780°, or magnesium oxide is heated at 650–750°,

in a current of hydrogen cyanide, alone or in admixture with a neutral gas or with ammonia. L. A. COLES.

Manufacture of calcium arsenate. C. ELLIS and V. T. STEWART, Assrs. to CHADELOID CHEM. CO. (U.S.P. 1,690,627—8, 6.11.28. Appl., [A] 5.3.23, [B] 16.10.24. Renewed [B] 4.4.28).—(A) Powdered quicklime is treated with a solution of arsenic acid containing more water than is necessary to hydrate the lime; the product is dried by adding more quicklime. (B) The quicklime and arsenic acid are ground in the presence of insufficient water to render the product pasty. F. G. CLARKE.

Recovery of hydrochloric acid from the end-liquors obtained in the manufacture of fuller's earth etc. E. MAAG (G.P. 449,993, 16.6.26).—The liquors are evaporated at a relatively low temperature, while a current of air is passed over the surface so as to remove excess water and leave as concentrated acid as possible. The concentrated liquors are then passed to a distillation apparatus in which the hydrochloric acid is distilled from the dissolved salts and is recovered in a sufficiently concentrated form for use again in the process. A. R. POWELL.

Production of alumina while simultaneously obtaining hydrochloric acid and alkali compounds. A. L. MOND. From I. G. FARBENIND A.-G. (B.P. 300,184, 4.8.27).—Aluminous materials such as clay, bauxite, etc. are dissolved in sulphuric acid, separated from insoluble residues (silicic acid etc.), and an amount of alkali (usually sodium) chloride is added equivalent to the sulphuric acid employed; the mixture is evaporated and heated to 700° whereby hydrochloric acid is evolved and a mixture of alkali sulphate and alumina (together with iron) remains. The mixture is next heated at 1000°, preferably in a rotary drum furnace, with carbon, so that part of the alkali sulphate is reduced to sulphide and a little carbonate, while the remainder forms aluminate. After lixiviation and filtration, about 40% of the sulphide is first separated and the alumina precipitated by the regulated addition of hydrogen sulphide and/or carbon dioxide. More sulphide (80–85%) is crystallised from the remaining liquor, which can then be used for further lixiviation, or for the production of soda *via* the bicarbonate. By using carbon dioxide alone and not allowing the sulphide to crystallise out in the first place, soda may be produced direct. Also by using a mixture of sulphuric and hydrochloric acids greater relative yields of alumina are obtained, as the amount of alkali chloride added is always equivalent to the sulphuric acid used. P. E. L. FARINA.

Manufacture of aluminium chloride. A. M. McAFEE and G. I. ROBERTS, Assrs. to GULF REFINING Co. (U.S.P. 1,690,990, 6.11.28. Appl., 26.9.27).—A mixture of aluminous material and carbon is disposed in a reaction chamber, so that the mass is denser at the walls than elsewhere, and chlorine and a gas supporting combustion are passed through the mass under conditions to form aluminium chloride, and so that the gases preferentially travel in a path away from the walls. W. G. CAREY.

Manufacture of tribarium aluminate. G. W. MOREY (U.S.P. 1,688,054, 16.10.28. Appl., 23.11.25).—A mixture of barium carbonate or sulphate and alumina

or a compound which yields alumina on heating is heated at 1150–1400° until reaction ceases. The product is soluble in water and barium hydroxide may be crystallised from the solution. A. R. POWELL.

Manufacture of base-exchange silicate. E. NORDELL, Assr. to PERMUTIT Co. (U.S.P. 1,687,036, 9.10.28. Appl., 21.11.25).—The gel formed by the reaction between solutions of a soluble aluminium salt of an organic acid and an alkali silicate is partially dried, leached, broken up, and screened. W. G. CAREY.

Adsorbing agent. A. ROSENHEIM (B.P. 289,418, 12.7.27. Ger., 27.4.27).—Silicic acid sols, produced by the treatment of zeolites, water-glass, etc., with acid, are mixed with iron, aluminium, magnesium, or titanium salts (generally chlorides). Silica gel gradually separates out, and is dried at temperatures up to 330°, then extracted at ordinary or, preferably, increased temperature with water or 1–10% hydrochloric, acetic, or sulphuric acid to remove metallic compounds, and again dried. Drying may also be effected in a stream of heated gases (air, carbon dioxide, carbon monoxide, nitrogen, etc.). If desired, the original mixture of silicic acid and metal salt is treated with a regulated amount of soda lye, a mixed gel being obtained which can then be freed from the metallic oxides by acid washing. P. E. L. FARINA.

Manufacture of cobalt carbonyl. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,714, 28.7.27. Addn. to B.P. 250,132; B., 1926, 487).—Metallic cobalt or reduced cobalt ore is treated with carbon monoxide at high temperature and pressure (e.g., 150–170° and 200 atm.). The bulk of the cobalt carbonyl is separated prior to the release of pressure at not less than 50°, and the remainder by passage through a solvent or by cooling to a lower temperature in an apparatus the narrow portions of which are maintained above 50° by the hot reaction gases. W. G. CAREY.

Protection and fireproofing of materials. C. LICHTENSTADT (U.S.P. 1,691,726, 13.11.28. Appl., 10.2.28).—A composition of zinc ammonium phosphate, monoammonium phosphate, and ammonium chloride is used. H. ROYAL-DAWSON.

Separating sulphur from other materials. K. M. BAUM (U.S.P. 1,689,545, 30.10.28. Appl., 3.9.27).—Crude sulphur is melted in the presence of an aqueous solution of zinc chloride having a sp. gr. greater than that of molten sulphur and less than that of the impurities at the same temperature. The upper layer containing the sulphur is removed. F. G. CLARKE.

Treatment of sulphur. J. W. SCHWAB, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,692,422, 20.11.28. Appl., 16.3.27).—Molten sulphur contaminated with hydrocarbons is agitated with anhydrous aluminium chloride and separated from the combined impurities. H. ROYAL-DAWSON.

Recovery of iodine from crude sodium nitrate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,670, 14.7.27).—The crude nitrate is heated in a rotary tube furnace at 200–250° in a current of gas, and after the iodine has volatilised the temperature is raised to decompose the nitrate. W. G. CAREY.

Production of nitric acid from ammonia. L. H. GREATHOUSE, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,692,298, 20.11.28. Appl., 6.9.23).—See B.P. 221,513; B., 1925, 713.

Furnace for the decomposition of alkali chlorides. J. KERSTEN (U.S.P. 1,692,175, 20.11.28. Appl., 19.9.24. Ger., 6.10.23).—See B.P. 243,104; B., 1926, 88.

Crystallisation of solid substances [rock-salt] in a coarse granular form from solutions. I. ISAACHSEN, Assr. to A./S. KRYSTAL (U.S.P. 1,693,786, 4.12.28. Appl., 10.9.25. Norw., 17.9.24).—See B.P. 260,133; B., 1927, 11.

Manufacture of alkali cyanides. S. COULIER (U.S.P. 1,691,960, 20.11.28. Appl., 18.2.26. Belg., 24.2.25).—See B.P. 265,305; B., 1927, 251.

Manufacture of aluminium sulphate. L. G. PATROUILLEAU, Assr. to Soc. ALUMINE & DÉRIVÉS (U.S.P. 1,692,692, 20.11.28. Appl., 23.9.25. Fr., 29.9.24).—See B.P. 240,435; B., 1925, 990.

Base-exchange compound and its manufacture. H. J. WHEATON, Assr. to AMER. DOUCHÉ Co. (U.S.P. 1,694,155, 4.12.28. Appl., 3.11.21. U.K., 5.7.21. Renewed 4.5.28).—See B.P. 177,746; B., 1922, 372 A.

Purifying gases containing phosphorus. G. PISTOR and E. BORSBACH, Assrs. to I. G. FARBERIND. A.-G. (U.S.P. 1,692,787, 20.11.28. Appl., 8.12.25. Ger., 6.2.25).—See B.P. 247,219; B., 1927, 556.

Fire-extinguishing foam (B.P. 299,097).—See I. Mixtures of hydrogen and carbon monoxide (B.P. 300,328). **Hydrogen from gaseous mixtures** (B.P. 299,167).—See II. **Catalysts for methyl alcohol synthesis** (B.P. 272,864).—See III. **Phosphorescent compositions** (B.P. 299,228).—See XIII. **Fertilisers** (B.P. 300,402). **Mixed fertiliser** (B.P. 300,329).—See XVI. **Denitration of waste sulphuric acid mixtures** (B.P. 276,972). **Azides** (B.P. 300,401).—See XXII.

VIII.—GLASS; CERAMICS.

Transmissive properties of eye-protective glasses and other substances. W. W. COBLENTZ and R. STAIR (U.S. Bur. Stand. Tech. Paper No. 369, 1928, 22, 555—578).—Transmission curves of optical crown glass, several commercial eye-protective glasses, five makes of glass partially opaque to ultra-violet light, for use in sunlight, and of a series of cobalt-cerium-iron glasses are given. In addition, the transmissions of several miscellaneous materials, viz., mother-of-pearl, sextant, and signal-red glasses, heat-transmitting glass, greenish-brown glass, and the organic materials "polopas" and "protectoid" have been included. Animal tissue, 0.5 mm. thick, was found to be quite opaque to light of short wave-length, but of rapidly increasing transparency as the wave-length became greater. Average samples of colourless, vitreous silica were much more opaque than was quartz to ultra-violet radiations of less than 240 μ . A formula for specifying the shade number of protective glasses is given, based on experimental tests, together with a table of transmissions and tolerances corresponding to shade numbers, as proposed by a Federal specification committee. A. COUSEN.

Effect of lepidolite in a high-tension electrical porcelain body. R. TWELLS (J. Amer. Ceram. Soc., 1928, 11, 644—648).—Lepidolite (analysis given) showed a refractoriness of cone 02 (1110°), whilst this value for potash felspar was cone 8 (1290°). No eutectics in the felspar-lepidolite series were found, but mixtures of these two minerals possessed larger ranges of deformation with temperature than either alone. A series of bodies containing lepidolite was obtained by blunging the ingredients and drying on plaster moulds. From these bodies test cups were made, and these were fired to cone 9. It was shown that the vitrification range of a standard body is greatly reduced by the replacement of felspar by lepidolite. Small amounts of this mineral should promote the early vitrification of the body without greatly affecting the firing range. Lepidolite increased the resistance of the ware to thermal shock, whilst the whiteness and general appearance were not impaired.

A. T. GREEN.

Kilns, furnaces, bodies, and glazes for small commercial production and educational work. P. E. COX (J. Amer. Chem. Soc., 1928, 11, 649—657).—Descriptions of a small test kiln and a frit kiln, together with the constitutions of suitable glazes, are given, and methods of producing copper-red glazes are discussed.

A. T. GREEN.

Effect of reducing gases on the transverse strength of fireclay bricks. A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1928, 11, 633—638).—Three preliminary experiments, in which small specimens (1×1×9 in. for transverse strength and 2½×2½×4½ in. for compressive strength measurements) were packed in crushed coke in saggars and heated in an atmosphere of "city" gas (carburetted water-gas) either to 550° or 1100°, were carried out. Of the eight brands tested in this way three showed definite disintegration by shattering after treatment at 550°. No bricks showed disintegration after treatment at 1100°. In another series of tests batches of sixty full-sized bricks representing three brands were subjected to the above reducing conditions at 550° and at 1100°, whilst a third batch was reheated to 1125° in a normal atmosphere. After treatment at 550° two of the three brands suffered pronounced decrease in strength, but the third was not appreciably affected. Reducing conditions at 1100° had little effect on the bricks. Examination of the bricks treated at 550° showed that carbon had accumulated at the "iron" spots, and that the cracks causing the disintegration radiated from these areas.

A. T. GREEN.

Electric furnaces in the ceramic industry. S. R. HIND (World Power, 1928, 10, 599—607).

Action of fuel ash on refractories. PACUKOV.—See II.

PATENTS.

Manufacture of light-diffusing glass. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 287,545, 9.2.28. Ger., 24.3.27).—Clear glass is pulverised in a ball mill, sintered just to form a viscous mass, and shaped with the blowpipe for the making of objects such as incandescence lamp bulbs. The powder may be compressed

before sintering, in order to prevent flaws, and an opalising agent may be added, if desired, to the powdered glass. A. COUSEN.

Seal for hard glass. W. B. GERO, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,691,436, 13.11.28. Appl., 28.7.26).—A tungsten-molybdenum alloy serves as a conductor and seal for the hard-glass container.

H. ROYAL-DAWSON.

Manufacture of abradant material. BRIT. THOMSON-HOUSTON Co., LTD., Assecs. of L. E. BARRINGER (B.P. 281,711, 3.12.27. U.S., 3.12.26).—Mixtures of abrasive material with a glycerol-phthalic anhydride resin, with or without the addition of a plasticising agent, are moulded at about 150°, cooled to about 75°, discharged from the moulds, and cured at the ordinary pressure by prolonged heating at a temperature rising from 75° to about 190°. Alternatively, powdered abrasive material is applied to cloth or paper coated with a solution of the resin and a plasticising agent, and the resin is hardened by heat. L. A. COLES.

Manufacture [mounting] of abrasive articles. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 300,417, 21.12.27).

IX.—BUILDING MATERIALS.

Loosening of cement. I. H. GESSNER (Kolloid-Z., 1928, 46, 207—216).—A critical discussion of the literature of the last six years on the subject.

E. S. HEDGES.

See also A., Dec., 1928, **Hydration of dehydrated gypsum** (BUDNIKOV). **Dehydration of gypsum at various temperatures** (BUDNIKOV).

PATENTS.

Apparatus for making a cellular cement product. G. B. HINTON (B.P. 298,847, 27.2.28).—Cement pulp and a froth-flotation agent are fed together with air into the annular space between an outer tank and an inner concentric vessel, and the mixture is agitated by the rotation in the annular space of a single perforated cage fitted with vanes, or by two such cages rotating in opposite directions to overcome centrifugal action. Alternatively, the inner shell is rotatable and carries perforated plates alternating with those on a cage rotating in the opposite direction. W. G. CAREY.

Apparatus for making a spumous mass of cementitious material. Manufacture of a cellular cementitious product. G. B. HINTON (B.P. 300,842—3, 27.2.28).—(A) A mixture of cement, water, and a froth-flotation agent, with or without the addition of sand etc., is stirred in a tank provided at the bottom with an air inlet, the stirring apparatus comprising alternating perforated plates rotating in opposite directions. (B) Cement mixtures aerated as above to contain relatively large cells are mixed with mixtures containing smaller cells to form a product containing large cells surrounded with adherent smaller cells. L. A. COLES.

Curing cementing materials. L. MELLERSH-JACKSON. From BARBER ASPHALT Co. (B.P. 300,464, 23.4.28).—Evaporation from cement concrete during curing is prevented by applying to the surface, after

laying but before complete setting, a water-external-phase bituminous emulsion. W. G. CAREY.

Manufacture of finishing lime. V. M. FREY (U.S.P. 1,689,237, 30.10.28. Appl., 12.2.25).—Ground dolomite is decarbonated without admixture with other solid substances by agitating it at a temperature below that of sintering, and the product is hydrated.

W. G. CAREY.

Manufacture of magnesite bricks. B. M. O'HARRA and E. A. SLAGLE, Assrs. to AMER. SMELTING & REFINING Co. (U.S.P. 1,686,876, 9.10.28. Appl., 17.9.26).—Calcined magnesite is crushed and mixed with water containing magnesium chloride equivalent to 5—10% of the magnesite; sodium hydroxide or ammonia solution is then added equivalent to the magnesium chloride, the mixture is compressed, and the bricks are burnt to convert the magnesium hydroxide into oxide and finally into periclase.

W. G. CAREY.

Removing chemicals and moisture from lumber, wood, etc. H. C. BABEL, Assr. to M. L. BABEL (U.S.P. 1,687,822, 16.10.28. Appl., 5.1.25).—The material is placed in a retort wherein it is subjected to the action of a continuously-circulating heated medium which passes horizontally through it, returning *via* the bottom of the retort, where the lowest stratum of the medium is exhausted.

A. B. MANNING.

Manufacture of aluminous cements. L. G. PATROUILLEAU, Assr. to SOC. ALUMINE & DÉRIVÉS (U.S.P. 1,694,338, 4.12.28. Appl., 10.8.25. Fr., 19.12.24).—See B.P. 232,898; B., 1925, 549.

Apparatus for treating [seasoning] wood with ozone. G. LYON (U.S.P. 1,693,486, 27.11.28. Appl., 16.1.23. Fr., 27.1.22).—See B.P. 192,372; B., 1924, 58.

Bituminous emulsions (B.P. 298,842, 300,414—5, 300,821).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Soil corrosion studies. I. K. H. LOGAN, S. P. EWING, and C. D. YEOMANS (U.S. Bur. Stand. Tech. Paper No. 368, 1928, 22, 447—554).—An investigation, not yet complete, was undertaken on the corrosion of metals by soils in the absence of electric currents. Specimens buried included cast iron and steel pipes (bare or protected), cable sheaths, and specimens of other metals under service conditions. Classification of American soils is described in detail. They are arranged in groups dependent on climatic influences, the classification within the groups depending on physical structure together with geological origins. The pH values, soluble salt contents, and resistivities of the soils used in the tests are given. Corrosion rates were determined by loss in weight over periods from 1 year upwards. Little difference was found in the corrosion of welded iron and steel pipes under similar conditions. Where pitting of cast iron occurred the product was of graphitic type. Later tests were carried out in 6 typical soils only. In about 60% of cases rate of corrosion decreased with time, and in the remainder it increased. On the whole it tended to become more uniform. High-silicon cast

iron showed little corrosion in any soil. All commonly used pipe materials are, however, liable to severe corrosion in some soils, apart from electric currents or the presence of foreign matter in the soil. The source of the corrosion cannot be determined by the appearance. The soil is of more importance than the pipe material. Corrosion is particularly heavy with high or low values for p_{H^+} in the soil. Galvanised material resisted the action of most soils under test fairly well, but exceptions occurred. In clay galvanising gave good protection. Asphalt coatings on pipes were found in a number of cases to show deterioration leading to corrosion; in some cases the adhesion was lost. Pitch adhered better, but tended to flow. Little difference in value was found between antimonial lead and pure lead. Brass, except galvanised nipples, showed little corrosion whatever its composition. In coarse soils the distribution of corrosion is associated with that of moisture. Corrosion increases with temperature. Surface potential differences were detected between oxidised metal in a pit and the untouched metal, and these result in the widening but not the deepening of the pit. C. IRWIN.

Steel for case-hardening—normal and abnormal steel. S. EPSTEIN and H. S. RAWDON (U.S. Bur. Stand. J. Res., 1928, 1, 423—466).—Ehn's theory that soft spots on case-hardened articles are due to the steel used is confirmed experimentally. The microstructures characteristic of normal and abnormal carburising steels are described. Abnormal steel tends to give soft spots on quenching in water, but can be quenched satisfactorily in brine. Abnormality in commercial steel was associated with the use of aluminium as a deoxidiser. The results are consistent with the theory that abnormality is caused by the presence of finely-divided oxides. C. J. SMITHELLS.

Anomalies in the structure of steel. E. PIVOVARSKY (Stahl u. Eisen, 1928, 48, 1665—1669).—A number of cases of the formation of a peculiar structure in mild steel after a recrystallisation anneal are discussed. Samples of steel which had been deoxidised with aluminium or with ferrovanadium developed a structure on annealing in which the ferrite had segregated in wide-meshed networks enclosing areas of pearlite, and in some cases large ferrite areas were distributed irregularly through the specimens. Similar steels deoxidised with ferrosilicon or cast without a deoxidising agent developed the usual dendritic ferrite structure on annealing. No explanation of the effect of aluminium and vanadium could be discovered. A. R. POWELL.

Strain markings in mild steel under tension. H. S. RAWDON (U.S. Bur. Stand. J. Res., 1928, 1, 467—485).—Observations of strain markings developed on the surface of polished specimens of low-carbon steels stressed in tension show that they coincide with the yielding of the metal, and indicate closely the proportional limit. Strain markings first appear near the ends of the reduced section of a test piece and progress towards the centre. When loaded several times in succession, the strain markings which develop at each new loading are continuations of those already formed. The hardness is increased 5% locally by the straining which results in strain markings. X-Ray examination

shows no change in the material. Etched specimens develop slip-bands in certain grains at right angles to the direction of applied stress, and a general tilting of neighbouring grains results in the characteristic rippled-surface appearance of strain markings on steel.

C. J. SMITHELLS.

Physical properties of 72/28 and 90/10 brasses as functions of the final working and the preceding heat-treatment. J. M. FERNÁNDEZ-LADREDA (Anal. Fis. Quím., [Teen.], 1928, 26, 81—112).—Ordinary technical specimens of 72/28 and 90/10 brasses, used for cartridge cases and bullet casings, respectively, were submitted to processes of rolling and annealing, and changes in the metal were followed by determinations of tenacity, ductility, and hardness and by microscopical examination. The physical properties depend on the mechanical working preceding the final annealing, but these differences disappear at final annealing temperatures of 500—600° for the 72/28 brass and 350—400° for the 90/10 brass. The physical properties depend similarly on the annealing which precedes the final working. The differences which are caused by variation in the annealing temperatures in the preparatory processes disappear at a final annealing temperature of from 300 to 500° (according as much or little reduction in thickness has been made) for 72/28 brass, and at 250—300° for 90/10 brass. Hardness tests are a valuable means of controlling the manufacture and testing of samples, using for thin plates of metal Baby Brinell and Rockwell testing machines. The best series of rolling and annealing treatments were determined for the preliminary manufacture in which ingots of 32 mm. thickness are reduced to 3.35 mm. in the case of 72/28 brass and 1.55 mm. in the case of 90/10 brass. R. K. CALLOW.

Wear and mechanical properties of railroad bearing bronzes at different temperatures. H. J. FRENCH, S. J. ROSENBERG, W. LE C. HARBAUGH, and H. C. CROSS (U.S. Bur. Stand. J. Res., 1928, 1, 343—421).—The wearing and mechanical properties of two groups of copper-tin-lead alloys in both chill-cast and sand-cast conditions were tested under rolling and sliding friction, with and without lubrication up to 175°, and under tension, impact, and pounding up to 315°. None of the tests alone yielded sufficient information for general comparisons. Variations in chemical composition produced major changes in properties, but over small ranges were less important than variations in method of casting. Chill-cast bronzes were less resistant to wear and impact than sand-cast bronzes, but more resistant to pounding and tension. Increase in lead from 0.25% to 10% improved wearing properties, but lowered resistance to pounding, impact, and tension. Increase in tin improved all properties. Increase in temperature decreased the resistance in all tests, particularly above 175°. C. J. SMITHELLS.

Effect of temperature on the drop hardness of nickel. F. SAUERWALD [with A. FISCHNICH and G. NEUENDORFF] (Z. Metallk., 1928, 20, 408—409).—The drop hardness of nickel decreases slowly up to 300°, then rapidly between 300 and 400°, rises to an ill-defined maximum at 450°, again falls rapidly between 600° and 700°, rises slightly at 800°, and finally falls rapidly once

more. The changes between 300° and 400° are ascribed to the same cause as is the magnetic transformation in this temperature range, and the maximum at 450° to a similar phenomenon to that causing "blue-brittleness" in iron. No explanation of the other changes could be found.

A. R. POWELL.

Anomalous properties of eutectic alloys of high dispersion. N. V. AGEEV, S. A. POGODIN, and N. S. KURNAKOV (Ann. Inst. Anal. Phys. Chem., 1928, 4, 23—38).—The hardness of rapidly and of slowly cooled alloys of tin and lead and of silver and copper is compared; the greatest differences between the values was found for the eutectic alloys. The same alloys possess the lowest values for electro-conductivity, for which they have also the highest temperature coefficient. The coefficients of linear expansion of the alloys lie on a smooth curve. The extent to which any of the physical properties of an alloy possess anomalous values at the eutectic point varies for different systems, and depends on the degree of dispersion of the crystals of the alloy.

R. TRUSZKOWSKI.

Electrometallurgy of gold. W. GRAULICH (Chem. Fabr., 1928, 678—680).—After a brief discussion of the Wohlwill process for the electrolytic refining of gold, data are calculated from a specific example for the current consumption, daily output of refined gold, anodic corrosion, time required before replacing anodes and cathodes, and interest charges on the gold during refining.

A. R. POWELL.

"Galvanising" with cadmium. G. DE LATTRE (Rev. Mét., 1928, 25, 630—636).—A cadmium bath maintained at about 380° is operated at a lower temperature than that of the ordinary galvanising bath, and the dipping end of the bath is covered with a mixture of zinc ammonium chloride and glycerin, the metal during melting being protected from oxidation by wood charcoal. The coating of cadmium on iron or copper is adherent and ductile, and possesses a silvery lustre. The fusion point is above that at which the wiping process could be carried out under olive oil, but this process is possible if an alloy of cadmium and zinc, lead, or tin is substituted for the pure metal, e.g., the eutectic alloy 83% Cd, 17% Zn (m.p. 266.5°). The hardness of an alloy coating is superior to that of pure cadmium, and the resistance to corrosion satisfactory if both constituents of the eutectic are electropositive to the base metal to be protected.

C. A. KING.

See also A., Dec., 1313, **Solid solutions of chromium and nickel and of iron and nickel** (BLAKE and others). 1314, **Magnetic saturation of iron carbide** (STÄBLEIN and SCHROETER). 1319, **Passivity of metals** (MÜLLER). **Permeability of metals towards gases** (LOMBARD). 1327, **Copper-tin alloys** (BRONIEWSKI and HACKIEWICZ). 1328, **Copper-tin system** (ISHIHARA). **Molybdenum-carbon system** (TAKEI). **Iron-iron sulphide system** (MIYAZAKI). **Iron sulphide-manganese sulphide system** (SHIBATA). 1344, **Iron crystals** (MCKEEHAN). 1347, **Determination of traces of lead** (SCHMIDT). **Determination of copper and zinc** (ROBERTSON). **Detection of copper in presence of iron** (SZEPELLÉDY).

Corrosion of condenser tubes. PHILO.—See XXIII.

PATENTS.

Metallurgical furnace. A. J. BRIGGS, Assr. to ONONDAGA STEEL Co., INC. (U.S.P. 1,687,925, 16.10.28. Appl., 16.10.26).—An indirect arc furnace comprises a horizontal rotating cylinder lined with refractory material and provided at one end with a stationary door having an opening through which one electrode may be introduced into or removed from the furnace and at the other end with an opening through which the second electrode is passed. The door is so mounted that when the electrode is withdrawn the whole may be swung away laterally.

A. R. POWELL.

Rotary-hearth furnace. Car-bottom furnace-hearth seal. Furnace. J. F. BAKER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,691,367—9, 13.11.28. Appl., [A] 18.6.27, [B] 25.6.27, [C] 14.7.27).—(A) An annular hearth projects through the bottom wall of the annular furnace chamber, and means are provided for rotatably supporting the hearth in operative position and for lowering the hearth out of engagement with the furnace to afford access to the furnace chamber. (B) In a car-bottom furnace having a horizontally removable hearth, the seal between the rear edge of the hearth and an adjacent wall of the furnace chamber comprises a sand trough carried by the hearth and a flange pivotally fastened to the wall. Means co-operating with movement of the hearth are provided for moving the flange into operative position with respect to the trough. (C) Means are provided for permitting relative vertical movement between a number of furnace walls and the hearth, for providing liquid and sand seals between the hearth and walls in operative positions, and for artificially cooling the seals.

J. S. G. THOMAS.

Cupola furnaces. J. W. JACKMAN & Co., LTD., and F. W. NEVILLE (B.P. 300,405, 8.12.27).—The area of the outlet of a tuyère is made adjustable by means of a "false" top plate pivoted at one end and operated by means of a quadrant and worm. As the division plate is moved, an attached damper plate closes the free space at the inlet end of the tuyère.

C. A. KING.

Apparatus for agglomerating and roasting minerals. PROGRÈS MINIER ET MÉTALLURGIQUE SOC. ANON. and A. DE SAMSONOV (B.P. 283,133, 21.12.27. Belg., 4.1.27).—The material, supported on a grate, is confined between walls extending along at least two sides of the grate, and, after treatment, is completely discharged by one movement of the grate across the furnace, or by one movement of a pushing element across the grate.

M. F. NOTTAGE.

Reduction of oxides to metallic form. S. WESTBERG (U.S.P. 1,690,916, 6.11.28. Appl., 16.7.25).—Oxides or oxide ores mixed with carbonaceous material are heated to reaction temperature, and the carbon monoxide formed is converted into hydrogen or a hydrogenous gas and introduced into the reaction zone.

W. G. CAREY.

[Annealing] furnaces. F. W. MANKER, Assr. to SURFACE COMBUSTION Co., INC. (U.S.P. 1,688,393—4, 23.10.28. Appl., 12.9.25).—(A) The heating chamber of the furnace is provided with a carriage to hold the charge running on rails in such a way that it is nearer

one side wall than the other. In front of the more remote wall a second wall is erected extending nearly to the roof of the furnace, and forming with the side wall a combustion chamber into which the gas flame is directed. (b) The heating chamber has an opening in one end wall over which combustible gases are passed and a working door in the other end wall. The products of combustion are passed transversely below the floor of the chamber through a conduit which opens into the chamber at the corners on either side of the working door and from which branch conduits lead towards the opposite end of the chamber. The waste gases pass through a recuperator on their way to the stack, and the preheated air from the recuperator is passed through conduits to and through the end wall over which the combustible gases pass.

A. R. POWELL.

Production of metals in electric furnaces.

E. G. T. GUSTAFSSON, Assr. to H. G. E. CORNELIUS (U.S.P. 1,691,439, 13.11.28. Appl., 31.3.26. Swed., 16.5.25).—The metalliferous charge in the electric furnace is smelted so as to form metal and slag. The furnace is then emptied, and on the bare bottom of it a quantity of slag in its original tapped condition is reintroduced as a protection against the action of the electric arc, and in readiness for the next charge.

M. E. NOTTAGE.

Concentration of ores. E. H. SNYDER and W. D. GREEN, Assrs. to COMBINED METALS REDUCTION CO. (U.S.P. 1,691,310, 13.11.28. Appl., 2.7.25).—Ores containing at least one of the metals of the group silver, lead, copper, are treated with an alkali thiosulphate to dissolve at least a portion of any of these metals, if present, the metal content of which is then precipitated as sulphide from the solution; the precipitate, together with any natural sulphides in the ore and any mineral particles to which a precipitated sulphide adheres, is then separated from the gangue by means of froth flotation, the solvent being regenerated by the action of the precipitant on the ore solution.

M. E. NOTTAGE.

Flotation concentration. R. E. PHELAN and S. P. LOWE, Assrs. to R. H. CHANNING, JUN. (U.S.P. 1,690,907, 6.11.28. Appl., 18.10.27).—Minerals containing mica and talc are separated by froth flotation from concentrates by mixing starch with the ore pulp and making the mixture non-alkaline to prevent the mica and talc from entering the froth.

W. G. CAREY.

Froth-flotation concentration of ores. F. SWINNEY (U.S.P. 1,688,998, 23.10.28. Appl., 24.5.27).—The aerated pulp is fed into the cell from both sides so that the opposing streams meet in the middle of the cell, whereby vigorous agitation and efficient aeration are obtained. The tailings outlet is placed at one end of the cell so that the general course of the pulp through the cell is in a direction at right angles to the incoming streams.

A. R. POWELL.

Differential flotation process for mixed sulphide ores. K. KAWAMURA, Assr. to Y. YOKOYAMA (U.S.P. 1,688,277, 16.10.28. Appl., 18.1.26).—The neutralised pulp is treated with chromic acid solution sufficient to inhibit flotation of iron and copper pyrites, and the zinc is then floated as usual.

A. R. POWELL.

Wet treatment of lead-bearing materials involving filtration. S. C. SMITH (B.P. 298,684, 5.4.27. Addn. to B.P. 264,569; B., 1927, 256).—For the extraction of lead from complex ores by chloridising etc. as previously described (*loc. cit.*), the ore and acid are agitated in a rubber-lined, horizontal, revolving steel drum containing a woven rubber fabric or perforated rubber filtering material supported away from the rubber walls of the cylinder by means of wooden slats. Provision is made for intermittent or continuous circulation of the leaching material through the cake, for re-pulping and re-forming the cake, and for removing the leach liquor and circulating wash liquors through the cake to extract the lead chloride formed.

A. R. POWELL.

Treatment of iron ores. A. HOLMBERG (B.P. 300,438, 11.2.28).—In order to increase the magnetic properties of low-grade iron ores, the ore, mixed with powdered fuel, is reduced partially by the controlled combustion of the fuel so as to prevent sintering. Reducing gases, *e.g.*, producer gas, may be used to increase the action of the solid fuel, and the differential pressure is reduced as combustion proceeds, to maintain a constant flow of gases through the charge.

C. A. KING.

Casting of ferrous metals. P. W. and E. B. SHIMER (U.S.P. 1,687,799, 16.10.28. Appl., 7.8.25).—A chaplet for use in the casting of ferrous alloys is provided with a coating of an iron alloy which melts at or below the casting temperature, *e.g.*, with a mixture of equal weights of powdered ferrophosphorus and ferromanganese fused on to the chaplet under a suitable flux.

A. R. POWELL.

Welding cast iron. H. WADE. From INTERNAT. NICKEL CO. (B.P. 298,781, 27.10.27).—Ferrous material (*e.g.*, cast iron) containing 1–40% Ni (preferably 5–20%), 2–4% C, and 2–3.5% Si is fed into the welded joint.

M. E. NOTTAGE.

Treatment of tinned scrap iron. A. DOSSMANN (B.P. 298,103, 29.6.27).—The scrap is cleaned by pickling in acid or electrochemically to free it from zinc, lead, tin, and varnishes, treated with hot caustic soda solution containing molasses to remove adhering, insoluble lead compounds, and pressed into briquettes of such a density that the penetration of the heating gas during their subsequent welding into billets is reduced to a minimum so as to avoid oxidation; if desired, before or after briquetting, substances to prevent oxidation and/or reduce the oxides formed may be added.

M. E. NOTTAGE.

Hollow drill steel. L. PRYCE (B.P. 275,560, 27.4.27. S. Afr., 9.8.26).—A tubular core of non-corroding ferrous alloy, such as chromium alloy, is incorporated in a steel ingot or billet, which is then reduced to form hollow rock-drill steel with the tubular core forming the surface of the axial hole.

M. E. NOTTAGE.

Manufacture of metal powders [iron and nickel] from metal carbonyls. GEN. ELECTRIC CO., LTD., L. D. GOLDSMITH, and J. F. JACKSON (B.P. 300,691, 18.8.27).—A stream of vapour obtained by passing nitrogen or carbon monoxide through boiling iron carbonyl is passed into a water-cooled reaction vessel through a

jet, on emerging from which it meets a stream of heated inert gas, e.g., carbon monoxide, whereby the carbonyl is decomposed liberating the metal as a fine powder instead of as a mirror, which is the case when the carbonyl is decomposed at a heated surface.

A. R. POWELL.

Heat-resisting alloy. H. M. WILLIAMS (U.S.P. 1,690,352, 6.11.28. Appl., 11.6.27).—The alloy consists of iron, 2.5–3% C, 3–3.5% Si, and 1.9–2.25% P.

H. ROYAL-DAWSON.

Rapid magnetising and demagnetising metal alloys. S. OSSER (U.S.P. 1,692,690, 20.11.28. Appl., 26.6.26).—An iron alloy containing 0.51% Mn, 0.1–0.15% P, and 0.3–0.5% Si is claimed.

H. ROYAL-DAWSON.

Welding rod. F. M. BECKET, ASST. to OXWELD ACETYLENE CO. (U.S.P. 1,689,577, 30.10.28. Appl., 30.3.26).—The rod consists of an iron alloy containing 1–4% Mn, 0.2–4% Ni, and 0.1–0.3% Si.

H. ROYAL-DAWSON.

Leaching process [for copper ores *in situ*]. R. F. GRANT, F. B. RICHARDS, and H. E. WETHERBEE, ASSTS. to H. M. HANNA (U.S.P. 1,690,446, 6.11.28. Appl., 31.3.25).—The ore is leached *in situ* with a 4% solution of ferric sulphate which is allowed to percolate through a series of drifts in the ore body connected with a central shaft from which the pregnant solution is subsequently pumped.

A. R. POWELL.

Heat-treatment of oxidised copper ores. T. J. and B. TAPLIN, and METALS PRODUCTION, LTD. (B.P. 300,701, 19.8.27).—The ore is mixed with 2% of carbon and 0.5% of sodium chloride and roasted in a reducing atmosphere at 600–650°. Roasting is then continued for a further period in an oxidising atmosphere, and is finally completed in a reducing atmosphere with more salt and carbon. The process may be carried out in separate furnaces or in a multiple-hearth furnace with alternating reducing and oxidising hearths. The roasted material is subjected to froth flotation to recover the copper.

A. R. POWELL.

Coalescing copper. COPPER DEOXIDATION CORP., ASSEES. of H. H. STOUT (B.P. 271,853, 9.5.27. U.S. 26.5.26).—The metal in the form of small pieces, e.g., broken or sheet cathodes, is heated at about 820° and treated with a carrier gas consisting of hydrogen, hydrocarbons, nitrogen, carbon dioxide, water vapour, or a mixture of hydrogen and carbon monoxide, to remove surface impurities such as arsenic, antimony, and sulphur. It is then subjected to a pressure sufficient to bring the surfaces of the pieces into intimate contact while the temperature is sufficient to cause grain growth across the original surface boundaries, the non-injurious atmosphere being maintained throughout the process.

M. E. NOTTAGE.

[Copper] alloys. E. MANOS (B.P. 282,095, 10.12.27. Switz., 11.12.26).—A non-oxidisable alloy, capable of being cast in green sand, may be made by melting copper alloy waste with not more than 10% of its weight of manganese, aluminium, magnesium, silicon, nickel, or iron; or an alloy of these metals, with or without the addition of small quantities of the constituents of the copper alloy, may be first formed and afterwards added

to the molten copper alloy; or the substances may be added individually to the molten copper alloy.

M. E. NOTTAGE.

Manganese bronzes. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 286,276, 2.3.28. Ger., 2.3.27).—Copper is alloyed with up to 25% of manganese, with or without one or more of the metals chromium, iron, titanium, molybdenum, tungsten, vanadium, or zirconium; lead and/or cadmium may also be added to reduce the toughness of the final alloy, which should not contain aluminium except as an impurity. Lead or cadmium may be partly replaced by arsenic, antimony, tin, thallium, or zinc. A suitable alloy contains 88.5% Cu, 6% Mn, 2% Pb, 1% Cd, 1% Zn (or Ti), 0.5% As, 0.5% Sb, and 0.5% Sn.

M. E. NOTTAGE.

Bearing alloy. C. H. BIERBAUM, ASST. to LUMEN BEARING CO. (U.S.P. 1,687,924, 16.10.28. Appl., 4.6.26).—The alloy contains 84.5% Cu, 10.5% Sn, and 5% Ni. The bearing is cast in a chill mould in such a way that the α -crystals are in a soft condition and a large proportion of hard δ -crystals is formed upon the chill surface.

A. R. POWELL.

Bearing-metal alloys. K. MÜLLER and W. SANDER (U.S.P. 1,691,931—2. 20.11.28. Appl., [A] 30.9.21, [B] 2.9.22. Ger., [A] 1.10.20, [B] 15.9.21).—The alloys comprise 70–75% Pb, 15–25% Sb, 3–6% Sn, and (A) 1–3% of a metal of the cobalt group and 0.6–2% Cu, or (B) up to 5% Cd.

H. ROYAL-DAWSON.

Heat-treatment of magnesium alloys. Z. JEFFRIES and R. S. ARCHER, ASSTS. to AMER. MAGNESIUM CORP. (U.S.P. 1,689,630, 30.10.28. Appl., 14.10.21).—Alloys with a magnesium base are heated for several hours at just below the m.p. of the most fusible constituent, and the plastic alloy is then subjected to mechanical deformation.

A. R. POWELL.

Improving the resistance to corrosion of magnesium and magnesium alloys. I. G. FARBERIND. A.-G. (B.P. 287,450, 9.3.28. Ger., 19.3.27).—The metal pieces are subjected to the simultaneous or successive actions of solutions of an acid other than sulphuric or hydrochloric acid, e.g., nitric acid, and of potassium bichromate. They are then washed first with cold and then with hot water which may, if desired, contain bichromate, and are then dried.

M. E. NOTTAGE.

Drawing section bars from sheets of magnesium alloys. I. G. FARBERIND. A.-G. (B.P. 284,317, 13.1.28. Ger., 28.1.27).—The material to be worked is drawn through dies at from 100–400° according to the thickness of the sheet and the desired degree of shaping. A suitable arrangement of dies is described.

M. E. NOTTAGE.

Aluminium alloys and their manufacture. P. BERTHÉLEMY and H. DE MONTBY (B.P. 283,926, 29.12.27. Fr., 20.1.27. Addn. to B.P. 252,028. Cf. U.S.P. 1,599,869; B., 1926, 1018).—The rich alloy produced by the process of the prior patent is incorporated with aluminium of greater (99–99.5%) purity, after which an aluminium-magnesium-cadmium alloy is added. The resulting alloys are light, of high resistance, and not attacked by sea water, salt air, or soft water.

M. E. NOTTAGE.

[Electrolytic] production of aluminium-silicon alloys. VEREIN. ALUMINIUM-WERKE A.-G., and H. SCHORN (B.P. 300,429, 23.1.28).—To the electrolytic bath, which consists mainly of aluminium oxide and metal fluoride, uncombined silicon is added in an amount corresponding to the desired proportion in the alloy to be produced, and at a rate equal to that at which the liberation of the aluminium takes place.

M. E. NOTTAGE.

Production of a metal-dust [zinc-lead] mixture. M. H. NEWELL, ASSR. to ALLOYS CO. (U.S.P. 1,687,034, 9.10.28. Appl., 11.4.27).—Zinc is vaporised, lead oxide is decomposed in the vapour, and the mixture is cooled sufficiently rapidly to condense the zinc in a finely-divided form.

W. G. CAREY.

Reducing the cadmium content of zinc ore. O. GERLACH and N. OSTMAN, ASSRS. to MATTHIESSEN & HEGELER ZINC CO. (U.S.P. 1,691,714, 13.11.28. Appl., 3.12.26).—The oxide ore is subjected to the action of steam, and an amount of reducing agent sufficient to reduce the cadmium in preference to the zinc contained in the ore, at a temperature at which both metals are reduced.

M. E. NOTTAGE.

Thermostatic element. P. H. BRACE, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,689,814, 30.10.28. Appl., 12.3.26).—One element comprises a nickel alloy with a high coefficient of expansion, and the other an alloy containing 1–20% Co, the remainder being iron and nickel in the ratio 3:1.

A. R. POWELL.

Protection of metals in the cementation process. SOC. ANON. DES ACIÉRIES ET FORGES DE FIRMINY (B.P. 277,307, 12.8.27. Fr., 7.9.26).—In the hardening of steel alloys by the process of B.P. 174,580 (B., 1923, 273 A), the parts to be protected are covered by a thick, compact, and adherent deposit of nickel.

H. ROYAL-DAWSON.

Production of [metallic] acid-resisting wrapping material. STANIOLFABR. BURGDOFF A.-G. (B.P. 292,162, 1.6.28. Switz., 17.6.27).—Metal foil is coated with a solution of bleached shellac in rectified spirits; salicylic acid is added to prevent mould formation when used for wrapping cheese etc.

M. E. NOTTAGE.

Reduction of metallic oxides. W. H. SMITH, ASSR. to GEN. REDUCTION CORP. (U.S.P. 1,692,587—8, 20.11.28. Appl., [A] 17.2.26, [B] 26.11.26. Can., [B] 28.6.26).—See B.P. 266,289; B., 1928, 451.

Cadmium-plating bath. A. W. YOUNG and M. E. LOUTH, ASSRS. to UDYLITE PROCESS CO. (U.S.P. 1,692,240, 20.11.28. Appl., 31.3.26).—See B.P. 266,985; B., 1927, 338.

Treatment of waste pickle liquors (B.P. 300,233).—See VII. Annealing furnace (U.S.P. 1,691,259).—See XI.

XI.—ELECTROTECHNICS.

See also A., Dec., 1297, Photo-electric properties of alkali metals (CAMPBELL). 1311, Magnetic properties of adsorbed substances (BHATNAGAR and others). 1314, Magnetic saturation of iron carbide (STÄBLEIN and SCHROETER). 1330, Gas-metal elec-

trodes (FRENCH and KAHLENBERG). 1331, Pyrochemical Daniell cells (LORENZ and MICHAEL). Characteristics of alkali cells (FLEISCHER and GOLDSCHMIDT). 1337, High-voltage arcing and alternating-current electrolysis (SHIPLEY and GODEVE). 1337, Preparation of potassium ferricyanide electrolytically (PAWEK and HIRSCH). 1345, Potentiometric analysis (KAMENISKI; HECZKO). 1349, Potentiometric measurement of high resistance cells (BEANS and WALDEN). Hydrogen electrode vessel for solutions of high resistance (BEST). Conductivity vessels (LOTTERMOSER and BUCHHOLZ). 1353 and 1371, Electrolytic oxidation of alcohols (KOIDZUMI). 1372, Electrolytic reduction of aldehydes (SHIMA).

Cottrell-Moeller process for dusts. VER EECHE.—See I. Bleach liquors. MÜLLER. Hydrogen peroxide. LÖWENSTEIN.—See VII. Electrical porcelain. TWELLS.—See VIII. Electrolytic corrosion prevention. PHILO.—See XXIII.

PATENTS.

Electric [arc] furnace. J. C. WOODSON, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,691,365, 13.11.28. Appl., 19.1.27).—Heating units comprising electrodes mounted in operative relation are electrically connected in parallel, and a separate heating unit is formed of a single electrode from each of these units.

J. S. G. THOMAS.

Electric furnaces. J. WEINTZ, ASSR. to STRONG, CARLISLE, & HAMMOND CO. (U.S.P. 1,692,478—9, 20.11.28. Appl., [A] 26.1.27, [B] 3.10.27).—(A) The refractory furnace lining has parallel depressions of curved section, the side portions meeting at an angle to form ridges, and a parallel, spaced, heating element is arranged before each depression and in front of the plane of the ridges. (B) Each of a bank of heating elements arranged in parallel extends between a pair of contactors, each contactor having separate backing means and being secured upon opposite furnace walls. Yielding connexions are provided between one of these backing means and its set of contactors.

J. S. G. THOMAS.

Furnace. Annealing furnace. K. TAMELE, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,691,258—9, 13.11.28. Appl., [A, B] 2.12.26. Ger., [A] 3.12.25, [B] 5.12.25).—(A) Electric heaters are arranged in the furnace chamber, and a pair of connected removable stoppers, fitting in a metallic pipe mounted in the furnace casing and extending vertically through the centre of the chamber, insulate the interior of the chamber thermally from the outside atmosphere. The pipe is also used as a manually-controlled cooling device. (B) A bright-annealing furnace comprises a cased furnace chamber provided with a cooling device below the chamber for condensing steam in the furnace, and means for exposing the furnace atmosphere to the action of the cooling device.

J. S. G. THOMAS.

[Electrical] conductors of high negative temperature coefficient. H. ANDRE (B.P. 280,862, 30.8.27. Fr., 17.11.26).—Powdered silver and sulphur are heated

above 800°, sulphur being present in excess of the amount required to form silver sulphide. J. S. G. THOMAS.

Insulated wire. E. B. WHEELER, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,689,311, 30.10.28. Appl., 9.10.24).—A conductor is coated with finely-divided selenium and cellulose acetate to make it flameproof.

H. ROYAL-DAWSON.

Incandescence [electric] lamp. E. HENKEL and H. WOLFF, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,687,496, 16.10.28. Appl., 8.11.27. Ger., 7.12.26).—A globe of the gas-filled type contains, besides the usual inert gas, an ammonium halide, *e.g.*, ammonium chloride or iodide. The products of dissociation prevent the undesirable discharges at the temperature of the incandescent filament.

F. G. CLARKE.

Electron-discharge device with oxide-coated filament. H. C. RENTSCHLER, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,691,446, 13.11.28. Appl., 23.3.25).—A plate and a cathode coated with an alkaline-earth carbonate are sealed in an envelope which is evacuated, and a metallic getter is vaporised within the envelope while the cathode is heated to reduce the carbonate to oxide.

J. S. G. THOMAS.

Manufacture of luminous electric-discharge tubes containing rare gases. J. B. J. M. ABADIE (B.P. 276,019, 13.8.27. Fr., 14.8.26).—The rare gas, after purification by an arc or electric discharge between electrodes of an alkaline or alkaline-earth metal or a metal of the tungsten group, having zones of different temperatures along which the gas is convected, is introduced into the discharge tube previously exhausted to a pressure greater than Ki/S , where i is the intensity of the current employed, S the area of the electrode, and K a constant depending on the natures of the electrodes and the gas employed.

J. S. G. THOMAS.

Treatment of filaments for vacuum tubes etc. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of G. R. SHAW and B. F. TELLKAMP (B.P. 289,837, 3.5.28. U.S., 3.5.27).—An incandescent filament is exposed to an atmosphere containing a non-metal, *e.g.*, carbon, and the duration of treatment is automatically controlled by the increase in resistance of the filament.

J. S. G. THOMAS.

Means for obtaining vacua [in vacuum tubes]. C. W. BALKE, Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,691,177, 13.11.28. Appl., 17.2.27).—Gases are absorbed by a piece of columbium arranged near a heated filament in the tube to be exhausted.

J. S. G. THOMAS.

Storage battery plate and method of rendering it permanent. W. L. REINHARDT, Assr. to WILLARD STORAGE BATTERY CO. (U.S.P. 1,688,399, 23.10.28. Appl., 11.10.22. Renewed 9.3.28).—The plates are treated with a solution of a substance which, when dry, will provide a moisture-proof coating, and are then heated by means of a current of hot inert gas.

A. R. POWELL.

Electrolytic rectifier. W. E. HOLLAND, Assr. to PHILADELPHIA STORAGE BATTERY CO. (U.S.P. 1,691,806, 13.11.28. Appl., 13.1.23).—An asymmetric cell contains a non-film-forming iron electrode containing 10%

Si or above, and a film-forming electrode is mounted so that its active surface is completely below an electrode mounted near the initial surface level of the electrolyte.

J. S. G. THOMAS.

Electrolytic apparatus. F. C. NIXON (U.S.P. 1,692,757, 20.11.28. Appl., 8.2.27).—Electrolytic apparatus for treating plates comprises insulating means for supporting the plates and rollers for engaging their edges and faces.

J. S. G. THOMAS.

Means for indicating the purity of liquids. H. L. RANGER (B.P. 300,229, 9.8.27).—Electrodes employed for indicating the electrical conductivity of the liquid, *e.g.*, boiler feed water, are mounted in insulators and are detachably secured to a socket forming a guard.

J. S. G. THOMAS.

Improving the efficiency of electro-thermic installations. H. V. DE CHRISTIANI and A. CUMINATTO (B.P. 300,799, 28.11.27).

Mounting of furnace electrodes. SOC. ELECTRO-MÉTALLURGIQUE DE MONTRICHER (B.P. 299,414 and 299,429, 20.12.27. Fr., 26.10.27).

Activation of carbonaceous material (B.P. 300,146).—See II. **Light-diffusing glass** (B.P. 287,545). **Seal for hard glass** (U.S.P. 1,691,436).—See VIII. **Furnace** (U.S.P. 1,687,925). **Magnetisable alloys** (U.S.P. 1,692,690). **Aluminium-silicon alloys** (B.P. 300,429).—See X. **Bleaching of fatty acids** (B.P. 299,149).—See XII. **Vitamin-D from ergosterol** (B.P. 296,093).—See XX. **Recording paper for telegraphic apparatus** (B.P. 282,759).—See XXI.

XII.—FATS; OILS; WAXES.

Acetone as fat-solvent in the rapid determination of iodine value in the technical laboratory. B. M. MARGOSCHES, B. KRAKOWETZ, and F. SCHNABEL (Chem. Umschau, 1928, 35, 300—305).—Acetone may be used with advantage as a solvent for fatty oils in the rapid determination previously described (*cf.* B., 1924, 639). The oil (0.1 g.) is dissolved in not more than 2 c.c. of acetone and the alcoholic iodine solution is added; after 5 min. the determination is completed by the addition of potassium iodide and water and by titration with sodium thiosulphate in the usual manner; the back titration may not be postponed. Exposure of the reaction mixture to daylight does not affect the result. The iodine absorption of the small amount of acetone necessary is negligible, but acetone should not be substituted for alcohol in the stock iodine solution.

E. LEWKOWITSCH.

Spontaneous heating of [vegetable] oils. N. J. THOMPSON (Oil & Fat Ind., 1928, 5, 317—326).—The tendency of oils to heat spontaneously does not run parallel with the iodine values: oils of lower iodine value in which oxidation has begun have greater heating tendencies than pure oils of higher iodine number; several oils (*e.g.*, lard oil) which would pass the Mackey test as safe oils were found to heat readily under suitable conditions. More representative results were obtained by the use of 30 g. of clean cotton waste impregnated with an equal weight of oil, and a modified Mackey tester is described by means of which the influence of moisture,

initial temperature, catalysts, etc. was investigated. Spontaneous heating is favoured by increased humidity of the cotton (up to 10%), increased initial temperature, and by preheating the oiled cotton. The presence of oxides, *e.g.*, iron rust, increased the fire hazard, and admixture of 50% of mineral oils or 0.5% of quinol merely postponed spontaneous heating.

E. LEWKOWITSCH.

Luminescence of fats and oils. A. VAN RAALTE (*Z. Unters. Lebensm.*, 1928, **56**, 195—198).—See B., 1928, 792.

See also A., Dec., 1304, **Fluorescence and chemiluminescence of cod-liver oil** (WOODROW and WISSINK). 1322, **Gels from vulcanised oils** (KNIGHT and STAMBERGER). 1327, **Triglycerides** (LOSKIT). 1354, **Glycerides of aliphatic acids** (HEIDUSCHKA and SCHUSTER). 1402, **Decomposition of olive oil by micro-organisms** (PIGULEVSKI and CHARIK). 1405, **Effect of quinol on vitamin-A content of stored oils** (HUSTON and others). **Vitamin-A content of wheat oil** (SURE). 1406, **Destruction of vitamin-D during irradiation of ergosterol** (VAN STOLK and others). 1408, **Colouring matter of cottonseed hulls** (GILL and GREENUP).

Fats as parent substances of petroleum. RAKUSIN. **Marine-animal oils as fuel.** MARCELET.—See II. **Linseed oil for lead-whites.** HELLER.—See XIII. **Cottonseed meal.** MENAUL.—See XIX.

PATENTS.

Bleaching of artificial fatty acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,149, 7.9.27).—The crude fatty acids resulting from the oxidation of hydrocarbons of the paraffin series may conveniently be bleached by means of oxidising agents such as chromic acid and a mineral acid or organic acids other than those treated. The process is preferably carried out in moderate concentration or under pressure at temperatures of 105°, rising at the end of the operation to 110—120°. An electrolytic oxidation process may also be employed.

E. HOLMES.

Cleavage of oils and fats. IMPERIAL CHEM. INDUSTRIES, LTD., and E. CHAPMAN (B.P. 300,360, 29.9.27).—The sulphonic acids and their salts, obtained by the process of B.P. 274,611 (B., 1927, 841), may be used as splitting agents in the Twitchell process; their usefulness is enhanced if the crude reagents are washed with dilute sulphuric acid of strength down to 20—25%. In an example exceptionally light-coloured free fatty acids are obtained from tallow.

E. HOLMES.

Recovery of oil from fatty solutions of proteins and glues in water. AKTIEBOLAGET SEPARATOR (B.P. 277,311, 18.8.27. Swed., 8.9.26).—Solutions obtained by boiling, *e.g.*, whale flesh with water are maintained above the coagulation temperature during the separation of the oil, the greater part of which is allowed to settle out in heated tanks, the remainder being removed in centrifugal apparatus.

L. A. COLES.

Treatment of [vegetable] oils. A. SCHWARZMAN, ASSR. to SPENCER KELLOGG & SONS, INC. (U.S.P. 1,692,226, 20.11.28. Appl., 3.6.25).—The oil is agitated with dilute caustic alkali solution corresponding in quantity to the free fatty acids present, and the mixture is allowed

to rest until the solid soaps formed have agglomerated. The oil is then heated almost to 100° and hot water is passed through it in finely-divided form until it is neutral, and the aqueous solution is then removed.

W. J. BOYD.

Extraction or filtration of oil-containing material, and removal of solvent from the residues. P. L. FAUTH (B.P. 283,216, 5.1.28. Ger., 7.1.27).—An apparatus has been devised whereby oil-containing material such as fuller's earth may be filtered and extracted effectively in thin layers. The material is introduced into a narrow annular space between an outer steam-jacketed shell and an inner filter cloth-covered sieving jacket. Modifications of the apparatus allow of thorough washing of the residue and the uniform removal of the solvent by reducing pressure on the inside of the sieving jacket, and heating the material by means of the outer steam jacket.

E. HOLMES.

Apparatus for extraction of water, oil, and fatty matter from solid material. J. R. STERLING (U.S.P. 1,694,361, 4.12.28. Appl., 8.3.28. U.K., 8.11.26).—See B.P. 286,752; B., 1928, 375.

Separation of fatty acids (B.P. 273,744 and 290,992).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Addition of lead oxide to paints. W. VAUBEL (*Farben-Ztg.*, 1928, **34**, 563).—The presence of litharge or red lead in oil paints gives rise to solid compounds produced by their reaction with glycerol probably liberated by their saponifying action. White lead, basic lead sulphate, and basic lead chloride do not react in this way.

S. S. WOOLF.

Manufacture of [linseed] oil for lead-whites. II. HELLER (*Farben-Ztg.*, 1928, **34**, 506).—The processes adopted to render linseed oil suitable for incorporation in white-lead paints are described. Filtered oil, expressed at low temperatures, is thoroughly mixed with 1—1½% of concentrated sulphuric acid in order to precipitate mucilaginous impurities, well washed with warm and cold water till free from mineral acid, and finally treated with open steam to decompose any sulphonated oil formed; the oil is dried under vacuum, bleached with decolorising earths (*e.g.*, 0.5—3% of "Tonsil"), and filtered.

E. LEWKOWITSCH.

Natural and artificial [weathering] experiments in testing the durability of oil-paints. A. EIBNER (*Farben-Ztg.*, 1928, **34**, 322—326).—The value of natural weathering tests and imitative accelerated laboratory tests in estimating the durability of paints is discussed. It is pointed out that in weathering tests considerable variations in the results are caused by accidental conditions of exposure—climate, season, weather, etc. Laboratory tests are rapid, produce constant results, and may readily be adapted to conform with any peculiar condition to which the paint may be exposed.

E. LEWKOWITSCH.

Pigment and vehicle. V. G. JOLLY (*J. Oil and Colour Chem. Assoc.*, 1928, **11**, 361—375).—The behaviour of pigment aggregates on being "ground" in a mill depends on (a) cohesive forces, (b) tenacity of

adsorbed gases, (c) shearing forces due to the mill, and (d) the relationship between the forces acting at the surfaces of the components. The dispersion of solids in liquid media is discussed from these aspects, particular attention being given to the assistance of the dispersion by the use of wetting agents. Methods for determining particle size by sedimentation, and consistency by a slightly modified Gardner mobilometer are described.

S. S. WOOLF.

Tinting strength of pigments. H. D. BRUCE (U.S. Bur. Stand. J. Res., 1928, 1, 125—150).—The term "tinting strength" is discussed as a two-factor property. A method of treating these properties spectrophotometrically is given, and certain empirical formulæ are developed which may be used for comparing the proper mixtures of white pigment with pigment of another colour in order to obtain a tint of some desired brightness or colorimetric purity. Colour is specified in the monochromatic terms of dominant wave-length, purity, and brightness.

W. E. DOWNEY.

Preparation of clear nitrocellulose lacquers. F. KOLKE (Farben.-Ztg., 1928, 34, 561—563).—On the manufacturing scale, nitrocellulose lacquers may be prepared by (a) incorporating all the ingredients together, (b) incorporating the nitrocellulose, resins, and plasticisers in the solvents and adding the diluents, or (c) dissolving the plasticisers and resins in the diluents, dispersing the nitrocellulose in the solution, and finally adding the solvents. The last method is considered the most successful, as maximum surface is exposed to solvent action, the clotting inherent to the other two methods being avoided. In the use of mixtures of high- and low-viscosity nitrocellulose, the former variety must be dispersed first. Clarification, preferably by centrifuge, is essential for large-scale production, although satisfactory results may be obtained by filtration or straining.

S. S. WOOLF.

Hardening and esterification of rosin. E. PYHÄLÄ (Farben.-Ztg., 1928, 34, 616—617).—Details are given of the preparation of glyceryl and various phenolic esters of abietic acid according to the authors' method (B., 1928, 100). Calcium and magnesium glycerate, calcium phenoxide, cresoxide, β -naphthoxide, and resorcinate are the intermediates described.

S. S. WOOLF.

Physical properties of shellac solutions. I. M. RANGASWAMI and M. VENUGOPALAN (Indian Lac Res. Assoc., 1928, Bull. No. 1, 14 pp.).—The variation of viscosity with concentration of shellac solutions in various alcoholic solvents was determined. "Solvent-power numbers" of the solutions, using water and light petroleum as non-solvents, are plotted against concentration and against viscosity, it being demonstrated that both solvent power and viscosity must be correlated in rating a solvent. Wood naphtha is shown to exhibit peculiar behaviour, a second peak in the solvent power occurring at about 30% concentration, such peak being absent in the case of the other solvents.

S. S. WOOLF.

Accelerated tests of organic protective coatings. P. H. WALKER and E. F. HICKSON (Bur. Stand. J. Res., 1928, 1, 1—17).—See B., 1928, 578.

See also A., Dec., 1350, Hungarian fossil resin (ZECHMEISTER).

PATENTS.

Manufacture of paint. T. GOLDSCHMIDT A.-G. (G.P. 447,469, 10.8.23).—An oil paint containing as pigment the highly dispersed form of litharge obtained by condensation of lead oxide fume is claimed. The pigment has no tendency to separate out from the oil and much less pigment is required to obtain a paint of good covering power than is the case with ordinary ground litharge.

A. R. POWELL.

Manufacture of paint. A. KNAPP (B.P. 298,843, 24.2.28).—A washable distemper is mixed with an emulsion of drying oil, ammonium resinate, and soap in aqueous solution.

W. G. CAREY.

Production of phosphorescent or luminous compositions. J. A. F. BEAVIS (B.P. 299,228, 22.12.27).—Such compositions acquire greater permanence by grinding the salts generally used with resins, such as fused copal, sandarac, etc. The resulting mixture is added in the usual way to the fatty oils, diluents, etc. functioning as vehicles; they may also be incorporated with rubber and/or plasticised cellulose esters or ethers.

P. E. L. FARINA.

Resins of the phthalic anhydride-glycerol type and their preparation. E. C. R. MARKS. From BAKELITE CORP. (B.P. 300,668, 11.7.27).—Mixtures of phthalic anhydride or, e.g., succinic acid, malic acid, etc., and a polyhydroxy-alcohol, e.g., glycerol, are heated at 150—175° to form a colourless, initial condensation product, and this is heated in moulds at 90—100° until it is of a rubber-like consistency, and finally at 125—135° until it is infusible and sufficiently hard.

L. A. COLES.

Manufacture of laminated products. E. C. R. MARKS. From BAKELITE CORP. (B.P. 300,836, 10.2.28).—Superposed sheets of fibrous material which have previously been impregnated with a solution of a resin of the glycerol-phthalic anhydride type, dried, and coated with a phenol resin are consolidated to a solid block by means of heat and pressure.

L. A. COLES.

Production of artificial materials, and/or articles made therefrom, from the condensation products of urea or urea derivatives with formaldehyde or substances yielding formaldehyde. F. SCHMIDT (B.P. 281,993, 16.6.27. Ger., 8.12.26).—Urea or its derivatives and formaldehyde etc. are condensed, and the products are subsequently dehydrated, at 40—45° in the presence of carriers, e.g., casein, cellulose or its derivatives, or natural or artificial resins, the mixture being kept in constant motion, e.g., in a kneading machine under the ordinary or reduced pressure, or by working it between rollers. Accelerating or retarding agents, softening agents, solvents, fillers, and other substances capable of condensing with formaldehyde, e.g., urethane, aniline, phenol, etc., may also be added to the mixture. The products are finally moulded and hardened at a raised temperature and under pressure.

L. A. COLES.

Production of lithopone fast to light. G. JANTSCH and P. WOLSKI, Assrs. to I. G. FARBENIND. A.-G. (U.S.P.

1,693,902, 4.12.28. Appl., 3.11.24. Ger., 30.11.23).—See B.P. 225,523; B., 1925, 601.

Production of condensation products of phenols with aldehydes. G. PETROV (U.S.P. 1,693,461, 27.11.28. Appl., 23.4.27. Union of Soviet Socialist Republics, 20.12.26).—See B.P. 283,002; B., 1928, 164.

Treating solutions [for recovering gummy or plastic substances]. NAUGATUCK CHEM. CO., ASSECS. OF W. S. JOHNSTON and A. W. KEEN (B.P. 292,137, 31.3.28. U.S., 15.6.27).—See U.S.P. 1,673,685; B., 1928, 762.

Cellulose ester coating compositions (B.P. 300,157).—See V. **Finely-divided iron oxide** (B.P. 298,926).—See VII. **Abradant materials** (B.P. 281,711).—See VIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Dispersoidological investigations on latex. P. P. VON WEIMARN (Kolloid-Z., 1928, 46, 217—223).—Latex is described as a polydisperse system of isoaggregate particles with a general liquid-jelly consistency. The particles, which are regarded as solid, are very small and for the most part indistinguishable under the ultra-microscope. The components of the dispersion medium (proteins, water, resins, etc.) enter also into the composition of the particles of the disperse phase. Experiments have been carried out on the coagulation of *Hevea* latex by a large number of substances. With substances which have a dispersing effect on proteins (such as the iodides and thiocyanates of the alkalis and alkaline-earths), different effects were observed according to the conditions of temperature and concentration. When the conditions were so chosen that rapid dispersion of the proteins in latex could not occur, the latex was not coagulated, but formed a pasty mass which readily became liquid on shaking. Under conditions favouring rapid dispersion of proteins, the whole system set to a gel in a short time, and by drawing out such a gel during its formation the final product has vectorial properties differing in elasticity according to the direction of stress. This is considered to support the author's views on the caoutchouc-like state of matter. The higher the temperature employed for coagulation, the more solid is the coagulum produced, and products of definite mechanical properties can be prepared by suitable choice of the temperature. Substances which coagulate proteins (e.g., citrates, tartrates, etc.) coagulate caoutchouc only after several days, but eventually give flakes or paste-like masses. Mixed coagula of caoutchouc with silk, casein, cellulose, etc. are described.

E. S. HEDGES.

Cleavage of stretched rubber and the displacement of the cleavage plane by the addition of fillers. M. LE BLANC and M. KRÖGER (Z. Elektrochem., 1928, 34, 725—734).—The influence of the chemical nature and degree of aggregation of a number of substances on the stress-strain curves and breaking values of unvulcanised rubber at temperatures ranging from -15° to -60° has been investigated. The following fillers were employed: zinc oxide, magnesium carbonate, barium peroxide (fine particles); barium sulphate, potassium thiocyanate, eosin, furol-B, furol-yellow-brown, methylene-blue (fine

and coarser particles); potassium chloride, bromide, and iodide, ammonium chloride, and boric acid (coarse particles). The same results are obtained, on the whole, as in normal vulcanisations. Zinc oxide, in particular, and magnesium carbonate have a strengthening effect, whilst the reverse is true of the alkali salts, with the exception of potassium thiocyanate. Methylene-blue and eosin also have a slightly weakening influence. The method of investigation is of importance, as by means of it the properties of fillers can be ascertained without carrying out a vulcanisation. The surfaces of the fractured test pieces were examined microscopically, and the adsorption processes of fillers in rubber are discussed. It is considered that the phenomenon of cleavage has been proved to exist for rubber, and the conception of cleavage-plane distortion is advanced.

L. L. BIRCUMSHAW.

Thermodynamic theory of rubber fillers. I. HOCK (Z. Elektrochem., 1928, 34, 662—664).—A conception is given of the heat of adhesion between the filler and the rubber which is related to the total interfacial energy. It is the difference between the calorimetric heat of swelling in benzene, say, of the filled rubber and the sum of the heat of swelling of the pure rubber in benzene and the heat of wetting of the filler in the same solvent. This latter quantity depends on the concentration of the filler in the rubber, and its maximum value, U_0 , at "infinite dilution" may be determined by extrapolation of experimental values. The interfacial free energy, A , of the system is a function of the breaking strength, A , which, as before, may be extrapolated to A_0 . Measurements show that A_0/U_0 is about 0.5, showing that approximately half of the calorimetrically measured total interfacial energy is used in strengthening the rubber. The practical determination of A and U is discussed.

S. K. TWEEDY.

Isomerisation of rubber under electric discharge. I. HOCK (Z. Elektrochem., 1928, 34, 664—667).—The action of a glow discharge on decalin solutions of natural rubber in an atmosphere of hydrogen was investigated (cf. B., 1923, 508 A), the solvent being repeatedly replaced to eliminate the effects of its three-fold polymerisation. Polymerisation occurs with the disappearance of double linkings so that the degree of unsaturation progressively becomes smaller. The viscosity, m.p., and softening point of the rubber also decrease as the duration of the discharge increases. A powder with the same composition as the original rubber is obtained as the final product. Synthetic rubbers show the same variations except that the properties pass through a maximum after 1—2 hrs., indicating that these rubbers differ structurally from natural rubber.

S. K. TWEEDY.

Gelatinisation of vultex. P. P. VON WEIMARN (Kolloid-Z., 1928, 46, 223—225).—Photomicrographs of gels of latex and vultex are given and indicate a fibrillar arrangement of the particles. These become arranged parallel to each other when the gel is stretched. The particles have an inner fibrillar structure, and the thickness of the ultimate fibrils may not exceed that of one molecule.

E. S. HEDGES.

Vulcanisation accelerators. R. DITMAR and M. RACHNER (Chem.-Ztg., 1928, 52, 935).—Data for a

number of proprietary organic accelerators mainly of the aldehyde-amine type are tabulated indicating their behaviour towards various solvents, their approximate relative effect on vulcanisation in the presence of oxides of lead, zinc, magnesium, or cadmium, and their influence on several pigments of mineral or organic nature.

D. F. TWISS.

PATENTS.

Production of raw rubber from rubber latex. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,719, 27.8. and 15.9.27).—Latex is treated with proteases in the presence of substances such as hydrocyanic acid, hydrogen sulphide, or saline substances possessing buffer properties. Coagulation in this way is rapid, but may be retarded or accelerated as desired by adjustment of the p_H value of the latex. Suitable proteases are found in *Succus caricae papayae*.

D. F. TWISS.

Preservation and treatment of latex. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,394 and 300,456, [A] 18.11.27, [B] 2.4.28. Addn. to B.P. 294,412; B., 1928, 721).—(A) The preservation of latex which has already been effected by sulphonates of soap-like properties can also be effected with sulphonates possessing tanning properties; both types can also advantageously be applied to concentrated or vulcanised latex. (B) In the use of such sulphonates, the additional presence of aliphatic or aromatic alcohols, halogenated or otherwise, ketones, hydrocarbons, or halogeno-, nitro-, or amino-derivatives of the last-named, further improves the latex both with respect to stability and to ease of penetration of fabrics.

D. F. TWISS.

Preservation of rubber. GOODYEAR TIRE & RUBBER CO., Assecs. of J. TEPPEMA (B.P. 281,616, 13.10.27. U.S., 30.11.26).—Aryl- β -naphthylamines, especially phenyl- β -naphthylamine 2 : 2'-dianilino-1 : 1'-dinaphthylmethane, or $\alpha\beta$ - or $\beta\beta$ -dinaphthylamine, are used as anti-agers in rubber mixers. Only non-accelerators are claimed. [Stat. ref.]

C. HOLLINS.

Vulcanisation of rubber. E. C. R. MARKS. From RUBBER SERVICE LABS. CO. (B.P. 300,287, 9.5.27).—Vulcanisation accelerators are produced by treating Schiff's bases or their derivatives with an acid and, after neutralisation of the acid, submitting the product to the action of an aldehyde or carbon disulphide.

D. F. TWISS.

Rubber tubing. A. S. GREGG (B.P. 300,357, 27.9.27).—Rubber tubing is rendered less perishable and more serviceable by covering with fabric or braiding, and then applying non-rubber-doping materials, e.g., nitrocellulose varnishes.

D. F. TWISS.

XV.—LEATHER; GLUE.

Determination and nature of water-soluble [matter] in leather tanned with wattle bark extract. R. O. PAGE (J. Amer. Leather Chem. Assoc., 1928, 23, 495—523).—Samples of the leather were extracted repeatedly with small quantities of water and the dry residue was determined in each. From the results a curve with two breaks was obtained, the first corresponding to the removal of tannin from the leather com-

pound, and the second to its hydrolysis. Wilson's method of determining water-soluble matter in leather was shown to give high results. Extraction of the leather with 0.001N-sulphuric acid gave a better end-point, but introduced other complications. By the Wilson method both "free" and "combined" water-soluble matter is removed. The former was determined by shaking 8 g. of the leather for 8 hrs. with 100 c.c. of water; the amount was approximately proportional to the concentration of the final liquor used in the tanning. The "combined" water-soluble matter was determined by difference and shown to depend not on the concentration of the tanning liquors employed, but on the treatment of the pelt prior to tanning; the amount was increased by more drastic liming or treatment of the pelt with solutions of calcium salts, acids, or alkalis, but decreased by treatment of the pelt with solutions of chromium sulphate. Variations in the p_H of the tan liquors used had no effect on the content of "combined" water-soluble matter within the range p_H 4—8, but it was increased for p_H values < 4 and decreased for values > 8 . Neither the "free" nor the "combined" water-soluble matter was much affected by extending the tanning period beyond 6 weeks. D. WOODROFFE.

See also A., Dec., 1318, Adsorption and swelling (KUBELKA and WAGNER). 1323, Salting-out of gelatin (McBAIN and KELLOGG). 1387, Degradation of gelatin and gelatin-peptone with acetic anhydride (FODOR and others).

XVI.—AGRICULTURE.

Comparison of Rhenania phosphate and superphosphate as fertilisers. A. v. RÄTH (Z. Pflanz. Düng., 1928, 7B, 505—516).—In field trials with cereals and sugar beet, superphosphate (on a water-soluble basis) and Rhenania phosphate (on an ammonium citrate-soluble basis) were compared. Rhenania phosphate produced the greater crop increases in both grain and straw of barley and oats. On sugar beet the two fertilisers gave practically similar results. In all cases crop increases were of a profitable order. A. G. POLLARD.

Use of artificial fertilisers in the light of results from field trials and soil examination. J. v. CSIKY and E. BECKER (Z. Pflanz. Düng., 1928, 7B, 516—525).—The efficiency of fertilisers is dependent on the physical nature and hydrogen-ion concentration of the soil, and is usually greater in alkaline soils than in acid ones. On soils exhibiting much exchange acidity the soil reaction is appreciably affected by the reaction of added fertilisers. In agreement with the observations of Râth (preceding) it is shown that Rhenania phosphate is more efficient than superphosphate on soils exhibiting exchange or hydrolytic acidity. The significance of p_H determinations of potassium chloride extracts of soil, in consideration of fertiliser requirements, is discussed. A. G. POLLARD.

Potato fertiliser experiments. J. H. STALLINGS (Soil Sci., 1928, 26, 351—362).—The yield of potatoes following fertilisation with potassium sulphate was no greater than when the chloride was used. Comparison of organic nitrogen, the nitrates of calcium and sodium, and ammonium sulphate showed that no special

advantage results from the customary practice of manuring potatoes with nitrogen having varying rates of availability. "Ammono-Phos" was not a satisfactory source of nitrogen for potatoes. Top dressings of ammonium sulphate and potassium sulphate proved successful.

A. G. POLLARD.

Soil reaction and fertiliser requirements. F. SEKERA (Z. Pflanz. Düng., 1928, 7B, 525—527).—Statistical examination of the results of fertiliser-requirement experiments (Neubauer method) and the reaction of a number of arid soils shows that phosphate deficiency is associated with highly acid or highly alkaline conditions. Acid conditions favour the leaching out of phosphate, and in alkaline soils the solubility of phosphates is depressed below the minimum necessary for plant growth. Potash deficiency is characteristic of acid soils. The physical condition of neutral and alkaline soils in arid climates favours a rapid capillary rise of dissolved bases from the subsoil.

A. G. POLLARD.

Identification and composition of the soil aluminosilicate active in base exchange and soil acidity. H. W. KERR (Soil Sci., 1928, 26, 385—398).—Prolonged mechanical separation of soil fractions by various means showed the base-exchanging material of soil to be confined almost entirely to the clay fraction. Adaption of the base-exchange theory of equilibrium lead to the conception that the base-exchange complex in soils closely resembles bentonite clay and differs considerably from the natural zeolites. The active silicate concerned is assigned the formula $\text{H}_2\text{O}(\text{Al}_2\text{O}_3, 6\text{SiO}_2), 8\text{H}_2\text{O}$, and was prepared from bentonite by leaching with 0.1N-hydrochloric acid. An aqueous suspension of the acid had p_{H} 2.73.

A. G. POLLARD.

Influence of fertiliser treatment on the content of exchangeable cations in Hagerstown silt loam. F. G. MERKLE (Soil Sci., 1928, 26, 377—384).—Repeated use of fertilisers affects the relative proportions of replaceable bases in soil. Adsorbed ammonia from ammonium salts may under suitable conditions form a large proportion of the adsorbed cations, replacing considerable amounts of calcium. A portion of the potassium in fertilisers is retained in an exchangeable form. Other fertiliser salts tend to bring about the replacement of exchangeable calcium by hydrogen ions.

A. G. POLLARD.

Adsorption of potassium from different sources, and nitrification studies with Norfolk sandy loam. G. V. C. HOUGHLAND (Soil Sci., 1928, 26, 329—343).—Measurements of adsorption were made by leaching experiments. Addition of sodium chloride to a leaching solution of potassium chloride did not affect the amount of potassium adsorbed by the soil. Slightly more potassium was adsorbed from a sulphate solution than from a chloride solution, and the adsorbed potassium from the chloride was more easily displaced by acid than that from the sulphate. Potassium chloride retarded the nitrification of fish meal, sewage, and ammonium sulphate to an extent increasing with the amount of potassium chloride present. This effect was intensified by the admixture of sodium chloride. Slight stimulation of nitrification was observed following treatment with potassium sulphate.

A. G. POLLARD.

Distribution of nitrate in three layers of fallow soil. J. B. SMITH (Soil Sci., 1928, 26, 347—350).—Examination of a fallow soil to a depth of 2 ft. from the surface indicated that the loss of easily nitrifiable nitrogen during a season was very small. Alternate leaching by rain and the subsequent capillary rise of nitrates from the subsoil caused sufficient variations of nitrate concentration in the surface soil to explain nitrate deficiencies under a growing crop.

A. G. POLLARD.

Nutrient requirements of arable soils. B. DIRKS and F. SCHEFFER (Landw. Jahrb., 1928, 67, 789—821; Bied. Zentr., 1928, 57, 438—441).—The effect of carbon dioxide on the solubility of soil nutrients is examined. The solubility of soil phosphorus is favoured by an acid reaction and a reduced lime content. Extraction of fresh soil with water free from carbon dioxide shows the extracted phosphorus to be restricted according to the calcium carbonate present. Easily soluble phosphorus is determined by extraction with calcium bicarbonate solutions in the case of neutral and alkaline soils, and with water free from carbon dioxide for acid soils. A single extraction suffices to indicate the proportion of easily soluble phosphorus present, and any deficit from an agreed limiting amount is representative of the phosphate requirement of the soil. Comparison with standard methods is recorded.

A. G. POLLARD.

Relation of soil type to the exchangeable calcium and magnesium in some Illinois soils. M. P. CATHERWOOD and E. E. DE TURK (J. Amer. Soc. Agron., 1928, 20, 657—678).—A study of the relation in surface samples between exchangeable calcium and magnesium and soil type has been made and the results are summarised.

CHEMICAL ABSTRACTS.

Intake of nutrients and root development of barley. F. SEKERA (Z. Pflanz. Düng., 1928, 7B, 527—530).—During the early growth of barley the intake of nitrogen, phosphate, and potash increases steadily with the extent of the root system. The flowering period represents the maximum intake of nutrients and corresponds exactly with maximum root development. Subsequently no further nutrients are absorbed, and there is a definite shortening of the root system. During the formation and ripening of grain there is a transference of nutrients from stem and leaves to the grain. Under the conditions of the experiment some potash actually returns to the soil (cf. following abstract).

A. G. POLLARD.

Mineral changes in barley. F. SEKERA (Z. Pflanz. Düng., 1928, 7B, 533—539).—The decreased potash content of barley during ripening formerly observed (cf. preceding abstract) is confirmed. Fertilisation with phosphate and nitrogen leads to an increased intake of these nutrients by the plant and a smaller withdrawal of potash during the ripening of the grain. By increasing the amount of nitrogenous fertiliser the potash content of the barley is maintained throughout the later stages of growth; the nitrogen intake is increased but suffers a reduction during the ripening stage. It is suggested that whilst the plant can only utilise nitrogen, phosphorus, and potassium in definite proportions, the presence of relatively high concentrations of these

elements in the soil leads to absorption by the plant in amounts greater than the economic ratio. After the cessation of the intake of nutrients at the flowering period the surplus nutrient returns to the soil. The effect of the excessive proportions of mineral matter on the osmotic relationships of the cell sap and general growth of the plant is discussed. A. G. POLLARD.

Influence of chilling on certain crop plants. J. P. F. SELLSCHOP and S. C. SALMON (J. Agric. Res., 1928, 37, 315—338).—The effects of chilling, at temperatures above 0°, on plants of a tropical and subtropical character, e.g., cow-peas, velvet and soya beans, buckwheat, rice, tomatoes, etc., have been investigated, and experiments have been carried out with reference to the protective action of certain inorganic salts against injury by chilling. E. A. LUNT.

Bacteriology of silage. J. H. WALTON (Agric. Res. Inst., Pusa, 1928, Bull. No. 182, 13 pp.).—In laboratory ensilages of maize and *juar* (*Sorghum vulgare*) a good product always followed the production of 2% of (lactic) acid in the juice within 3—5 days. Slow development of acidity occurred with immature maize (moisture above 80%) which occasionally showed signs of slight putrefaction. This was more marked with *juar*, which always yielded a somewhat inferior product. The acid-forming organism was typical *Streptococcus lactis*, which increased in number from the first few days and subsequently diminished. A similar behaviour was shown by the organisms developing on agar plates, but the microscopic count showed a much less marked decrease.

F. E. DAY.

New method of soil analysis. F. SEKERA (Z. Pflanz. Düng., 1928, 7B, 530—533).—Sampling of soil by the customary methods for physical examination is unsuitable since natural conditions are destroyed. A large block of soil is removed with a spade and broken into smaller clods by allowing it to fall to the ground. An unbroken sample is selected, which contains no portion of the surface cut by the spade and is free from obvious large portions of stone, plant root, etc. A cylindrical tinned can about 10 cm. diam., with base and lid perforated, is lined with linen, and the unbroken sample placed therein embedded centrally in sand, with which the vessel is then completely filled. The sample is transported to the laboratory in the vessel, which is subsequently placed in water till soil and sand are saturated and allowed to drain thoroughly. The sand is then carefully removed from above the soil sample and a portion removed by a specially constructed borer. The water content of the sample determines the water capacity of the soil, and measurements of pore space etc. may be made with subsequent portions.

A. G. POLLARD.

The buffer method and the determination of exchangeable hydrogen for determining the amounts of lime required to bring soils to definite p_H values. W. H. PIERRE and S. L. WORLEY (Soil Sci., 1928, 26, 363—375).—Buffer capacities of soils can be determined by the dialysis-colorimetric method, and a 3-day time of contact of soil and baryta is necessary. The ratio of the amount of lime necessary to bring an acid soil to a definite p_H value in the laboratory to that

required in field or greenhouse conditions ("liming-factor") averaged 1.5. Soils treated with lime equivalent to their exchange-acidity reached p_H 6.5 in the majority of cases examined. Determinations of buffer action and exchangeable hydrogen (Parker's barium acetate method) are recommended as a basis for lime-requirement determinations. The "liming-factor" is considered as associated with the non-exchangeable soil complex. A. G. POLLARD.

See also A., Dec., 1406, Toxic action of electrolytes towards plants (MAUME and DULAC). Influence of soil reaction on ionisable constituents of tomatoes (HABER). 1407, Nitrogen and carbon nutrition of plants (BORNEMANN). Determination of migration of nitrogenous substances in plants (COMBES). Increasing the iodine content of plants (SCHARRER and STROBEL). Liberation of iodine by soils (SCHARRER and SCHWAIBOLD).

Soil corrosion studies. LOGAN and others.—See X.

PATENTS.

Manufacture of manures [fertilisers] containing nitrogen and potassium [and free from chlorine]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,402, 3.12.27 and 18.8.28).—Potassium bisulphate practically free from chlorine is produced by passing a hot gas or vapour, e.g., superheated steam, through a mixture of equimolecular proportions of potassium chloride and sulphuric acid, d 1.53—1.71, at about 110—120°, and subsequently evaporating off the water. The product is treated at the ordinary temperature with ammonia, e.g., in a revolving tube furnace, the material being heated to about 50° towards the end of the reaction. The product contains about 10% N and 30—35% K_2O , approximating to the formula, KNH_4SO_4 .

L. A. COLES.

Manufacture of a mixed fertiliser containing urea and phosphate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,329, 24.8.27).—Stable fertilisers are obtained by mixing with urea phosphates of aluminium and/or iron. These phosphates may be obtained on neutralising with ammonia the solutions of raw phosphates for the recovery of ammonium phosphate, where ordinarily they are insoluble waste products. Other fertiliser salts such as potassium chloride or sulphate may be admixed without affecting the stability. The mixing may be by either dry or wet methods, with subsequent drying or spraying in the latter case. The ratio $N:P_2O_5$ or $N:P_2O_5:K_2O$ may be varied within wide limits. P. E. L. FARINA.

Production of an alkaline reacting nitrogen fertiliser. N. CARO and A. R. FRANK (B.P. 279,421, 30.9.27. Ger., 23.10.26).—Ammonia is passed at 500—900° and at ordinary or increased pressure over granulated artificial or natural calcium carbonate, or over mixed carbonates such as dolomite, until a product containing 10—30% of combined nitrogen is obtained. W. G. CAREY.

Calcium cyanamide etc. (B.P. 279,811—2).—See VII. Drying of agricultural products (B.P. 300,758).—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Detection and determination of sucrose by the ammonium molybdate method. N. W. MATTHEWS (Maryland Acad. Sci. Bull., 1928, 7, 35–37).—The dilute solution, *e.g.*, condenser water (5 c.c.), is heated in a boiling water-bath for 6 min. with concentrated hydrochloric acid (3 drops) and 4% ammonium molybdate solution (3 c.c.); in presence of sugar a transient blue colour develops. Permanent standards for quantitative work may be prepared with ink or (for concentrations below 0.0125%) Fehling's solution.

CHEMICAL ABSTRACTS.

See also A., Dec., 1938, **Determination of reducing sugars** (VAN SLYKE and HAWKINS; HÉRISSEY and CHALMETA). 1360, **Polysaccharides** (DREW and HAWORTH). 1392, **Blood-sugar determinations and separation of sugars with yeast** (RAYMOND and BLANCO). 1402, **Alcoholic fermentation of sugars** (NEUBERG and KOBEL). **Transformation of cellulose into dextrose by bacteria** (WOODMAN and STEWART). 1408, **Gum arabic** (AMY).

New carbohydrate in rye flour. TILLMANS and others.—See XIX.

PATENTS.

Manufacture of starch and glucose. C. R. BROWN and H. E. NELSON (U.S.P. 1,690,360, 6.11.28. Appl., 31.10.25).—Insoluble iron salts are removed from starchy materials by treatment with dilute hydrochloric acid, or with an acid of similar avidity, and subsequent washing.

W. J. BOYD.

Manufacture of corn sugar. C. R. BROWN and H. E. NELSON (U.S.P. 1,690,359, 6.11.28. Appl., 31.10.25).—Corn grits, prepared by cracking, degerminating, and dehulling of corn kernels, are first treated with dilute mineral acid of a concentration just sufficient to solubilise the contained basic salts, and then washed prior to further treatment.

W. J. BOYD.

XVIII.—FERMENTATION INDUSTRIES.

Fermentation in open and closed vessels. F. W. WINDISCH (Woch. Brau., 1928, 45, 547–553).—Observations on actual brewery fermentations confirm the conclusions of Stockhausen and Windisch (B., 1928, 685) that fermentation in closed vessels, from which the carbon dioxide can be collected, improves the quality of the yeast and to a less degree that of the beer. It is stated that in the brewery in question the plant for recovery of carbon dioxide yields a nett profit of 25%.

F. E. DAY.

Milk-fermenting yeast. C. S. R. AYYAR (Agric. Res. Inst., Pusa, 1928, Bull. No. 183, 5 pp.).—The organism was isolated from spoiled "sterilised" milk, and in 48 hrs. cultures on purple lactose agar at 30° (optimum) appear as glistening colonies with a smooth edge. The cells are generally 5–7.5 μ long and 4.8 μ broad after 24 hrs. culture. No spores are formed at 25° or 30°. Dextrose, laevulose, galactose, sucrose, and lactose are fermented, but not maltose, raffinose, arabinose, or mannitol. It differs only in size and thermal death point (66°) from *Torula lactis* (Adametz).

F. E. DAY.

Influence of artificial acidification of mash or wort on the composition of the resulting worts and beers. W. WINDISCH, P. KOLBACH, and W. BÄNHOLZER (Woch. Brau., 1928, 45, 523–528, 535–539, 553–558).

—The composition of worts from malt mash prepared at different reaction was studied and their behaviour compared with that of worts adjusted to similar reaction after preparation in the ordinary manner. Acidification with hydrochloric or lactic acid gave similar results. When the final wort had p_H 5.1 (approx.), maximum extract was obtained, but the maxima for fermentable extract and apparent maltose were at about p_H 5.5 and 5.2, respectively. These differences were taken to indicate an increase of soluble nitrogenous compounds between p_H 5.5 and 5.1, and of reducing but non-fermentable dextrins between p_H 5.5 and 5.2. The optimum for proteolysis was about p_H 4.8, or possibly lower, but below p_H 5.2–5.3 a marked falling off of attenuative capacity was found, which puts a limit to the applicability of acidification in practice. The authors point out that the enzymes may have been affected by abnormal reaction of the mash after addition of acid or alkali before the buffering substances had gone into solution. The colour of alkaline worts was darker than that of acid worts, and boiling increased this difference. The difference was greater when the reaction was varied in the mash, probably owing to the effect on the oxidase. Acidification of the mash increased buffering over the phosphate (p_H 7.07–5.67) and protein (p_H 5.67–4.27) ranges, hence though the initial p_H values of such acidified worts were lower than that of normal worts, the further fall during fermentation was less and the p_H of the final beers was virtually unaffected. When normal worts were subsequently acidified the buffering was unaltered, and the p_H of the beers was below the normal value.

F. E. DAY.

Influence of formaldehyde on the germination of steeped grain. B. LAMPE (Z. Spiritusind., 1928, 51, 343–344).—The experiments were made with a barley which showed a germination of 98–99% in eight days under normal steeping conditions. The treatment of the grain during the first two days was divided into two periods, in each of which the corns were steeped in water for 16 hrs. followed by aeration for 8 hrs. On the third and following days the germinating grain was frequently turned and sprinkled. Additions to the steep water of 0.25% and upwards of formalin checked the germination and reduced the number of germinating corns by about 50%. If, however, 0.25% of formalin was added only at the second period of steeping, the diminution in the germinative energy of the corns was restricted to 5%. Similarly, if smaller additions of 0.15 and 0.20% of formalin were confined to the period of the second steep, the proportion of grains which had germinated by the eighth day was greater than with the barley steeped in untreated water, although up to the fourth day the converse held good.

C. RANKEN.

Separation of fusel oil from the first distillate. B. LAMPE and W. KILP (Z. Spiritusind., 1928, 51, 351–352).—The factors which govern the separation of fusel oil from the first distillate through the agency of added water are the relative volumes of water and

spirit, the proportions of ethyl alcohol to fusel oil, and the temperature of the mixture. The optimum separation results from admixture of the distillate and water in equal volumes. With less water an emulsion forms which clears with great difficulty, whilst with too great a proportion of water the yield of fusel oil is low owing to its solubility in the water. The separation of the fusel oil is incomplete if the content of ethyl alcohol in the distillate is high. With ratios of fusel oil to alcohol such as 1:0.73 and 1:1.24 the separation of oil is immediate, whereas with a ratio of 1:1.31 the separation is imperfect. In addition, the lower the temperature of the mixture of distillate and water the greater is the yield of fusel oil and the more rapid is the separation. Optimum results are obtained if cold water is added to the already cooled spirit distillate.

C. RANKEN.

See also A., Dec., 1400, **Activator of malt amylase** (NISHIMURA). **Invertase** (CANALS and GOMBERT). **Stereochemical specificity of ketone-aldehyde mutase** (NEUBERG and SIMON). 1401, **Decomposition of β -hydroxybutyric acid by liver enzymes** (KÜHNAU). **Crystalline urease** (SUMNER and HOLLOWAY). **Influence of ions on action of urease** (MYSTKOWSKI). **Uricase** (PRYZLECKI and TRUSZKOWSKI). 1402, **Uricase** (PRYZLECKI). **Relation of co-zymase to phosphatase activity** (RAYMOND). **Hexosephosphates and alcoholic fermentation** (RAYMOND and LEVENE). **Alcoholic fermentation of sugars** (NEUBERG and KOBEL). **Activator Z** (VON EULER and others). **Specificity of carboxylase** (NEUBERG and WEINMANN). **Proteolytic enzymes from bacteria** (MOYCHO). **Decomposition of olive oil by micro-organisms**. (PIGULEVSKI and CHARIK). **Growth of *Bacterium radiculicola*** (SNIESZKO). **Transformation of cellulose into dextrose by bacteria** (WOODMAN and STEWART). 1408, **Catalase content of conifer leaves** (DOYLE and CLINCH). **Rice; oxidase** (HIGUCHI). **Enzymes of wheat flour** (NEUENSCHWANDER).

Bacteriology of silage. WALTON.—See XVI.

PATENTS.

Invertase preparation and method of preparing and utilising the same. L. WICKENDEN, ASST. to J. J. NAUGLE (U.S.P. 1,689,607, 30.10.28. Appl. 6.11.25).—An invertase-containing preparation is prepared by heating yeast at 55–65° in the presence of a sugar syrup of pH 4–5.

W. J. BOYD.

Production of citric acid. A. FERNBACH and J. L. YULL, ASSTS. to ROWNTREE & Co., LTD. (U.S.P. 1,691,965–6, 20.11.28. Appl. 3.11.25).—See B.P. 266,414–5; B., 1927, 344.

Raw rubber from latex (B.P. 300,719).—See XIV. **Dry yeasts** (B.P. 300,039).—See XX.

XIX.—FOODS.

Inoculation of pasteurised milk. A. WOLFF (Milch. Zentr., 1928, 57, 277–283, 293–297, 341–346).—A description is given of the treatment of pasteurised milk, before or after heating, with a variety of lactic

acid bacteria which have been found to survive the pasteurisation process, in order that the milk may undergo a normal lactic fermentation on long keeping and not be spoiled.

F. R. ENNOS.

Provisional definitions for preserved milk products. F. E. NOTTBOHM (Z. Unters. Lebensm., 1928, 56, 63–72).—Criteria and methods of evaluation of preserved milk products are discussed, and provisional definitions drawn up by the Commission of the Verein Deutscher Nahrungsmittelchemiker and the Verbands Deutscher Dauermilchfabrikanten are given.

W. J. BOYD.

Electrometric determination of chlorine in milk. T. SUNDBERG (Z. Unters. Lebensm., 1928, 56, 32–38).—Various methods of making this determination are compared, and a new form of apparatus is described. The author recommends the use of acetic acid in place of sulphuric acid in the potentiometric method, 5 c.c. of glacial acetic acid being added to 20 c.c. of milk diluted to 150 c.c. The chlorine content of the milk can be obtained from that of the serum by multiplying by 0.96 in the case of milk containing 3.4% of fat or 0.99 in the case of skimmed milk. The potentiometric method is shown to be superior to that of Volhard.

W. J. BOYD.

A new carbohydrate in rye flour and the detection of rye flour in wheat and other flours thereby. J. TILLMANS, II. HOLL, and L. JARIWALA (Z. Unters. Lebensm., 1928, 56, 26–32. Cf. Schulze and Frankfurt, B., 1894, 533; 1895, 377).—The alcoholic extract of rye flour showed a higher alkali-binding power per unit of nitrogen than that of wheat flour. Also in titrating the alcoholic solutions with alkali a precipitate was obtained in the rye flour extract which proved to be the sodium salt of a carbohydrate, whereas the wheat flour extract remained clear. The precipitate was soluble in acidified water or 70% alcohol, but insoluble in alkaline 70% alcohol or in acidified 95% alcohol. The free carbohydrate was obtained by decomposing the sodium salt with 20% perchloric acid, neutralising the solution to litmus with sodium hydroxide solution, and adding a large bulk of absolute alcohol. The precipitate was separated by decantation of the liquid, washed with 96% alcohol, and dried *in vacuo* over sulphuric acid. The yield was 1% of the flour taken. The white crystalline substance so obtained corresponded in properties and elementary composition to trifructose anhydride. It was not found in maize, rice, oat, or barley flours. In the detection of rye flour in other flours, 5 g. of the sample are shaken for 15 min. with 20 c.c. of 70% alcohol and the mixture is cooled to –3° with frequent stirring for 10 min. in a mixture of ice and salt. The liquid is separated by centrifuging and decantation followed by filtration if necessary. 10 c.c. of the clear liquid are treated with 0.5 c.c. of N-sodium hydroxide solution in 70% alcohol. Pure wheat flour gives only a slight turbidity, whereas in the presence of rye flour a dense turbidity or precipitate is formed. In this way 10% of rye flour in a sample may be detected.

W. J. BOYD.

Detoxicated cottonseed meal. P. MENAUL (Oil & Fat Ind., 1928, 5, 333–335).—Gossypol may be

precipitated by light petroleum from an ethyl ether extract of cottonseed. The product is a highly active form of gossypol, and decomposes readily at 100° into water and a non-poisonous, insoluble, reddish caramel. Gossypol *in situ* in the seed-glands or dissolved in cottonseed oil is not so decomposed at 100°. By raising the moisture content of cottonseed meal to 40% and heating above 100° by steam under pressure, the gossypol is caused to exude to the surface of the meal and to undergo decomposition; thus a non-toxic meal is obtained which has been used as a poultry and cattle food with very satisfactory results, the proteins present having exceptional growth-promoting qualities.

E. LEWKOWITSCH.

Injury to onions and fruits caused by exposure to ammonia. G. B. RAMSEY and L. F. BUTLER (J. Agric. Res., 1928, 37, 339—348).—The yellow, brown, and red pigmented tissues of onions, bananas, apples, and other fruits when exposed to ammonia (0.8—3.2%) at 31.5° and R.H. 83% are discoloured brown or greenish-black. The injury varies from mere discoloration to actual softening of the complete fruit in the case of the pear, banana, and peach.

E. A. LUNT.

See also A., Dec., 1397, **Digestibility of certain varieties of oats and of bulrush millet** (HALMAN). **Effect of polishing and of methods of cooking of rice on its absorption** (SUGIMOTO and others). **Calorific value of soluble carbohydrates in feeding-stuffs** (ALLEN). **Nutritive value of linseed cake** (STEWART). 1405, **Colorimetric determination of vitamin-A** (VON EULER). **Effect of quinol on vitamin-A content of stored oils** (HUSTON and others). **Vitamin-A content of wheat oil** (SURE). **Vitamin-A and -B content of the pigeon pea** (MILLER). **Vitamin content of barley germ** (SCHITTENHELM and EISLER). **Concentration of vitamin-B** (LEVENTE). 1408, **Rice; oxidase** (HIGOCHI). **Enzymes of wheat flour** (NEUENSCHWANDER).

Milk-fermenting yeast. AYYAR.—See XVIII.

PATENTS.

Drying of hops, grain, and other like agricultural products. T. E. DAVIES (B.P. 300,758, 15.10.27).—Water-gas or semi-water-gas is burnt in a current of air which passes upward to the hops or other material supported on racks in the upper part of the building. Advantages claimed are economy of fuel and a slight bleaching action on the material.

W. J. BOYD.

Manufacture of flour. E. A. FISHER and C. R. JONES (B.P. 300,291 and 300,537, 10.6.27).—(A) A current of moist, hot air is circulated for not more than 3 hrs. over thin layers of flour on trays so that the flour is kept at 54—82° without substantial loss of moisture. The R.H. of the heated air should not fall below 60%. The maximum improvement is effected in 1½—2 hrs. at 60° or in 10—20 min. at 71°. The product gives dough of greater stability and water absorption, loaf of greater volume and better shape, and frequently a better colour of crumb. (B) The flour is thrown into the form of a cloud and treated by a

strong air-current from which it is subsequently recovered by means described.

W. J. BOYD.

Manufacture of a flour improver. N. V. NOURY & VAN DER LANDE'S HANDELSMAATSCHAPPIJ, and J. A. L. VAN DER LANDE (B.P. 300,515, 12.5.27).—A wheaten cereal is heated at atmospheric pressure in a stationary atmosphere, without increasing the moisture content, to 70—90° until 5—70% of the gluten becomes non-retainable in a washing test in which the dough is kneaded under running water. As a moisture content of 15—20% in the cereal is desirable, a closed vessel may be used, and exclusion of oxygen or the presence of indifferent gases or vapours is often found to be favourable. The improver so obtained causes a marked increase in the strength of flour to which 0.5—2% has been added, but it will not by itself yield a satisfactory bread.

W. J. BOYD.

Manufacture of cassava meal. S. W. DUNCAN (B.P. 300,673, 15.8.27).—Before being dried, the grated roots are treated in a suitable press whereby the greater part of the starch, a valuable by-product, is removed in the expressed liquid.

W. J. BOYD.

Manufacture of cheese. L. H. STEDEFORD (B.P. 298,174, 1.7.27).—Curd is ground in a coarse grinding mill and then emulsified and pasteurised by agitation in a thin layer at 60—68° in a jacketed cylinder inside which is mounted a revolving drum with vanes; the semi-liquid cheese is then cooled and solidified in moulds. An emulsifying salt, or sour milk, a lactic acid culture, or ground mature cheese may be added to the ground curd.

W. G. CAREY.

Medicated animal food stuffs. O. STINER and B. DIETHELM (B.P. 274,915, 26.7.27. Switz., 26.7.26).—Bran and similar seed husks are mixed with a dilute solution of a medicament (potassium iodide) arsenious oxide, strychnine, with or without heating, until swelling occurs, when the mass is dried.

B. FULLMAN.

Manufacture of iron compounds of the phosphorus-containing bodies of egg-yolk proteids. S. and T. POSTERNAK (B.P. 283,866, 17.1.28. Switz., 17.1.27. Cf. B.P. 268,805—6; B., 1928, 138, 315).—An excess of a solution of a soluble iron compound (*e.g.*, ferric chloride) is added to a suspension or solution of egg yolk before or after digestion with pepsin and trypsin or to a solution of the α - or α - and β -compounds described in B.P. 268,806 (B., 1928, 315), and the iron compound is precipitated by neutralising or acidifying the alkaline solution. The phosphorus-containing α -compound described in B.P. 268,806 (*loc. cit.*) binds not only 2 equivalents of iron for each phosphoric acid radical present, but somewhat more than 3 equivalents, so that when the substance is precipitated by a mineral acid in presence of 1.2 and fewer equivalents of iron the iron is "organically combined" (*i.e.*, otherwise than as a salt), and the free phosphoric acid hydroxyl groups can be subsequently saturated by alkali or alkaline-earth. The acid iron salts are yellowish-white and contain about 14.5% Fe and 10.5% P. The normal iron salts formed by mixing a neutral solution of the α - and β -compounds

with excess of a soluble iron salt contain about 18% Fe and 9.5% P and are reddish-yellow. W. J. BOYD.

Preservation of beverages. F. J. ANDRESS, Assr. to BROWNIE CORP. (U.S.P. 1,691,538, 13.11.28. Appl., 16.5.25).—The milk or flavouring beverage is treated with hydrogen peroxide at a temperature at which the latter is an active germicide without injurious action on the beverage. The acidity of the resulting product is removed by adding a sufficient quantity of an innocuous neutralising agent. W. J. BOYD.

Destruction of insect pests in food products and other material. A. M. KOBIOŁKE (B.P. 300,529, 9.8.27).—The material is placed in a gas-tight kiln which is then exhausted. Carbon monoxide is admitted till the pressure in the kiln is equal to that of the atmosphere, and the gas is withdrawn again by means of a vacuum pump. Carbon disulphide vapour or other fumigating gas is then passed into the kiln and again withdrawn. Finally air is admitted and circulated by means of a suction pump and fans. The carbon disulphide container may be heated by steam coils to facilitate vaporisation of the fumigant, and the kiln may be heated similarly to increase the effect of the fumigant.

W. J. BOYD.

Wrapping material (B.P. 292,162).—See X.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Stability of anæsthetic ether. F. W. NITARDY and M. W. TAPLEY (J. Amer. Pharm. Assoc., 1928, 17, 966—968).—The influence of storage conditions on the formation of peroxides in anæsthetic ether has been examined. Ether stored in tins or glass containers was found to develop peroxides in every case investigated. In general, most anæsthetic ether will form peroxides within six months after manufacture, and they will remain in the ether for one or more years and then gradually disappear with formation of aldehyde, which ultimately is transformed into acid. When exposed to heat and pressure ether developed peroxides in 1 hr. The formation of peroxides can be prevented by storing ether in containers the interior of which presents an adequate surface of copper or copper salts. Samples stored under these conditions developed no peroxides under storage of several years or with heat and pressure treatment for 60 hrs. Copper does not remove peroxides from ether. E. H. SHARPLES.

Stability of morphine in aqueous solution, especially on sterilisation. C. STICH (Pharm. Ztg., 1928, 73, 1513).—In view of a publication by Dietzel, attention is drawn to the work of Schäfer and Stich (Münch. Med. Wochenschr., 1917, 676), in which it was shown that the yellow decomposition product formed when morphine hydrochloride solutions are sterilised does not affect the absorption spectrum, and is probably insignificant, and in which it was suggested that a trace of hydrochloric acid should be added to such solutions in order to counteract the alkalinity of the glass vessels.

E. W. WIGNALL.

Process of formation of essential oils and resins in conifers. XI. Composition of the oil from the

needles of *Pinus silvestris*. Process of formation of essential oils and resin in pine trees. G. W. FIGULEVSKI and N. B. RISKINA (J. Russ. Chem. Soc., 1928, 60, 1069—1077).—The essential oil and resin obtained by steam distillation of the needles of *Pinus silvestris* was fractionated and analysed. α -Pinene (α_D —14.88°), esters, free alcohols (all strongly *lævo*-rotatory), and *l*-cadinene (α_D —5.52°, α_F/α_C 8.96, b.p. 127—132°/10 mm.) were present. The non-volatile resin consisted of free acids, together with a certain amount of esters. In order to elucidate the process of formation of essential oils and resin in the needles and twigs of pine trees, the quantity and nature of these products were examined in a number of individual trees from the forests near Leningrad. The amount of essential oils varies between 0.21% and 1.30%, and the optical rotation from α_D +10.84° to α_D —15.94°. The resin content was 7—12%. The essential oil contained in twigs (0.46—1.28%) had α_D —8.16° to 47.17°.

M. ZVEGINTZOV.

See also A., Dec., 1313, Adsorptive power of aluminium silicates used pharmaceutically (PORTILLO). 1328, Pharmaceutical combinations (PFEIFFER and SEYDEL). 1329, Formation and decomposition of sodium salicylate (DAVIES). 1332, Explosibility of anæsthetics (HORNER and GARDENIER). 1348, Determination of bismuth in substances for treatment of syphilis (BOUILLENNE and DUMONT). 1374, Derivatives of *p*-methoxycinnamic acid (FOOTE). 1375, Pungent principles of ginger (NOMURA and IWAMOTO). 1376, Plumbagin (MADINAVEITIA and GALLEGO). Digitalis glucosides (JACOBS and GUSTUS). Strophanthin (JACOBS and others). 1377, Occurrence of *d*- β -pinene (RUTOVSKI and VINOGRADOVA). Mono- and sesqui-terpene series. West Indian sandalwood oil (DEUSSEN). 1380, Preparation of ethyl phenylmalonate and of 5-phenyl-5- β -hydroxyethylbarbituric acid (NELSON and CRETCHER). 1386, Alkaloids of *Hyoscyamus reticulatus*, L. (KONOVALOVA and MAGIDSON). Chloroarsinosoquinine (ERBEN). Tests for morphine and related alkaloids (FULTON). Cresolarsinic acids (FINZI). 1387, Preparation of 4-chaulmoogrylaminobenzenearsinic acid (DEWAR). 1388, Compounds of albumin and metallic salts (BECHHOLD; SCHORN; HEYMAN and OPPENHEIMER). 1389, Colour reactions of the bile acids (CUNY). 1393, Determination of thymus-nucleic acid (WIDSTRÖM). 1399, Toxicology of anæsthetics containing bromine and chlorine (LUCAS). 1403, Isolation of secretin (MELLANBY). 1404, Evaluation of thyroid preparations (MORCH).

PATENTS.

Manufacture of albumin preparations [containing silicates]. A. BUSCH, Assr. to J. A. VON WÜLFING (U.S.P. 1,688,228, 16.10.28. Appl., 18.12.24).—Commercial sodium silicate solution is treated with sodium hydroxide sufficient to convert it into the metasilicate, and casein is added to the diluted solution as long as it dissolves readily. The liquid is then evaporated under reduced pressure to obtain a water-soluble powder of therapeutic value. A. R. POWELL.

Photochemical production of vitamin-D from ergosterol. I. G. FARBENIND. A.-G. (B.P. 296,093, 22.8.28. Ger., 26.8.27).—A solution of ergosterol is exposed to ultra-violet radiation, the exposure being interrupted before the point of maximum absorption between $\lambda 250$ and $\lambda 230$ is substantially exceeded. Under these conditions the presence of a small proportion of unaltered ergosterol may be demonstrated. Using a photoelectric cell a device may be arranged whereby the source of radiation is automatically cut off when the maximum content of vitamin-D has been attained, or the rate of flow of the solution through the irradiation vessel may be adjusted so that the required period of exposure is obtained.

W. J. BOYD.

Manufacture of dry yeasts for medical and pharmaceutical purposes. G. M. CLARK. From MATRO GES.M.B.H. (B.P. 300,039, 24.12.27).—Fresh, moist (or freshly dried) yeast, if necessary after treatment with water or liquids of alkaline reaction, is extracted with pure or denatured alcohol at 55–65°, pressed, washed, dried, and finally roasted at 150–160° (to remove traces of solvent). The by-products include phosphorus derivatives resembling lecithin, a product containing phosphorus and sulphur, and crude fat.

B. FULLMAN.

Manufacture of a new medicine [from iris flowers]. C. A. WRIGHT (B.P. 299,819, 2.6.27).—The flowers are treated with, e.g., methylated spirit, the spirit is allowed to evaporate, and the flowers, together with the deposited extract, are boiled with water containing a small quantity of sugar; or the alcoholic extract is decanted, the flowers are boiled with water containing sugar, and the aqueous and alcoholic extracts united after straining. An ointment is prepared by mixing the flowers, after pulverisation in a mortar with methylated spirit, with lard etc. and sieving.

B. FULLMAN.

Hæmostatic. F. MARTINEZ and A. GOMEZ (U.S.P. 1,690,869, 6.11.28. Appl., 10.8.26).—The preparation consists of an aqueous extract of the active content of fibres from the outside of the fruit of *Cocos nucifera*.

H. ROYAL-DAWSON.

Enteric coated capsule. E. H. VOLWILER, Assr. to ABBOTT LABORATORIES (U.S.P. 1,690,760, 6.11.28. Appl., 5.1.27).—A gelatin shell containing a medicine, or a gelatin medicinal capsule, is covered with a cellulose ester by coating with a cellulose lacquer. F. G. CLARKE.

Manufacture of amino-aromatic esters for use as anæsthetics. E. RITSERT (U.S.P. 1,692,224, 20.11.28. Appl., 27.4.26. Ger., 14.5.25).—See B.P. 260,493; B., 1927, 60.

Manufacture of anæsthetics, benzamine borate, and pharmaceutical products. W. J. POPE, Assr. to BRIT. DRUG HOUSES, LTD. (U.S.P. 1,692,695—8, 20.11.28. Appl., [A] 18.3.26, [B—D] 7.12.26. Renewed [D] 11.9.28).—See B.P. 260,346; B., 1927, 29.

Manufacture of therapeutically-active aromatic compound containing mercury in a lateral chain. M. BOCKMÜHL and A. SCHWARZ, Assrs. to WINTHROP

CHEM. Co. (U.S.P. 1,693,432, 27.11.28. Appl., 5.5.23. Ger., 12.5.22).—See Austr. P. 99,678; B., 1926, 218.

Egg-yolk proteids (B.P. 283,866). Medicated animal feeds (B.P. 274,915).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic emulsions. F. WEIGERT and F. LÜHR (Z. Elektrochem., 1928, 34, 605–610).—Experiments show that the light-sensitive constituent both in process emulsions and also in silver halide emulsions not containing excess of soluble silver salts is mainly metallic silver in complex combination with the silver halide in the gelatin. "Primary- (or ripening-) silver" and "photo-silver" (cf. Weigert, B., 1926, 109) have different colours and absorption spectra, which explains the varying behaviour of emulsions of different opacities and under differently coloured illuminations. The "primary-silver" in unilluminated emulsions is determined by the analytical method of Cox (A., 1925, 999). Relatively large quantities occur in process emulsions, due to reduction of the excess of soluble silver salts by the gelatin during the ripening. The amounts formed of both the "primary-" and "photo-" silver increase during illumination more and more rapidly in successive equal time intervals; the total quantity of such silver increases rapidly with the ripening time in the cases of chloride and bromide emulsions, and decreases slowly in the case of iodide emulsions. The light sensitivity increases with the amount of "primary-silver." The results of Eggert and Noddack are adversely criticised (cf. A., 1925, 573).

S. K. TWEEDY.

Photographic applications of diazo compounds. D. A. SPENCER (Phot. J., 1928, 68, 490–494).—Diazo compounds suitable for photographic papers should be sufficiently sensitive to light, and stable towards heat, mechanical shock, and to the paper base upon which they may be coated in sufficient concentration by dissolution in an inert solvent. They should couple readily with amines and phenolic compounds to give dark-coloured substances which are insoluble in water and stable to light. The decomposition products should not interfere with the colour or stability of the image. The following types of paper are possible: (a) the diazo compound is decomposed by light and is developed by application of an alkaline coupler which reacts only with the unchanged diazo compounds; (b) the diazo compound and coupler are coated together, but reaction is prevented by the presence of an organic acid, such papers being developed by weak alkali solutions or ammonia vapour; (c) the diazo compound is coated with a coupler in an unreactive state, e.g., as an additive compound, development being effected by breaking up the additive compound by heat, ammonia, etc.; (d) the diazo compound is decomposed by light into a substance which will react with the original diazo compound either on keeping or in presence of alkali.

J. W. GLASSETT.

Fogging by acids and oxidising agents and the intensification of the photographic latent image.

E. P. WIGHTMAN (J. Amer. Chem. Soc., 1928, **50**, 2923—2929).—Further evidence is given in support of the author's theory of the cause of the latent fog produced by the action of certain oxidising agents and acids on silver emulsions (cf. B., 1927, 765), in answer to the criticism of Lüppo-Cramer (B., 1928, 285, 731).

S. K. TWEEDY.

See also A., Dec., 1295, **Spectrum temperature of magnesium- and flash-light** (ARENS and EGGERT). 1297, **Photo-electric properties of alkali metals** (CAMPBELL). 1321, **Action of light on silver hydro-sols** (GALECKI and PSYCHALSKI). 1338, **Light reaction between ferric chloride and oxalic acid** (KORNFELD and MENCKE). 1339, **Photolysis of medium-free silver bromide** (KIESER). 1340, **Photochemistry of silver halides** (SCHMIDT). 1341, **Action of light on celluloid stained with malachite-green** (RAY-LEIGH).

PATENTS.

Photographic plates. E. S. VILASECA (B.P. 300,809, 9.12.27).—One side of the glass to be used for the support of photographic emulsions, not the coating side, is roughened by means of sand or hydrofluoric acid. Retouching pencils etc. may then be applied directly to the rear side of finished negative without the necessity of varnishing.

J. W. GLASSETT.

Pyro-recording paper and the like, suitable for use in picture and the like telegraph receiving apparatus. MARCONI'S WIRELESS TELEGRAPH CO., LTD. (B.P. 282,759, 31.12.27. U.S., 31.12.26).—The following solutions, if coated upon a suitable paper base, will afford recording papers which are practically insensitive to light and rapidly give a permanent discoloration by the application of heat alone without any subsequent treatment: (a) 100 pts. of water, 10 pts. of gelatin, 5 pts. of sodium chloride, and $\frac{1}{2}$ pt. of phenolphthalein; (b) 9.8 g. of aniline hydrochloride, 99 c.c. of water, 0.026 g. of gelatin, and 8 g. of potassium nitrate; (c) 100 c.c. of water, 5 g. of nickel nitrate, 3 g. of sodium thiosulphate, 3.6 g. of sodium nitrite, and 5 c.c. of a 1% solution of gelatin. The best results are obtained if the heat is applied by means of a jet of hot air which travels backwards and forwards across the paper; the heat from an electric spark may also be used.

J. W. GLASSETT.

Composition for joining strips of motion-picture films. O. W. COOK, ASSR. to EASTMAN KODAK CO. (U.S.P. 1,690,622, 6.11.28. Appl., 6.5.25).—A dye which absorbs light having wave-lengths in the red and orange portions of the spectrum is added to the composition in order to render it visible under light of these wave-lengths.

F. G. CLARKE.

Colouring the image on photographic plates, films, etc. G. WHEELER (U.S.P. 1,693,500, 27.11.28. Appl., 19.1.27. U.K., 27.2.26).—See B.P. 271,578; B., 1927, 621.

Production of coloured pictures from photographic prints. E. H. FARMER (B.P. 289,411, 26.4.28. U.S., 26.4.27).—See U.S.P. 1,669,869; B., 1928, 549.

XXII.—EXPLOSIVES; MATCHES.

Rapid determination of picric acid in pastes obtained by the nitration of phenol. L. DESVERGNES (Ann. Chim. analyt., 1928, [ii], **10**, 317—320).—The volume V of a large weight P of the paste of picric acid and its saturated solution in water are determined; then if p_1 is the weight of dry picric acid in the mixture, and p_2 the weight of the saturated solution present, $p_1 + p_2 = P$ and $p_2 = (1.56178V - 1.005P)/0.551$. From these equations the weights of acid and water in the mixture are calculated and the percentage of dry acid in the mixture is then readily found.

A. R. POWELL.

Bleaching of gangwa wood (*Excoecaria agallocha*) in the manufacture of matches. R. L. DATTA and T. BASU (Dept. Industries, Bengal, Bull. No. 18, 1928, 6 pp.).—In the bleaching of gangwa wood so as to render it suitable for match splints, a cold, alkaline, aqueous solution of bleaching powder containing 0.20% of available chlorine gave the best results. Boiling with water or treatment with sulphurous acid or potassium permanganate solution was unsuitable.

S. BINNING.

Preparation of a solution containing phosphoric acid direct from bone ash for the impregnation of match sticks to render them non-glowing. R. L. DATTA and T. BASU (Dept. Industries, Bengal, Bull. No. 27, 1928, 6 pp.).—The sticks are impregnated with a solution containing phosphoric acid obtained by extracting 3 pts. of bone ash with 3 pts. of commercial sulphuric acid (d 1.55). A suitable density for this solution is 1.035; it then contains 3.78% P_2O_5 and 0.19% H_2SO_4 of which 0.05% is free acid. This free acid turns the splints brown, but neutralising the acid with ammonia solution overcomes this defect where necessary.

S. BINNING.

Chemical composition for matches. R. L. DATTA and T. BASU (Dept. Industries, Bengal, Bull. No. 23, 1928, 4 pp.).—Unsuccessful attempts were made to form a damp-proof composition for matches by rendering the glue in it insoluble by treatment with formaldehyde or tannin before the remaining components were added. Compositions containing potassium dichromate gradually became damp-proof as a result of the oxidation of the glue by this ingredient.

S. BINNING.

See also A., Dec., 1366, **Reactions of stable azides** (BERTHÉ).

Explosibility of coal dusts. MASON and WHEELER.—See II.

PATENTS.

Nitration of glycerin, glycol, and similar alcohols. A. SCHMID and J. MEISSNER (B.P. 284,700, 28.1.28. Ger., 5.2.27).—Glycerin etc. is nitrated by circulating the reaction mixture, by means of a screw or air-injector, between two separate compartments in the same nitrator, the separation between the two being formed by cylindrical cooling coils, the innermost coil of which has its windings in close contact with one another. One compartment serves for cooling the mixture, and

the other for mixing acid and glycerin etc. and distributing the heat of the reaction. Nitration may be carried out continuously by withdrawing a small portion of the reaction mixture.

S. BINNING.

Denitration of waste sulphuric acid mixtures.

W. BÜSCHING (B.P. 276,972, 9.8.27. Ger., 3.9.26).—Sulphuric acid vapours from the dephlegmating column of a still in which sulphuric acid is being concentrated are used to denitrate a waste sulphuric acid-nitric acid mixture in a second column. The nitric acid vapours given off during the denitration may be washed and dried by sulphuric acid in a third column and condensed to give concentrated nitric acid. By combining the processes a continuous denitration of mixed acid with recovery of concentrated sulphuric and nitric acids is effected.

S. BINNING.

Nitroglycerin dynamite.

T. O'HERN, Assr. to A. T. O'HERN (U.S.P. 1,690,872, 6.11.28. Appl., 2.4.26).—The dynamite contains sodium nitrate 64 pts., infusorial earth 30 pts., comminuted wood product 20 pts., potassium nitrate 3 pts., ground magnesium carbonate 3 pts., and nitroglycerin 180 pts. by wt.

H. ROYAL-DAWSON.

Manufacture of heavy-metal azides. O. MATTER (B.P. 300,401, 25.11.27).—Sodium azide for use in the manufacture of lead azide is freed from alkali carbonate by adding hydroxides or salts of alkaline-earth metals to its aqueous solution; the alkaline-earth carbonate is precipitated and may be removed by filtration. The filtrate may be converted into solid sodium azide or used directly in solution for the preparation by a highly active lead azide, free from contamination by lead carbonate.

S. BINNING.

Manufacture of smokeless [flashless] explosives.

F. I. and E. DU PONT, and U.S.F. POWDER Co. (B.P. 298,543, 7.4.27).—See U.S.P. 1,627,638—9; B., 1927, 542.

XXIII.—SANITATION; WATER PURIFICATION.

Design of sludge digestion tanks. R. H. GOULD (Proc. Amer. Soc. Civil Eng., 1928, 54, 2655—2665).—In order that sewage sludge may be efficiently and economically reduced to an inoffensive state, digestion under anaerobic conditions in separate digestion tanks is recommended. These tanks should be of such a design that the fresh sludge may be added daily and distributed throughout the tank so as to mix evenly with the riper sludge already present. Fresh solids to the extent of 2% of the ripe sludge (on a dry basis) may be added daily without seriously interfering with the process. Some sludge should be withdrawn daily and either disposed of at once or stored until conditions are suitable for disposal. During the digestion period the sludge becomes denser, and so water, as free from objectionable solids as possible, must be withdrawn periodically. Covers should be provided for the tanks, even if it is not desired to collect and utilise the gases produced, as by this means unpleasant odours are suppressed, anaerobic conditions are maintained, and the formation of a scum of semi-dried sludge which might easily become objectionable is prevented. The optimum p_H value

and temperature should be maintained by addition of lime and by heating; the rate of digestion may thereby be so increased that the tank capacity required may be reduced from 2.5 to 0.9 cub. ft. *per capita*, thus permitting a considerable reduction in capital cost which would more than outvalue the cost of control.

C. JEPSON.

Disposal of septic-tank effluent [at Dacca] by dilution, irrigation, and digestion. F. C. GRIFFIN (Inst. Civil Eng., Selected Eng. Papers, No. 65, 1928, 13 pp.).—A general description of the disposal of sewage at Dacca is given.

C. JEPSON.

Electrolytic corrosion prevention of condenser tube corrosives.

F. G. PHILO (J. Amer. Water Works' Assoc., 1928, 20, 505—534).—Corrosion in condenser tubes may be due to (a) dezincification, *i.e.*, the dissolution of both copper and zinc and redeposition of the former resulting in a layer or spot of porous copper which may become the seat of deeper corrosion; (b) local differences of potential set up by foreign substances deposited in the tube, by air clinging to the walls, or by the existence of a permanent air space; (c) by differential aeration due to alternate wetting and drying of the surface, as when emptying the condenser; (d) high velocity and turbulent flow which tend to remove the metal ion or so facilitate further change from metal to ion by reducing the back *E.M.F.*; (e) stray electric currents which may reverse the normal state of iron anode and brass cathode and start corrosion on the brass side of the element. The corrosion may be reduced considerably by attention to the design of the plant with the object of obtaining uniformity of flow over all parts of the tube surface with a gradual change of flow at the inlets and low condenser velocities. Nozzles about 8 in. long with about half of their length inserted in the ends of the condenser tubes have been shown to carry the major part of the corrosion, and being easily removed and replaced considerably lengthen the effective life of the tube proper. The tubes should be of well-annealed, close-grained metal, and should be frequently cleaned to remove deposit. Three distinct forms of deposit have been observed, one on the steam side which was black, and consisted of copper and zinc from the tube itself and a certain amount of organic matter, probably oil, and two on the water side, one green and one brown. The green one, which was next to the metal, was a mixture of the hydroxides, oxychlorides, and basic carbonates of zinc and copper, and the brown one, which covered it, was similar except for a quantity of ferric hydroxide. Protection from stray electric currents, which is of special importance in power stations and on ships, can be given by making the tubes the cathodes of an electrolytic cell having an *E.M.F.* of 6—10 volts. The anodes used (cast iron blocks weighing 30—40 lb.) are placed in the water-box but insulated from it. The direction of the current is thus maintained so that corrosion occurs at the iron electrode instead of at the brass. The current density should be about 2.5—5.0 amp. per 1000 sq. ft. of cooling surface. The types of corrosion met with in power station practice are illustrated by photographs of actual tube sections.

C. JEPSON.

Chlorophenol tastes from creosoted wood-stave pipe. D. E. KEPNER (J. Amer. Water Works' Assoc., 1928, 20, 473—478).—The use of creosote as a preservative for wood staves in pipes used for carrying drinking water is shown to be the cause of the chlorophenol taste present when a chlorinated water is supplied. The extent to which the offensive taste develops depends on the amount of creosote taken up by the water and the amount of residual chlorine in the water entering the pipe when prechlorination is practised. C. JEPSON.

Chlorinated copperas—a new coagulant [in water purification]. L. L. HEDGEFETH and N. C. and W. C. OLSEN (J. Amer. Water Works' Assoc., 1928, 20, 467—472).—Copperas, oxidised to the ferric state by means of an aqueous solution of chlorine in the proportion of 7.8 pts. of copperas to 1 pt. of chlorine, is an efficient and economical coagulant for colour removal. The addition of this reagent must be followed by an alkaline coagulant to remove the more resistant colour and ensure satisfactory precipitation of the metal of the coagulant. To be effective the primary coagulation must take place at p_H 4.2—4.7 and the secondary at p_H 5.5—6.8. C. JEPSON.

Succinchloroimide for the treatment of small quantities of potable water. C. B. WOOD (J. Amer. Water Works' Assoc., 1928, 20, 535—549).—A satisfactory disinfectant for small quantities of potable water, especially under the conditions obtaining with troops in the field, should be (a) solid, easily soluble in water, and without offensive taste or appearance; (b) stable and retain its disinfectant power on keeping under adverse conditions; (c) suitable for use in small quantities so that it can be used by the individual soldier without supervision and be without harmful internal effect. The author suggests that succinchloroimide meets the above conditions. Its bactericidal powers appear to be satisfactory, but its toxic effect on the human system has yet to be determined. C. JEPSON.

The oxygen consumed determination [in water analysis]. G. W. BURKE (J. Amer. Water Works' Assoc., 1928, 20, 677—683).—In order to remove proved inaccuracies in the American standard method of determining the oxygen consumed from potassium permanganate when dealing with wastes of high organic content, it is suggested that the amount of permanganate taken should be increased from 10 c.c. to 25 c.c., and that at least 10 c.c. instead of 5 c.c. should remain unaffected after digestion for 30 min. in boiling water. The amount of sample taken (made up to 100 c.c. with distilled water) should be adjusted to reduce as nearly as possible 15 c.c. of potassium permanganate. Oxalic acid is recommended in place of ammonium oxalate when determining excess permanganate. A blank determination using 100 c.c. of distilled water is also advisable. C. JEPSON.

The phototurbidimeter in the determination of water turbidity. A. A. BADO and R. A. TRELLES (J. Amer. Water Works' Assoc., 1928, 20, 674—676).—When light rays of equal intensity are passed through transparent containers of equal size, one containing the water the turbidity of which is to be measured and the other

the same water freed from turbidity by passing through a candle filter, the intensities of the rays resulting are inversely proportional to the respective turbidities. A modified Bunsen photometer designed for this purpose and the method adopted at Buenos Aires are described.

C. JEPSON.

Elimination of false presumptive tests [in the bacterial analysis of water]. A. C. JANZIG and I. A. MONTAUK (J. Amer. Water Works' Assoc., 1928, 20, 684—695).—By increasing the p_H value of the lactose broth used from the normal of 6.8 to 8.0 before sterilisation an elimination of 47% of false presumptives was effected, whilst true *B. coli* results were concordant. The change in p_H value was found to have no effect on pure cultures of *B. coli-aerogenes* organisms beyond an increase in the rate of production of gas. C. JEPSON.

Determination of the bacterial count of water. W. PLÜCKER and W. BARTELS (Z. Unters. Lebensm., 1928, 56, 51—60).—The nutrient medium of Prall (Arb. a. d. Kaiserl. Gesund., 1902, 18, 436) containing 5% of gelatin and 0.75% of agar is much superior to media containing 10% of gelatin for determination of the bacterial count of water. The most suitable p_H value is 7.0. For the counting of colonies the automatic counting apparatus of Brudny (Zentr. Bakt. I., 1911, 57, 478) is recommended, or for microscopic counting the method of Hesse-Niedner (Z. Hyg., 1906, 53, 267). W. J. BOYD.

PATENTS.

Heatable sludge-digestion chamber for sewage treatment. K. IMHOFF (U.S.P. 1,692,446, 20.11.28. Appl., 18.6.27).—The sludge-digestion chamber of a two-storey settlement tank is subdivided by means of a partition in the form of a grid, and may be heated at its lowest portion to facilitate the process of digestion.

C. JEPSON.

Disincrusting and purifying composition for boiler feed-water. H. BREYER (B.P. 284,629, 1.2.28. Fr., 1.2.27).—A mixture of myrobolan, quebracho, sodium bicarbonate, and sodium fluoride either dry or in aqueous solution, is used. W. G. CAREY.

Treatment of polluted acid wastes. J. T. TRAVERS, Assr. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,685,300—1, 25.9.28. Appl., [A] 21.6.27, [B] 9.7.28).—(A) The liquid waste is passed through porous calcium carbonate, the period of contact being 2—5 min. (B) The calcium carbonate used has a porosity equal to that of travertine. W. G. CAREY.

Furnaces for burning sewage sludge and similar materials. E. V. SPRINGBORN (B.P. 301,295, 19.1.28).

[Automatic devices for use in] the treatment of water. FILTRATORS, LTD., and V. SAKS (B.P. 300,664, 18.5.27).

Apparatus for cleaning sand filter-beds and the like. J. PEEBLES (B.P. 301,006, 23.8.27).

Water sterilisers. J. SLATER & Co. (ENGINEERS), LTD., and J. C. ROBINSON (B.P. 300,718, 27.8.27).

Base-exchange silicate (U.S.P. 1,687,036).—See VII. **Indicator for purity of liquids** (B.P. 300,229).—See XI. **Destruction of pests** (B.P. 300,529).—See XIX.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEBRUARY 1, 1929.

I.—GENERAL; PLANT; MACHINERY.

Evaporation at low temperatures [in the laboratory].—E. JANTZEN and H. SCHMALFUSS (Chem. Fabr., 1928, 701—703).—A copper bath with outer sheet-iron jacket is heated by a small burner, and the liquid contents are stirred by a high-speed screw running within a small vertical pipe inside the bath. The distilling flask of glass or Krupp V2A steel is connected by a wide glass tube to a copper condenser with seven longitudinal cooling-water tubes. The condensate passes to Woulff's bottles, connected to several water pumps and to a manometer. This apparatus will distil 7 litres of water per hr. at a temperature 6° above that of the cooling water. By the use of ice in place of cooling water liquids can be distilled at temperatures down to -19°, the bath being filled with a freezing mixture.

C. IRWIN.

Viscosimeters for oils. BLEYBERG, also HOLDE.
Fractionating columns. KOSTRIN.—See II. **Monel metal as tower packing.** WEISSENBERGER and PIATTI.—See X. **X-Ray equipment.** CLARK.—See XI. **High-vacuum grease.** HEINRICH and PETZOLD.—See XII.

PATENTS.

Furnaces. CARBORUNDUM CO., LTD., ASSEES. OF C. E. HAWKE (B.P. 282,720, 9.11.27. U.S., 27.12.26).—A "radiating combustion chamber" or carborundum tube, having a burner at one end, within which combustion takes place in a somewhat confined manner, is cooled on the side which would otherwise become hottest owing to its being unable to effect useful radiation, by means of the air for combustion on its way to the burner.

B. M. VENABLES.

Furnaces. METROPOLITAN-VICKERS ELECTRICAL CO., LTD. FROM WESTINGHOUSE ELECTRIC & MANUF. CO. (B.P. 300,862, 22.3.28).—In a furnace in which a series of hearths move in a cycle, and at one place are tilted, the travelling motion and tilting motion control each other reciprocally and are interlocked so that only one motion can take place at a time. Separate electric motors may be used with a common controlling switch which can only be on for one motion and off for the other.

B. M. VENABLES.

Furnace. G. F. BEACH, ASSR. TO F. J. RYAN & CO. (U.S.P. 1,690,710, 6.11.28. Appl., 4.6.26).—A furnace, e.g., a tunnel kiln, is provided with a heat-distributing chamber within the furnace walls, which is fitted with upper and lower ports leading into the heating chamber, the upper one being adjustable. B. M. VENABLES.

Regenerative furnace. H. BANGERT and G. HÜHN (U.S.P. 1,691,913, 20.11.28. Appl., 30.12.27. Ger., 20.1.23).—The furnace has two gas flues arranged to

communicate alternatively with the stack or with a source of gas supply and connected with separate regenerator chambers, a burner flue communicating with the furnace, and a flue for the supply of air. All these flues meet at a common mixing point where the gas mixture is ignited, and all but the last are so arranged with respect to one another that the products of combustion are divided into two portions, one of which passes along the burner flue and the other to the flue which is in communication for the time being with the stack. A. R. POWELL.

Furnace for malt or other drying kilns or for open firegrates or basket fires. J. SAUNDERS (B.P. 300,322, 20.8.27).—The air for combustion enters frontally at the sides of the firegrate and passes under side cheeks and a hollow fire bridge, then under and through the firegrate. Constructional forms for domestic fires, drying kilns, boiler furnaces, etc. are shown.

B. M. VENABLES.

Heat exchanger. A. P. BROCKLEBANK, ASSR. TO FOSTER WHEELER CORP. (U.S.P. 1,689,910, 30.10.28. Appl., 5.11.27).—For a heat exchanger of the nest-of-tubes type having a longitudinal baffle, a fluid-tight packing between the edges of the baffle and the shell is described. B. M. VENABLES.

Heat exchanger. C. B. GRADY (U.S.P. 1,690,108, 6.11.28. Appl., 30.10.24).—A hot outer fluid heats a liquid, and the vapour therefrom heats the inner fluid which is passed through the upper portions of a series of containers for the heat-transmitting liquid.

B. M. VENABLES.

Heat-exchange apparatus. G. T. JACOBS, ASSR. TO WHITLOCK COIL PIPE CO. (U.S.P. 1,691,012, 6.11.28. Appl., 10.12.26).—A method of construction of a heat exchanger comprising a nest of tubes with baffles so that the outer fluid flows in a zigzag course transverse to the tubes, is described. The outer shell is contracted tightly upon the flanges of the tube plates and the baffles, which are circular and of the same diameter.

B. M. VENABLES.

Heat-exchanging coil. E. I. HEINSOHN, ASSR. TO GLASCOTE CO. (U.S.P. 1,689,435, 30.10.28. Appl., 26.1.25).—A glass-coated pipe coil has its convolutions kept apart by spacers which are also glass-coated and integrally united with the tube. B. M. VENABLES.

Rotary heat exchanger. F. S. BROADHURST (U.S.P. 1,689,189, 30.10.28. Appl., 30.3.25).—A number of hollow discs are mounted on a rotating hub with inlet and outlet passages and ports corresponding to openings formed in the walls of the discs, the whole forming a tortuous passage for the inner fluid. The outer fluid flows over the outside of the discs and hub between

them and an outer fixed shell, the passage being made tortuous by annular baffles extending inwards from the shell and interleaved with the hollow discs.

B. M. VENABLES.

Apparatus for heat-treating articles. W. L. SCHULENBERG (U.S.P. 1,690,555, 6.11.28. Appl., 15.2.27).—The seal between a movable hearth and the fixed portion of the furnace is formed by intercalating beams; one with a trough-like pocket, the other with a flange dipping into the trough, and a seal of loose, powdered, heat-insulating material is placed therein.

B. M. VENABLES.

Apparatus for heat application. W. A. DARRAH (U.S.P. 1,689,379, 30.10.28. Appl., 29.12.24).—A main body of gases of combustion is kept in circulation by a blower passing over the object to be heated. A smaller portion of fresh hot gases is taken from a combustion device and added to the main stream, and a corresponding quantity of used gases is exhausted through a relief valve. The fuel supply to the combustion device is regulated by a thermostat in the circulating gases.

B. M. VENABLES.

Supplying heat in high-pressure reactions. I. G. FARBENIND. A.-G. (B.P. 275,248, 28.7.27. Ger., 28.7.26).—In processes such as the hydrogenation of oil or coal or the conversion of alcohols such as methyl or ethyl alcohol into higher alcohols by the action of carbon monoxide or water-gas, the heat is supplied wholly or partially by a preheated non-metallic gas which is not decomposed at the temperature of the reaction, but which is not necessarily inert. The gas is brought into direct contact with the materials and is preferably used in large amounts to avoid local overheating. The preheating is effected by means separate from the reaction vessel, and the preheater is protected from access of reaction products. The reacting materials may themselves be preheated, but not up to the temperature of the reaction.

B. M. VENABLES.

Process and apparatus for transferring heat. R. C. NEWHOUSE (U.S.P. 1,689,927, 30.10.28. Appl., 14.12.23).—A fluid is heated by gases of combustion in such a way that, although the incoming fluid is below the dew point of the gases, no condensation is caused. The incoming cold liquid flows through the inner of pairs of concentric tubes, and the emerging hot liquid returns through the annular spaces between the inner and outer tubes. The outer tubes are heated by the gases, and one at least of a pair of tubes is tapered so that during at least one pass the liquid flows with decreasing velocity.

B. M. VENABLES.

Heat-responsive material. P. B. COCHRAN, ASST. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,693,369, 27.11.28. Appl., 21.4.27).—The material consists of a waterproof vehicle, and a composition which includes about 30 pts. of cuprous iodide and 70 pts. of mercury iodide.

H. ROYAL-DAWSON.

Wet grinding of materials in tube or ball mills. F. KRUPP GRUSONWERK A.-G. (B.P. 292,941, 18.4.28. Ger., 27.6.27).—In the type of grinding mill having a discharge chamber between the grinding chamber and the hollow outlet trunnion, the last-mentioned is provided with a double conical deflector which can be

adjusted axially to return more or less of the ground material back to the grinding chamber.

B. M. VENABLES.

Grinding mill. C. E. NEEDHAM, ASST. to BETHLEHEM FOUNDRY & MACHINE CO. (U.S.P. 1,690,125, 6.11.28. Appl., 3.2.25).—A grinding mill of the centrifugal roll type is constructed in such a way that it can be assembled either right or left hand from identical parts.

B. M. VENABLES.

Pulverising mill. L. C. BONNOT, ASST. to BONNOT CO. (U.S.P. 1,690,712, 6.11.28. Appl., 23.5.25).—A rotating drum with balls or other grinding media is fed with material through one end of the drum at the axis. At the other end is a fixed, axial, air-exhaust pipe, cranked upwards within the mill and terminating (commencing) as a perforated dust collector extending substantially right across the mill, through which the dust is withdrawn by suction, the air necessary being admitted to the mill through inlets near the axis at each end of the mill. The middle and lower interior parts of the mill are thus left without obstruction to the grinding material.

B. M. VENABLES.

Pulveriser. T. C. ALFRED (U.S.P. 1,691,951, 20.11.28. Appl., 12.12.25).—In a disintegrator the grizzly bars are held in recesses in the side walls of the machine and are put into compression by a wedge member.

B. M. VENABLES.

Mills. F. E. MARCY (U.S.P. 1,690,493—6, 6.11.28. Appl., [A] 7.6.27, [B, C] 28.12.27, [D] 21.3.28).—The patents relate to discharge grates for a Marcy mill. In (A) the grate is provided with deflectors which move the balls and ore away from the grate at intervals, thus freeing it from oversize material. In (B) the grates are inclined to the vertical and provided with members to free them from oversize. In (C) the discharge circle is made up of rectangular grates alternating with triangular, imperforate deflectors which are triangular in cross-section also. In (D) both the grates and imperforate sections are sector-shaped and grouped round a many-sided pyramid which keeps the grates free of oversize.

B. M. VENABLES.

Mill. C. G. WILLARD, ASST. to MINE & SMELTER SUPPLY CO. (U.S.P. 1,690,447, 6.11.28. Appl., 10.6.27).—In a mill [of the Marcy type] (cf. preceding abstract) deflectors are placed near to both the feed and discharge ends of the drum, to effect lateral displacement of the charge without reducing the length of the grinding chamber.

B. M. VENABLES.

Crusher. H. H. RUMPEL, ASST. to SMITH ENGINEERING WORKS (U.S.P. 1,691,553, 13.11.28. Appl., 20.12.26).—A cone crusher is described in which the axis of the conical head moves in a circle and is always parallel to the axis of the machine.

B. M. VENABLES.

Crusher. J. A. DORMER and J. G. MALONE (U.S.P. 1,692,161, 20.11.28. Appl., 25.6.26).—Between the gyratory crushing head and the fixed part of the crusher are spaced rings which are moved to a certain extent by the movement of the head and multiply the number of crushing zones.

B. M. VENABLES.

Rotary separators. PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (B.P. 300,364,

3.10.27. Addn. to B.P. 278,900; B., 1928, 3).—The secondary part of the separator (for fine dust) consists of a fan in front of an outlet comprising one or more perforated discs or cones; the fan rotates in such a direction that it produces a pressure against the flow of air or gas.

B. M. VENABLES.

Centrifugal drying machines. A. K. CROAD. From COWLES ENGINEERING CORP. (B.P. 300,874, 12.4.28).—A centrifuge is rendered safe against the dangers of uneven loading by providing a heavy weight fixed on the axis within the basket, and by supporting the whole of the rotating part on a flexible diaphragm in such a manner that severe distortion of the diaphragm will apply a brake by causing suitably lined rotating parts to rub against stationary parts.

B. M. VENABLES.

Rotary drying apparatus. J. B. VERNAY (B.P. 294,953, 30.12.27. Fr., 2.8.27. Addn. to B.P. 262,464; B., 1927, 719. Cf. B.P. 282,432; B., 1928, 430).—The central driving shaft is omitted and the apparatus supported on rollers and driven by gearing. In place of the shaft is a heated, fixed, central cylinder, through, then over, which the material is pushed. Means are described for preventing clogging in the feed hopper and for control of inlet and outlet of material.

B. M. VENABLES.

Washing and drying machines. SIEMENS-SCHUCKERTWERKE A.-G., and W. WEIMERS (B.P. 300,048, 24.1.28).—The apparatus comprises a basket mounted on a spindle which when vertical rotates at centrifugal speed, and when inclined at an angle less than 90° to the vertical rotates at a much slower speed partly in the washing liquid.

B. M. VENABLES.

Apparatus for the [dry] separation of solid substances of different sp. gr. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,936, 20.8.27).—The powdered material, which has already been screened into portions of uniform particle size, is fed through a side inlet to a vertical receptacle, where it is kept just in suspension by a current of air coming up through a travelling perforated belt or screen which forms the bottom of the shaft, and draws off the heavier material, while the lighter material is caused to overflow through a side outlet opposite to and higher than the inlet.

B. M. VENABLES.

Centrifugal clarifying separator. NAT. ACME Co., Assees. of D. S. PATERSON and G. B. PETSCH (B.P. 294,524, 9.5.28. U.S., 25.7.27).—The bowl comprises a number of annular compartments; in the outermost one the muddy portion flows once the full axial length of the machine; the partly clarified liquid, after traversing nearly the full length of the outer compartment, flows inwards to the second compartment, where it flows axially back again, spreading over the exterior of a filter cylinder and passing inwards through it. Any mud collected on the filter is flung off.

B. M. VENABLES.

Centrifugal extractor. C. A. KELSEY, ASSR. to GEN. ELECTRIC Co. (U.S.P. 1,689,490, 30.10.28. Appl., 10.12.27).—A centrifugal extractor having a perforated basket is provided with a continuous feed and discharge device comprising a cylindrical member within and rotating with the basket, but capable of vertical reciprocation relative to the basket.

In the annular space between the cylinder and the basket are a number of flaps hinged to the cylinder and pressed outwards against the basket on the down stroke, but inclining away from it on the up stroke. There are several "storeys" of the flaps, and the feed is through the wall of the inner cylinder between two of the upper storeys, and the discharge for separated solid material is through large apertures in the outer basket below the lowest story of the scraping flaps, the liquor being discharged through the wall of the basket before the solid matter is pushed so far down.

B. M. VENABLES.

Centrifugal separators. AKTIEBOLAGET SEPARATOR (B.P. 292,127, 12.6.28. Swed., 14.6.27).—Means for discharging the heavier liquid are described.

B. M. VENABLES.

Separation apparatus and process. A. R. SILVERSTON, ASSR. to COKE RECLAMATION CORP. (U.S.P. 1,689,536, 30.10.28. Appl., 15.11.24).—Liquid is caused to flow in a substantially horizontal direction between guide vanes which divide it into sections, the vanes being provided with adjustable flaps at the ends to cause the sectional streams of liquid to turn more or less into the vertical direction. The material is fed adjacent to the ends of the vanes and the heavier particles are collected and withdrawn by a transverse conveyor or elevator and the lighter particles by a longitudinal one.

B. M. VENABLES.

Regulating the [consistency of pulps by the] concentration of liquids. ZELLSTOF-FABR. WALDHOF, and R. HAAS (B.P. 280,876, 21.10.27. Ger., 20.11.26).—A paddle is rotated in the pulp by means of an electrical motor in which the current will vary if the viscosity varies, and the change of current may be made to regulate the flow of a diluting liquor. Another method is to have a constant-speed shaft driving the paddle shaft in the reverse direction through a chain of 3 bevel wheels; the axis of the middle wheels (duplicated for convenience) can turn about the axis of the other shafts and is spring-controlled so as to deflect more or less according to the torque.

B. M. VENABLES.

Deposition of [tapering] cakes of solid matter from fluids in which the solids are suspended. FILTRATION ENGINEERS, INC., Assees. of F. W. YOUNG (B.P. 280,170, 9.8.27. U.S., 4.11.26).—Filter cakes are made in non-uniform thickness by obstructing the flow of filtrate in the drainage member behind the filter medium at those places where the cakes are required to be thinner. One application is the manufacture of tapering shingles from asbestos cement slurry.

B. M. VENABLES.

Filter. C. ST. C. BURNS, ASSR. to F. KAY (U.S.P. 1,691,867, 13.11.28. Appl., 23.1.26. Renewed, 28.9.28).—The pulp is admitted to a pressure-filtering chamber through the walls of which the filtrate escapes, and from the walls the cake is scraped and drops into a worm-conveyor which presses it through a discharge valve. Both sides of the valve are subject to chamber pressure, one side through the pulp in the conveyor, the other through a direct pipe; the latter pressure tends to close the valve, which action is supplemented by a spring.

B. M. VENABLES.

Filters for liquids. A. GRØNNING (B.P. 300,600, 16.8.27).—The filter comprises a pile of spaced discs, with an axial inlet and peripheral outlet or *vice versa*. On the face of each disc is formed a zig-zag ridge extending from the central opening to near the periphery, and the narrow space between the ridge and the next disc is the filtering passage. B. M. VENABLES.

Detection of suspended matter in fluids and operation of means for indicating its presence. W. KIDDE & Co., Inc., Assees. of W. H. FREYGANG (B.P. 272,914, 14.6.27. U.S., 17.6.26).—The particles of suspended matter reflect light into a photo-electric cell which is otherwise not illuminated. B. M. VENABLES.

Mixing of liquids. F. E. SMITH, A. P. H. DESBOROUGH, W. T. THOMSON, H. F. REYNOLDS, and E. W. BLAIR (B.P. 299,942, 26.8.27).—One of the liquids is injected tangentially into the wider portion of a cylindrical shell having two different diameters, and forms a film which eventually issues at the narrow end as a cylindrical sheet. The other liquid is sprayed by any convenient means within the cylindrical film, *i.e.*, either within the narrow portion of the shell or at the outlet. In the case of viscous liquids the shell may be rotated. B. M. VENABLES.

Apparatus for mixing liquids. H. J. LLOYD (B.P. 300,341, 7.9.27).—The apparatus is suitable for adding a small quantity of one liquid to another liquid flowing under pressure. The small quantity of liquid is contained in a closed vessel to the top and bottom of which there lead two passages from a Venturi tube in the main flow of liquid—one from a mouth, the other, which is also the outlet for the liquid being added, from the throat of the Venturi. The flow of the small quantity of liquid is controlled by needle valves. B. M. VENABLES.

Fractionating tower. Bubble still trays for fractionating towers. POWER SPECIALTY Co. (B.P. 270,720 and 299,479, 3.5.27. U.S., 5.5.26).—The trays are constructed to give uniform distribution of liquid even though of a large diameter, and in the tower provision is made for drawing off all or part of the liquid falling from any tray. Reboiling may be effected in different sections, and entrainment is minimised. B. M. VENABLES.

Apparatus for filtering and bottling fluids under pressure. G. STRIFFLER (U.S.P. 1,692,393, 20.11.28. Appl., 20.1.28).—A cylindrical shell is provided with, in order downwards, an air-tight detachable lid, a filter diaphragm, a false bottom, a real bottom. Compressed air can be supplied to the highest and lowest compartments, and there is a communication from above to below the false bottom. B. M. VENABLES.

Centrifugal disintegrator for liquids. W. S. BOWEN (U.S.P. 1,692,617, 20.11.28. Appl., 19.7.28).—A rotating disc has a collar-like liquid container on its top surface. Openings are formed in the collar just above the disc and in the disc beyond the collar. B. M. VENABLES.

Apparatus for filtering gases. GEN. AIR FILTERS CORP. (B.P. 288,992, 8.11.27. U.S., 18.4.27).—In the type of apparatus where the dust is caught in a viscous film spread upon baffle plates, the retention of liquid

up the baffle plates is improved by the provision of slits and liquid spreading devices. One suitable form of baffle is a sheet of expanded metal slit but not stretched. B. M. VENABLES.

Apparatus for cleansing and cooling gases. O. HUBMANN, Asst. to METALLBANK & METALLURGISCHE GES. A.-G. (U.S.P. 1,690,635, 6.11.28. Appl., 8.11.26. Ger., 8.8.25).—The gases and a washing liquid are caused to pass simultaneously through a number of converging-diverging narrow passageways formed in the wall of a cylinder rotating within a casing. B. M. VENABLES.

Air and gas cleaner. C. S. HANSARD and A. E. NETZEL (U.S.P. 1,691,971, 20.11.28. Appl., 10.10.27).—The inner of two concentric shells has a closed top on which water is sprayed, thus producing a shower in the annular space between the two shells, through which the gas passes. The lower part of the inner shell is splayed out nearly to touch the outer, and a water reservoir is formed below that point. B. M. VENABLES.

Apparatus for purifying steam, vapours, and gases centrifugally. W. ALEXANDER (B.P. 300,841, 25.2.28).—Forms of apparatus having no moving parts for cleaning steam etc. are described. B. M. VENABLES.

Apparatus for purifying and moistening air. A. SCHÜCKNER (U.S.P. 1,691,827, 13.11.28. Appl., 15.2.24. Austr., 19.2.23).—A fan is provided with a hollow shaft through which liquid is supplied to the interior of a drum attached to the shaft and having porous walls to spray the liquid. B. M. VENABLES.

Treating sprayed materials with gases. INDUSTRIAL SPRAY-DRYING CORP., Assees. of B. F. UHL (B.P. 296,421, 19.11.27. U.S., 1.9.27).—A spray dryer is constructed so that the drying gas on entry is split up among a number of parallel passages the aggregate cross-section of which is greater than of the entry duct, but the passages themselves are of considerable length and free from abrupt changes of direction or cross-section. B. M. VENABLES.

Determining the contents of a gas. H. SCHMICK, Asst. to SIEMENS & HALSKE A.-G. (U.S.P. 1,691,138, 13.11.28. Appl., 3.6.26. Ger., 4.6.25).—In the method of determining the composition of a gas by radiation measurements, a selective radiator is used such that its emission bands overlap as little as possible the absorption bands of the test gas. A. R. POWELL.

Impregnating brake bands, brake coverings, etc. W. OTTMANN (B.P. 279,496, 21.10.27. Ger., 21.10.26).—Silicic acid is precipitated in and/or on a band of fabric, *e.g.*, asbestos with or without cotton and brass. The precipitation may be effected by alternate treatment with water-glass and acids or ammonium salts, or by the interaction of silicon tetrachloride and water or water-glass. B. M. VENABLES.

Heat-treating furnace. A. BESTA (U.S.P. 1,695,224, 11.12.28. Appl., 26.3.26. Ger., 28.2.25).—See B.P. 248,394; B., 1927, 554.

Apparatus for separating solids [coal] from liquids. R. F. KRALL, Asst. to WOODALL-DUCKHAM (1920), LTD. (U.S.P. 1,695,193, 11.12.28. Appl., 24.4.26. U.K., 28.8.25).—See B.P. 261,850; B., 1927, 64.

[Fuel supply control for] furnaces. J. GORDON & Co., LTD. (B.P. 283,522, 3.1.26. U.S., 12.1.27).

Refrigerating plants. A./S. AMUNDSEN REFRIGERATOR Co. (B.P. 291,039, 24.10.27. U.S., 29.10.26).

Indicating [by audible signals] the exhaustion of gas-purifying substances [in respirators]. R. H. and R. W. G. DAVIS and C. G. ROSLING (B.P. 301,532, 31.8.27).

II.—FUEL; GAS; TAR; MINERAL OILS.

Hydrogenation and liquefaction of coal. V. Influence of composition, pressure, temperature, and catalysts on hydrogenation. D. G. SKINNER and J. I. GRAHAM (Fuel, 1928, 7, 543—555; cf. B., 1927, 242).—About 30 different coals have been hydrogenated at 430° and under approx. 150 atm. pressure; a rotating autoclave was used, each charge consisting of 200 g. of coal and 100 g. of phenol. The yields of (A) phenol-insoluble residue, (B) phenol-soluble, chloroform-insoluble powder, (C) phenol-soluble, chloroform-soluble oil, (D) light spirit boiling below 150°, (E) water, and (F) gas have been tabulated, together with the yields of products from the carbonisation of the coals at 450° in the Gray-King assay. The para-bituminous coals (Seyler's classification) appeared to be the most amenable to hydrogenation, although exceptions to this generalisation were observed. The addition of ferric and nickel oxides increased the oil yields appreciably, but the most effective catalyst for this purpose was ammonium molybdate. The results of the hydrogenation of Warwick slate vitrain and of the residue after extracting the coal with phenol at 185° indicate that the extract is not converted into oil in the process, but appears in the phenol-insoluble residue A. On the other hand the pyridine-soluble constituents of a Staffordshire coal appeared to be completely converted into oil. Fine grinding of the coal increases its tendency to coke during the process. The residue A is richer in carbon, and generally, but not always, poorer in hydrogen than the original coal. On carbonising this residue at 600° usually only a powdered coke was produced; the products from Warwick slate vitrain and from Graigola coal, however, produced coherent cokes, the latter giving a more strongly swollen coke than the original coal. The powder B is definitely a hydrogenation product; its yield varied from 0.6 to 33% with different coals. On carbonisation it produced a greatly swollen and fragile coke. The oil C contained 86—87% C, about 7.5% H, and about 4% O. The iodine value of the fraction below 300° varied from 94 to 358, whilst that of the light oil D lay between 120 and 150.

A. B. MANNING.

Propagation of a zone of combustion in coal. V. Effect of temperature. Temperature of spontaneous propagation. S. H. JENKINS and F. S. SINNATT (Fuel, 1928, 7, 556—562; cf. B., 1927, 802).—The rate of propagation of a zone of combustion through a train of coal dust varies with the degree of fineness of the dust; the smaller the particle size of the coal below a certain limit the more rapidly is combustion propagated. No combustion at all occurs with particles above the limiting size. Other conditions being the

same, the rate of propagation increases with a rise in the original temperature of the coal. The temperature at which spontaneous propagation takes place was investigated by placing a cone of powdered coal in an electrically heated furnace maintained at a constant temperature, and recording the rise of temperature in the interior and at the surface of the cone by means of thermocouples. Below a certain temperature, in the neighbourhood of 160—170° for the coals investigated, no combustion occurred; above that temperature spontaneous combustion took place.

A. B. MANNING.

Heating value of coal in nickel-lined bombs. A. E. STOPPEL and E. P. HARDING (Ind. Eng. Chem., 1928, 20, 1214—1218).—When the cal. value of a coal is determined by combustion under pressure corrections are necessary for the heats of formation of nitric and sulphuric acids, and when the material of the bomb is attacked by these acids further adjustments must be made. The amount of nickel removed from a nickel-lined bomb is measured by boiling the bomb-washings to remove carbon dioxide, titrating the solution with 0.1N-sodium hydroxide and methyl-red indicator, and then continuing the titration with phenolphthalein until the colour persists on boiling; this method has been checked satisfactorily against gravimetric determinations. The heat of formation and solution for $\text{NiO-N}_2\text{O}_5\text{-Aq}$ is 54,500 g.-cal., or 2.73 g.-cal. per c.c. of 0.1N-nickel nitrate. A series of experiments carried out with sucrose in a nickel-lined bomb, with and without appreciable amounts of nitrogen, showed good agreement with these figures on the assumption that the surface contained both metallic nickel and its oxide. A correction of this kind must be made in the water equivalent of the calorimeter unless all nitrogen is excluded. When coal is burned, a further allowance is necessary for sulphate formation; the total heat of formation of $\text{Ni-O}_2\text{-SO}_2\text{-Aq}$ is 157,350 g.-cal., or 7.868 g.-cal. per c.c. of 0.1N-nickel sulphate, and is equivalent to 14 g.-cal. per cg. of sulphur. Results in an ilium bomb are compared with those in one with a nickel lining for coals varying in sulphur content from 0.45 to 8.82%, and the true cal. value agrees well in the two series of tests.

R. H. GRIFFITH.

Hydrogen-volatile matter ratio in American coals and its use in producer-gas calculations. W. J. HUFF (Ind. Eng. Chem., 1928, 20, 1371—1372).—A relationship between the hydrogen content of a coal and the percentage of volatile matter in it, previously found by Fieldner and Selvig (U.S. Bur. Mines Tech. Paper No. 197, 1918) for 2000 American coals with calorific values between 12,000 and 14,500 B.Th.U./lb., has been expressed in a more convenient mathematical form, and illustrations of its application are given.

R. H. GRIFFITH.

Average quantitative composition of Ruhr coal ash. D. J. W. KREULEN (Brennstoff-Chem., 1928, 9, 399).—An average sample, containing the united ash from some 4000 samples of Ruhr coals, was made up of 4.8% soluble in water (mainly sodium, magnesium, and calcium sulphate), 41.0% soluble in hydrochloric acid (mainly ferric oxide and alumina), and 54.2%

insoluble in hydrochloric acid (mainly silica and alumina). The average sample contained 41.5% SiO_2 , 18.4% Fe_2O_3 , and 31.5% Al_2O_3 . W. T. K. BRAUNHOLTZ.

Characteristics of various kinds of coal, and the nomenclature of brown coals. W. GOTHAN, K. PIETSCH, and W. PETRASCHKE (Braunkohle, 1927, 26, 669—674; Chem. Zentr., 1928, ii, 409—410).—Characteristics useful in the differentiation of peat, brown coal, and hard coal are enumerated. Chemical methods are not serviceable; peat, however, is characterised by the greater cellulose content. A nomenclature and a description of brown coals are included. A. A. ELDRIDGE.

Chemistry of the naturally occurring humic acids.

B. G. SIMKE (Brennstoff-Chem., 1928, 9, 381—385).—The humic acids were extracted from lignite with alkali, and the alkaline solution was exposed to the air for several weeks to allow complete oxidation of oxidisable "proto"-acids; the acids were precipitated with dilute hydrochloric acid, purified by electro-osmosis, and dried. The final product contained (on the dry and ash-free basis) 63.63% C, 5.14% H, and 1.35% N. It was separated by means of solvents into the following constituents: resinous acids (soluble in alcohol and ether) 9.57%, acids (soluble in alcohol, insoluble in ether) 6.09%, acids (insoluble in alcohol, soluble in pyridine) 15.23%, residue (insoluble in alcohol and pyridine) 69.11%. The resinous acids contained 2.13% OMe, which is sufficient to account completely for the methoxyl content of the original product. The separated fractions were treated with phenylhydrazine (to determine their content of carbonyl oxygen), and with methylating agents; fairly well-defined products were obtained by the action of halogens and of nitric acid. The supposed nitro-compounds obtained with nitric acid were reduced to amines, diazotised, and coupled to give azo-dyes; the humic acids themselves could also be coupled with diazonium salts. The various humic acid derivatives dye animal and vegetable fibres yellow and brown shades which are extremely fast to light. The above observations lend support to Fischer's conception of the aromatic nature of humic acids. W. T. K. BRAUNHOLTZ.

Water-gas equilibrium and the temperature of the flame. Y. KAUKO (Förh. Ill nord. kemistmötet, 1928, 167—168).—A method for investigating the above equilibrium in flames without the necessity for taking samples of the gases has been devised. The composition of the fuel being known, the amounts of hydrogen, water, and nitrogen are functions of the percentages of carbon monoxide, carbon dioxide, and oxygen in the flame gases, the two former themselves depending on the oxygen content. The amount of heat available for the combustion and the temperature of the flame can also be calculated from a knowledge of the carbon monoxide and dioxide contents. Assuming that water-gas equilibrium results in the flame, a relationship can be found between the variables temperature, carbon monoxide, carbon dioxide, hydrogen, and water vapour, and, by elimination of the two last-mentioned, two equations involving carbon monoxide, carbon dioxide, and temperature can be derived, hence the composition of the flame gases can be determined simply from temperature measurements. Since the contents of carbon monoxide

and dioxide are dependent on the amount of air admitted, there is a dependence also between this factor and the temperature of the flame. Employing the above method the author finds that the water-gas equilibrium in an alcohol flame takes place at 1300°. H. F. HARWOOD.

Flame movement in gaseous explosive mixtures.

O. C. DE C. ELLIS (Fuel, 1928, 7, 195—205, 245—252, 300—304, 336—344, 408—415, 449—454, 502—508, 526—534; cf. B., 1927, 354).—The propagation of a flame through explosive mixtures, principally of carbon monoxide and air or oxygen, has been studied by a photographic method. The movement of the flame surface in vessels of different shape, when the mixture is ignited either centrally or excentrically, is illustrated by numerous plates. A. B. MANNING.

Synthesis of higher hydrocarbons from water-gas [at atmospheric pressure]. II.

D. F. SMITH, C. O. HAWK, and D. A. REYNOLDS (Ind. Eng. Chem., 1928, 20, 1341—1348; cf. B., 1928, 434).—A catalyst containing cobalt, manganese, and copper carefully reduced in hydrogen under such conditions as prevented local overheating was used. The water-gas used was freed from oxygen, hydrogen sulphide, carbon dioxide, and water vapour, and finally from all sulphur compounds and heavy gases through a trap cooled in liquid air. A mixture of the fused nitrates of sodium, potassium, and lithium was used as the heating liquid in the thermostat containing the reaction tube in which the catalyst was placed. Since the reactions evolve considerable quantities of heat, the thermostat temperatures only approximate to those of the catalyst mass. The reaction tube was designed to reduce as far as possible this probable difference in temperature. Experiments were made at temperatures between 203° and 287° at space velocities from 120 to 260. At 203° 18% of the gas is converted in a single passage at a space velocity of 230. It was noticed that the activity of the catalyst fell very rapidly (as judged by the amount of gas converted) as the volume of gas passed over increased, to reach a fairly steady value. Passage of hydrogen over the catalyst reactivated it. Most of the oxygen appears in the product as water, and the ratio $\text{H}_2\text{O}/\text{CO}_2$ increases rapidly with decreasing temperature. At the higher space velocities and at the higher temperatures relatively more unsaturated hydrocarbons are formed; with lower velocities and at the higher temperatures, in general, more heavy hydrocarbons are formed. The total hydrocarbon products contain, according to conditions, from 20% by wt. upwards of methane. In one experiment at 260° with a space velocity of 260 the composition of the hydrocarbon products was: methane 21%, "gasol" 45%, motor fuel 34%. The yields of hydrocarbons other than methane vary from 92 to 156 g./m.³ of hydrogen and carbon monoxide converted. Some degree of control can be exercised over the nature of the product by suitable choice of the variable conditions.

H. INGLESON.

Synthesis of methane from carbon dioxide and hydrogen. M. RANDALL and F. W. GERARD (Ind. Eng. Chem., 1928, 20, 1335—1340).—The equilibrium $\text{CO}_2 + 4\text{H}_2 = \text{CH}_4 + 2\text{H}_2\text{O}$ (gas) has been studied both

in order to determine the equilibrium constants and examine the efficiencies of two catalysts for the reaction. The catalysts employed were nickel supported on porous brick prepared by reduction of the nitrate, and reduced nickel activated by ceria. Both forward and reverse reactions were carried out by the streaming method at velocities varying from 6.9 to 12.4 c.c. of exit gas (N.T.P., water vapour considered uncondensed) per hr. per c.c. of apparent volume of catalyst. The temperatures at which the forward reaction was carried out varied from 521.8° Abs. to 665.6° Abs. The equilibrium constants calculated from these experiments according to the equation— R (in ° Abs.) = $-4.5787 \log[p(\text{CH}_4) \cdot p^2(\text{H}_2\text{O})/p(\text{CO}_2) \cdot p^4(\text{H}_2)]$, where p = molar fraction of the gas \times total pressure in atm., are in good accord; e.g., R for forward reaction at 648.1° Abs. = -16.51 , whilst for the reverse change at 650.1° Abs. = -16.44 . At temperatures below 595° Abs. the flow rates were too rapid to admit of equilibrium being established. In the experiments of Hightower and White (B., 1928, 147) equilibrium would appear not to have been attained. The results obtained by Pease and Chesbro (A., 1928, 707) are in good accord with those of the present authors, which do not agree with those of Mayer and Henseling (A., 1909, i, 753) and of Neumann and Jacob (A., 1925, ii, 532). The free energy of methane calculated from these experiments, $-11,582$ g.-cal., is in agreement with the value $-11,617$ g.-cal. found from the direct synthesis from graphite and hydrogen carried out by Mayer and Altmayer (A., 1907, i, 457). The reasons for the deposition of carbon in the catalyst are discussed, and it is concluded that the reaction taking place between carbon dioxide and methane is a possible cause, although the mechanism is too complex for this to be proved by the experiments. A gas-sampling device and an automatic pressure-regulating valve are described in detail.

H. INGLESON.

Ignition of mixtures of air with natural gas and with methane by induction coil sparks. E. G. MEYER (Ind. Eng. Chem., 1928, 20, 1353—1354).—Natural gas, which contains varying amounts of methane, has ignition points in air somewhat different from that of the pure substance; a comparison of the two gases has been carried out under standard conditions with an electric spark as the firing mechanism. The natural gas contained 88.9% of methane, 7.4% of ethane, 2.4% of propane, 0.8% of butane, 0.1% of higher hydrocarbons, and 0.4% of nitrogen. The apparatus and method used were similar to those of Sastry (B., 1916, 682) and of Wheeler (B., 1920, 592 A), in which a variable spark gap was employed. The results showed that the most easily ignited mixtures of natural gas with air can be fired slightly more easily than any methane-air mixture, but the differences are never large. The optimum mixture contains 7.3—8.6% of combustible gas.

R. H. GRIFFITH.

Radiation of heat from gas-lighting burners. H. J. HODSMAN (Gas J., 1928, 184, 794—796).—The energy represented by the light itself is a very small fraction of that supplied to ordinary illuminating agents. With electric light more than 95% of the

energy is dissipated as heat, and with a gas-lighting burner more than 99%. The distribution of the energy of the fuel, using several types of gas-lighting burners, was examined. The proportion of heat radiated downwards was determined calorimetrically and found to be for a batwing burner 7.1%, of the cal. value of gas consumed; upright incandescence burner, without chimney, 19.7%; inverted incandescence burner, without globe, 25.9%; with globe 16.2%. These amounts could be increased by placing a reflector above the burner and by lining the top of the burner with refractory material, when the proportion of heat radiated reached 37%.

C. B. MARSON.

Catalytic processes for utilisation of coal-tar crudes. A. O. JAEGER (Ind. Eng. Chem., 1928, 20, 1330—1335).—Difficulties are experienced in the economic working up of certain coal-tar crudes for anthracene by present methods, particularly when the content of this hydrocarbon is as low as 14%. When the tars are derived from cannel or other highly paraffinoid coals, the paraffins cannot be removed satisfactorily by any method used hitherto. In the process described the crude anthracene is vaporised and mixed with air, preferably by spraying it into heated air. The mixture is passed over a suitable heated catalyst and the impurities are oxidised without any considerable loss of anthracene. It has been found that almost any selective oxidation catalyst can be employed, but that it is preferable to "stabilise" the catalyst with compounds of alkali or alkaline-earth metals, or of metals of which the oxides are reduced with difficulty. E.g., a catalyst prepared from ferric oxide stabilised by potassium nitrate and supported on pumice when employed at 400—440° permits of the preparation of 94—96% anthracene from a cannel coal-tar crude containing 25—30% of the hydrocarbon. Other suitable catalysts described include oxides of the fifth, sixth, and eighth groups stabilised by potassium compounds. If it is desired to prepare anthraquinone direct from the crude, after this preliminary removal of most of the impurities, the anthracene vapour is passed together with air through a contact mass suited for its oxidation. Such catalysts are zeolites containing vanadium tetroxide as one of the amphoteric oxides. A new solvent process distinct from the catalytic process described permits of the recovery of the carbazole and phenanthrene present in the crude anthracene. By one crystallisation of crude anthracene from furfuraldehyde, the anthracene content may be raised from, say, 31 to 86%, and at the same time the carbazole content reduced from 16 to about 6%. The anthracene recovery is about 96%, and such a product is suitable for catalytic oxidation to anthraquinone of a high purity. Carbazole and phenanthrene are recoverable from the mother-liquor, and are not lost as is the case in the catalytic oxidation.

H. INGLESON.

Critical temperatures and oil cracking. R. H. MCKEE and H. H. PARKER (Ind. Eng. Chem., 1928, 20, 1169—1172).—The temperature above which an oil can no longer exist as a liquid is of interest in connexion with cracking processes, and the critical temperatures for a large number of petroleum fractions, such as gas

oils or gasolines, have been determined by heating in tubes of hard glass until the meniscus was not visible. Precautions are necessary on account of variation of the ratio of liquid to total volume, of the time of heating, and of the possibility of cracking; the overall accuracy is within 10° limits. A general formula relating critical temperature to the average b.p., as measured by the A.S.T.M. method, is $t_c = 1.05t_b + 160$, where t_c and t_b are the critical and the average boiling temperatures, except in the case of aromatic compounds for which the relation becomes $t_c = t_b + 208$. When an oil contained asphaltic substances in solution, no precipitation occurred if it was heated above the critical temperature. It is shown that many so-called liquid-phase cracking processes really take place with the hydrocarbons present entirely as vapour.

R. H. GRIFFITH.

Fundamental factors of liquid-phase cracking.

A. N. SACHANEN (SACHANOV) and M. D. TILITSHEYEV (J. Inst. Petroleum Tech., 1928, 14, 761—767; cf. B., 1927, 576).—The following fractions from crude oils, chiefly from Grosny, were cracked for 64 min. at 425° in an autoclave equipped as a pressure still: gasoline or naphtha, b.p. 180—220°, kerosene 220—270°, gas oil 270—300°, solar oil 300—325°, engine oil 250—280°/6 mm., cracked kerosene 220—270°, and cracked gas oil 270—300°. The final pressures registered were 100, 85, 70, 72, 72, 85, and 61 atm., respectively. The products were, usually, gas, gasoline, kerosene, gas oil, and heavy residue. Comparing fractions of similar b.p. from the starting materials and from the cracked products, those from the latter are, in general, heavier. Thus cracking produces light products of decomposition, but also products of condensation with high sp. gr. At constant temperature the velocity of cracking gradually diminishes owing to the diminishing concentration of the original oil and of the newly-formed heavier fractions. In the initial stages the gasoline formed is nearly proportional to the time; hence cracking commences immediately the necessary temperature is reached, there being no "induction period." The velocity of cracking is greater for heavier fractions, and greater for straight-run oils than for cracked oils of similar b.p. The velocity of cracking is doubled for every 10° rise in temperature, but for liquid-phase cracking 450—475° is apparently an optimum above which rapid formation of coke and gas occurs. With constant temperature and time, velocity of cracking is independent of pressure. The rise in sp. gr. of similar fractions on cracking is not always accompanied by the formation of a fraction of high b.p. The latter is the material from which coke is formed. Thus coke is not produced during the earlier stages when a distillate is cracked, and is formed with increasing velocity as the duration of cracking increases. Distillates of the same sp. gr. give less coke when cracked than residues or heavy crude oils. Since the velocities of formation of gasoline and of coke respectively decrease and increase with increasing duration of cracking, it is more economical to stop when coke first appears, and to recycle those fractions which do not, primarily, give coke. The once-cracked material yields gasoline at a lower rate, however, and this becomes very apparent if cracking is repeated a third or fourth time. Moreover, with each repetition the gas formation increases. Hence

there is a limit beyond which repeated cracking is uneconomical.

W. S. NORRIS.

Cracking of Rangoon paraffin wax with aluminium chloride at temperatures below 330°.

H. I. WATERMAN and L. L. W. VAN SOEST (J. Inst. Petroleum Tech., 1928, 14, 756—760).—Rangoon paraffin wax (mol. wt., Rast, 414; setting point, Shukoff, 57.7°) with about 10% of aluminium chloride is gradually heated in an atmosphere of carbon dioxide under slightly reduced pressure until gasoline is evolved. The mixture is maintained at this temperature (about 180°) until the reaction slackens markedly, when more heat is gradually applied until finally, at about 300°, only paraffin wax distils. The gas evolved corresponds in average composition with butane; there are traces of gaseous olefines, but hydrogen is not found in practically important quantity. The residual wax, about 50—62% of the original, has setting point (Shukoff) 57.1—59.5°, mol. wt. (Rast) 362—434; the wax distillate has setting point 39.7—50.0°, mol. wt. 249—300. Both the residual and the distillate wax are fully saturated. The yield of gasoline is 18—23% on the original wax or 35—54% on the wax used up; it has b.p. 25—220°, d_{20}^{20} 0.65—0.68, n_D^{20} 1.381—1.397, and bromine value (McIlhenny) practically zero (max., 2). These results agree with those of Pictet and Lerczynska (cf. B., 1916, 1098), using Galician paraffin wax and 10% of aluminium chloride. The latter appears to act in two ways. Primarily, cracking is brought about at a lower temperature, and is therefore less intensive than when wax is cracked alone. Secondly, the unsaturated products are attacked by the aluminium chloride with the formation of saturated products.

W. S. NORRIS.

Desulphurising action of silica gel. III. H. I. WATERMAN and M. J. VAN TUSSENBROEK (Brennstoff-Chem., 1928, 9, 397—398. Cf. B., 1927, 179; 1928, 323).—Solutions of diphenyl sulphide, dibenzyl sulphide, benzyl mercaptan, and sulphur (true and colloidal solutions) in kerosene were shaken with silica gel of different degrees of fineness. Sulphur is not taken up by silica gel, but the three aromatic sulphur compounds are adsorbed to quite a considerable extent, as is also thiophen. When the kerosene alone was shaken with silica gel its aniline point was raised 1.1—1.2°, indicating that the aromatic constituents are selectively adsorbed.

W. T. K. BRAUNHOLTZ.

Analysis of cracked spirits. Determination of aromatic, olefine, naphthene, and paraffin hydrocarbons. F. H. GARNER (J. Inst. Petroleum Tech., 1928, 14, 695—721).—The following determinations are made: (a) The iodine value (by Hanus' method; cf. Faragher, Gruse, and Garner, B., 1922, 90 A) of the petrol is found. (b) Olefines and aromatic hydrocarbons are removed simultaneously by agitation with 2½ vols. of fuming nitric acid at from —10° to —5°. The percentage loss in vol. and the aniline-point difference before and after treatment are determined. The aniline point of the remaining spirit gives the % of paraffins and of naphthenes by reference to one of five curves, according as the mid-point of the petrol (temperature at which 50% is volatile) is 95°, 105°, 115°, 125°, or 135°, these curves being based on the figures for representative

pure hydrocarbons. If the iodine value of the original petrol is above 25, the aromatic hydrocarbon equivalent of the hydrocarbons (aromatic and olefinic) removed by the acid is found by reference to the curve of Tizard and Marshall (J.S.C.I., 1921, 40, 20 r). The difference between this equivalent and the percentage loss in volume is multiplied by 5/3, giving the olefine content of the original petrol. The difference between the percentage loss in volume and the olefine content is the % of aromatic hydrocarbons in the petrol. If the iodine value of the original gasoline is below 25, the olefine content is estimated by dividing the iodine value by 2.8 for aviation spirit (mid-point 95°), 2.7 for No. 1 spirit (mid-point 105°), and 2.6 for all other spirits. The aromatic hydrocarbon equivalent is determined as before from the difference in aniline points; from this figure is subtracted 40% of the olefine content, giving the % of aromatic hydrocarbons in the gasoline. Using this second method it is not necessary to determine the loss in volume on acid treatment. W. S. NORRIS.

Evaluation of the stability of naphtha products as regards coke formation. N. I. TSCHERNOSHUKOV (Izvestia Thermo-Tech. Inst. [Moscow], 1928, 6, 7—11).—The principal cause of the formation of coke from petroleum products under normal working conditions in engines is the oxidising polymerisation of the components of the oil. The formation of precipitates (type I) of asphaltic nature (asphaltenes, carboids) proceeds mainly at the expense of the resinous matters. Oxidation of the purely hydrocarbon constituents results in precipitates of type II, composed of acids of high mol. wt. with acid values between 60 and 110. There is, moreover, a possibility that resinous and asphaltic substances may arise, firstly, from such acids by loss of carbon dioxide and subsequent condensation of the unsaturated hydrocarbons thus formed, and, secondly, by condensation of aldehydes and other intermediate compounds formed during the oxidation of the hydrocarbons of the oil. Experiments were made in the standard Konradson apparatus with (1) a vaseline oil from Balachani petroleum, purified until it was colourless and separated almost instantaneously from water, with additions of asphaltenes, resins, and sulphonic acids, and (2) a number of other oils. The results show that the coke is formed mostly from the asphaltenes, and that asphaltenes, resins, or sulphonic acids do not cause formation of coke from the oil with which they are mixed. Since, however, in this test access of air to the heated oil is prevented, the conditions are different from those obtaining in the cylinders of either internal-combustion or steam engines. Results are given of experiments in which various oils were heated at 200° or 250° in air at 15 atm. pressures for 3 hrs., the resulting products being diluted with light petroleum (b.p. 65°) and the weight of the resulting precipitate determined; the Konradson test was also applied to these oils, and the "Akzys" resins were determined. No parallelism is observed between the quantities of precipitate formed on oxidation and the figures given by the Konradson test, and the proportions of "Akzys" resins do not reflect the tendency of the oils to coke formation. T. H. POPE.

Naphthenic acids from Baku crude oils. D. O. GOLDBERG and V. L. GURVICH (Azerbeid. Neft. Choz., 1928, No. 5, 46—51).—Balachani-Sabunchi crude oil contains 1.06% of monobasic naphthenic acids, of average mol. wt. 277 and acid value 202; the percentage is greatest in the distillate *d* 0.882. With increase in mol. wt. of the acids *d* and γ decrease, whilst η , *n*, and iodine value increase. CHEMICAL ABSTRACTS.

Crude oil in Kamchatka. S. S. NAMETKIN (Neft. Choz., 1928, 14, 650—651).—Oil from the Bogachevka district had d_{15}^{20} 0.849, Engler viscosity 1.17/20°, flash point (Abel-Pensky) 32.5°, excise resins 1.2%, S 0.05%. CHEMICAL ABSTRACTS.

Distillation [of mazout] in a current of nitrogen. A. ZAKHARENKO (Azerbeid. Neft. Choz., 1928, No. 6—7, 68—72).—A Baku mazout was distilled in circulating nitrogen. Of four fractions (residue 34%) the third (40—60%) was superior, as regards colour and acidity, to steam-distilled oil. CHEMICAL ABSTRACTS.

Mol. wt. and b.p. of mineral oils. A. N. SACHANOV and N. A. VASILEV (Neft. Choz., 1928, 14, 643—650).—The mol. wt. of fractions of Grozni, Balachani, Binagadi, and Kirmaki oils having the same viscosity depend on the nature of the oil, being highest for paraffin-base oils, intermediate for naphthenic-base oils, and lowest for asphalt-base oils. Mazouts of various oils give the following fractions of cylinder stock: Grozni paraffin-base 9, Balachani 14, Grozni non-paraffin-base 30, Binagadi 27, Kirmaki 33%. CHEMICAL ABSTRACTS.

Auto-ignition temperatures of liquid fuels. O. C. BRIDGEMAN and C. F. MARVIN, JUN. (Ind. Eng. Chem., 1928, 20, 1219—1223).—The ignition temperature of a liquid fuel depends on the material and volume of the vessel used for the test, on the concentration of oxygen, the time lag before ignition, the pressure in the system, and the composition of the fuel. As no investigation has developed a test which is standardised in all these respects the results obtained differ widely with the same substance. Experiments carried out with a bomb of design similar to that of the combustion chamber of a Liberty engine showed that the time required to produce firing depends on the rate of evolution of heat by combustion and the rate of heat exchange between the charge and the containing vessel, so that ignition temperatures which involve a time lag are composite functions of several properties of the fuel and the apparatus. Actual firing usually occurs when the temperature of the charge is raised, by chemical reaction, above that of the container; this point is known as the auto-ignition temperature, and is of interest in relation to fire risks, whilst the temperature necessary for ignition without time lag is the true ignition point and is a fundamental property of a given fuel under definite pressure and concentration conditions. Ideal adiabatic compression is the only method for measuring this temperature, but owing to experimental difficulties it would more easily be determined by extrapolation from non-ideal results.

R. H. GRIFFITH.

Determination of the carburetting properties of light motor fuels. N. D. GRAMENICKI (Izvestia Thermo-Tech. Inst. [Moscow], 1928, 6, 12—19).—The

influence of the nature of motor fuels on their vaporisation in the carburettor is discussed. The dependence of the percentage of the whole amount of the fuel which undergoes vaporisation on the temperature, pressure, volume, and surface and time of vaporisation is considered, and a means of evaluating this percentage is evolved. The relationships between the carburetting properties, viscosities, and sp. gr. of various Russian petroleum motor fuels are pointed out. T. H. POPE.

Vapour-pressure chart for paraffin hydrocarbons.

O. G. WILSON, JUN. (Ind. Eng. Chem., 1928, 20, 1363—1366).—An empirical equation has been found which gives the vapour pressure of normal paraffin hydrocarbons in terms of the temperature and of their b.p. at atmospheric pressure; the equation is most readily used in the form of a nomogram. A constant involved in the use of this expression has been determined from existing data for a number of paraffins. The application of these figures to petroleum derivatives is restricted because paraffins other than those of the normal series may be present, and even very close fractionation does not give pure substances. R. H. GRIFFITH.

Miscibility of castor oil with gasoline hydrocarbons. G. H. TABER and D. R. STEVENS (Ind. Eng. Chem., 1928, 20, 1185—1186).—The mutual solubilities of castor oil and gasolines are found to vary with the composition of the latter, as olefinic, aromatic, or naphthenic hydrocarbons are soluble in all proportions, whilst the solubility of paraffins is limited. No quantitative separation of paraffins, however, can be made in this way as the results are influenced by the simultaneous presence of other hydrocarbons. In the case of heptane, the composition of the liquid layers is not quite independent of the amount of heptane used; this is considered to be due to the presence of more than one substance in the castor oil. As the temperature of the mixture is raised, the volume of the lower layer increases until complete miscibility is attained at 33.5°. The viscosity of castor oil-gasoline mixtures measured at 25° agrees closely with that calculated by the Arrhenius formula. R. H. GRIFFITH.

Recovery of products from "spent doctor" and caustic solutions. F. J. MECHLIN (Ind. Eng. Chem., 1928, 20, 1352—1353).—"Spent doctor" solution was found to contain caustic soda, sodium sulphate, lead in the form of plumbite, and tarry substances, as well as suspended lead compounds; spent caustic solution contained sodium salts and tarry material. These liquids were treated by combined evaporation and atmospheric oxidation, attaining a maximum temperature of about 132°, and the layers of soapy matter which contained sodium salts of organic acids, or solid sodium sulphate which separated, were removed. The residual liquors contained about 80—90% of the original alkali or lead respectively, and could be used to treat further quantities of oil. R. H. GRIFFITH.

Shortening the viscosity determination on the Engler and Holde viscosimeters. W. BLEYBERG (Petroleum, 1928, 24, 1416—1417).—The duration of viscosity determinations of highly viscous oils using the Engler or Holde apparatus may be reduced by observing the time of flow of a smaller volume, V (e.g.,

10 c.c.), than the usual 200 c.c. or 100 c.c., respectively. The factors which give the time of flow of 200 c.c. (Engler) and of 100 c.c. (Holde) are, respectively: $0.2486/\{2.6617 - \log(458.9 - V)\}$ and $0.4470/\{2.1920 - \log(155.6 - V)\}$. W. S. NORRIS.

Metal viscosimeter for simultaneous testing of three oils. D. HOLDE (Petroleum, 1928, 24, 1412—1416).—Three viscosimeters substantially of the type already described (*ibid.*, 1918, 13, 505, 663) are included in a common water-bath. W. S. NORRIS.

Standardisation of oil testing. M. FRIEDEBACH (Chem.-Ztg., 1928, 52, 964—966).—Apparatus proposed by Schlüter (cf. B., 1928, 325) for testing flash points is criticised as unnecessarily complicated without any real improvement in accuracy. The exact position of the thermometer bulb in the oil is not of much importance. The essentials are a uniform rate of heating, which depends on the care of the observer, and a uniform gas pressure on the ignition flame, which can be obtained by the use of a laboratory gas-holder. A simple apparatus is described which it is claimed gives results as accurate as more elaborate ones. C. IRWIN.

Influence of physical properties and sizes of filling elements in fractionating columns on the fractionating capacity. K. V. KOSTRIN (Neft. Choz., 1928, 14, 616—626).—Experiments with a crude oil using Raschig rings showed that the nature and shape of the filling elements do not affect the separation of fractions, which depends on the surface of the elements. CHEMICAL ABSTRACTS.

Pyrolysis of methane. WHEELER and WOOD. Slow oxidation of hydrocarbons. LANDA.—See III. Explosive gas mixtures. HAID and SCHMIDT.—See XXII.

PATENTS.

Coke oven. E. COPPÉE & CIE. (B.P. 300,823, 2.1.28. Addn. to B.P. 253,887; B., 1927, 721).—The heating walls of the oven are divided into two independent parts, one on either side of the axis of the oven. When using rich gas, this is led to the lower communicating chambers and to the base of the flues by different ducts according to the direction of travel of the current, but the supply to the upper communicating chamber is led always through the same ducts. Below the oven are arranged alternately air and gas regenerators. The former are divided into separate chambers which communicate respectively with the end groups of flues of each half wall. The gas regenerators are also divided into separate chambers so arranged that the preheated poor gas is supplied simultaneously to the flues of the first group and to the upper communicating chamber between the first and second groups. A. B. MANNING.

Production of a fuel from pulverised coal. W. E. TRENT, ASSR. to TRENT'S PROCESS CORP. (U.S.P. 1,693,356, 27.11.28. Appl., 22.11.26).—Pulverised coal is heated under such conditions that the volatile matter evolved entrains and carries in suspension the fine carbonaceous particles. A. B. MANNING.

Manufacture of liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 275,670 and 295,587, 9.8.27. Ger.,

9.8.26).—Carbonaceous materials are treated with hydrogen, or with gases containing or producing it, under a minimum pressure of 20 atm. (preferably over 50 atm.), using as catalysts (A) silver, gold, ruthenium, rhodium, palladium, osmium, iridium, platinum, or compounds of these metals on supports of magnesia or magnesite, or (B) metallic lead or tin on magnesia or magnesite as carriers, either alone or in mixture with other substances also having a catalytic effect.

H. S. GARLICK.

Process for splitting coal into its constituents. A. PISTORIUS and F. C. BUNGE (B.P. 277,293, 13.5.27. Ger., 8.9.26).—The coal is extracted with a solvent such as pyridine, and the swollen residue is then carbonised. The coke, which may be used as powdered fuel or may be briquetted, can be made more porous by extracting with acids. The pyridine extract and the distillate are worked up by the usual methods of the tar industry.

A. B. MANNING.

Hydrogenation of coal. L. T. BATES (B.P. 281,240, 31.10.27. U.S., 23.11.26).—Finely-divided coal is mixed with a liquid hydrocarbon and subjected to intensive attrition in the presence of hydrogen. The temperature is maintained above the normal but below 300°; the pressure is not allowed to rise above 10 atm. The heat required may be partially or wholly generated by the friction of the grinding mechanism.

A. B. MANNING.

Separation of clay from clay-bearing materials [coal etc.]. CHEM. FABR. IN BILLWARDER, and F. L. KUHLEWEIN (B.P. 302,082, 8.2.28).—Clay is separated from coal etc. by passage upwards through an inclined worm conveyor in which it is treated with a counter-current of a xanthate solution containing, e.g., 4 kg. of sodium carbonate and 400 g. of potassium ethyl xanthate per ton of slime. The lower end of the conveyor communicates with a vibrating, precision grate sieve, and the upper end with a coarser sieve, the "throughs" from this passing on to the precision sieve. L. A. COLES.

Treatment of bone black or char. W. W. KEMP (U.S.P. 1,692,745, 20.11.28. Appl., 28.10.24).—The material is revived in a chamber in which is burning a mixture of fuel gas and air containing no more air than is sufficient for the complete combustion of the gas.

A. B. MANNING.

Gas producer. P. JAWORSKI, Assr. to BISMARCK-HÜTTE (U.S.P. 1,693,483, 27.11.28. Appl., 11.5.25. Ger., 13.5.24).—A gas producer for finely-divided fuels comprises three superposed cylindrical chambers of decreasing diameter, forming respectively an ash chamber, a gas-generating chamber, and a preheating chamber. Between the chambers are transverse walls with fuel ports at their outer edges, the ports in the lower transverse wall, i.e., that below the gas-generating chamber, serving also as air inlets. Above the transverse walls are mechanical rotating devices for imparting motion to the fuel supported thereon.

A. B. MANNING.

Production of a mixture of coal gas and water-gas. DESSAUER VERTIKAL-OFFEN GES.M.B.H., and W. BUEB (B.P. 300,767, 24.10.27).—Two externally heated retorts are charged respectively with coal and with

low-grade fuel, e.g., coke breeze. The retorts are arranged in series so that the distillation products from the coal pass through the heated coke. When the latter has become red hot, steam is introduced into the coal retort, in which it becomes superheated before passing into the coke retort for the production of water-gas. The process may be carried out in a single retort, the lower part of which is charged with coal, and the upper part with low-grade fuel.

A. B. MANNING.

Apparatus for producing gas and coke in alternately-working water-gas generators. A. BREISIG (U.S.P. 1,691,636, 13.11.28. Appl., 4.5.26. Austr., 7.5.25).—A combined coking retort and gas generator with an enlarged lower part has transverse channels branching off from the junction of the upper and lower parts and serving as outlets for the blow gases; the inclination of the bottoms of the channels is arranged to lie approximately in the plane of the slope formed naturally by the coked material when settling in front of the orifice.

A. B. MANNING.

Apparatus for heating by submerged flames. O. BRUNLER (B.P. 300,819, 21.12.27).—The apparatus is designed to permit the flame to burn in an upright position, the burner being so constructed that only the flame enters the vessel containing the liquid to be heated. The burner may be surrounded by a separate compartment through which a cooling liquid is circulated. One form of apparatus designed for the manufacture of Portland cement comprises a drum-shaped vessel with a number of flames entering from below; the vessel is provided with a refractory lining and is mounted on trunnions.

A. B. MANNING.

Apparatus for dehydrating tar. R. CRILLY (U.S.P. 1,693,549, 27.11.28. Appl., 20.5.27).—Above a tank containing the tar emulsion is a pipe coupling with four nipples, to which are attached, respectively, (a) a vertical pipe passing almost to the bottom of the tank, (b) two horizontal pipes, to one of which is attached a steam jet, and (c) a plug. A second vertical pipe is connected to the bottom of the steam jet and projects into the tank.

A. B. MANNING.

Apparatus for extracting gas and oil from waste. A. NAERT (U.S.P. 1,693,456, 27.11.28. Appl., 7.10.26).—The material is heated in a vertical retort which has a hollow base serving as a combustion chamber, from which flues pass vertically up through the retort and communicate with a horizontal flue lying within the upper end of the retort and in turn communicating with a smoke stack. A pipe from the upper end of the retort discharges the products of distillation into a tank containing water, and provided with valved outlets at the top and bottom.

A. B. MANNING.

Recovery of oils of high b.p. from the residues of mineral oils, tar oils, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 301,130, 5.9.27).—Decomposition losses in distillation under diminished pressure at temperatures above 300° are avoided by the introduction of gases and/or vapours which contain liquids of low b.p., e.g., water, benzene, toluene, alcohol, benzine, etc., in a liquid form and preferably in the form of mist.

H. G. GARLICK.

Removing hydrogen sulphide from coal gas or water-gas. W. LEUCHTENBERG (Re-issue 17,162, 11.12.28, of U.S.P. 1,629,396, 17.5.27).—See B., 1927, 626.

Apparatus for reclaiming used lubricating oil. P. F. MILLER, ASST. to DE LAVAL SEPARATOR CO. (U.S.P. 1,694,478, 11.12.28. Appl., 6.9.24).—See B.P. 239,506; B., 1926, 1004.

Burner for pulverulent fuel, gas, or oil. G. H. TURRALL (B.P. 301,851, 2., 7., and 9.9.27).

Supplying heat in high-pressure reactions (B.P. 275,248). Fractionating towers (B.P. 270,720 and 299,479).—See I. Concentration of graphite (B.P. 275,673).—See X.

III.—ORGANIC INTERMEDIATES.

Pyrolysis of methane. R. V. WHEELER and W. L. WOOD (Fuel, 1928, 7, 535—539; cf. Fischer, B., 1928, 844).—When heated in a quartz vessel methane begins to decompose at 650—685°. At higher temperatures (875—1100°) benzene, naphthalene, ethylene, and butadiene were among the products of decomposition. The optimum yield of benzene (0.2 gal. per 1000 cub. ft. of methane) was obtained by passing the gas at about 30 litres/hr. through a horizontal quartz tube maintained at 1050°. Above 1000° some carbon smoke is formed. Increasing the area of heated surface, whether this be of quartz, porcelain, or copper, had little influence on the course of the reaction, but in the presence of iron the gas is decomposed into its elements, no condensable products being obtained. Dilution of the methane with nitrogen had little effect on the yield of liquid hydrocarbons, but dilution with hydrogen produced a marked decrease in the yields. A. B. MANNING.

Slow oxidation of hydrocarbons. S. LANDA (Chem. Listy, 1928, 22, 294—298, 499—502).—Paraffin, m.p. 51°, b.p. 360°, was heated at 280—300°, a current of air being passed through the liquid. The products were formaldehyde, propaldehyde, butaldehyde, heptaldehyde, octaldehyde, and decaldehyde, together with acetone, methyl ethyl ketone, and methyl and ethyl alcohols. A small quantity of asphalt remained in the reaction flask. Under similar conditions *n*-triacontane yields formaldehyde, hexaldehyde, and heptaldehyde, and formic, butyric, and valeric acids. The above results confirm the view that alcohols and ketones are obtained only from the oxidation of side-chain hydrocarbons. R. TRUSZKOWSKI.

Sp. gr. of glycerol. L. W. BOSART and A. O. SNODDY (Ind. Eng. Chem., 1928, 20, 1377—1379; cf. B., 1927, 377).—The authors' original tables have been recalculated to the standard of absolute densities. The values are compared with those of the International Critical Tables, and are claimed to be more accurate. E. H. SHARPLES.

Use of *o*-cresol in working-up middle tar oil. F. BOVINI (Notiz. Chim.-ind., 1928, 3, 3—5; Chem. Zentr., 1928, ii, 112).—A description of dyes derived from *o*-cresol. A. A. ELDRIDGE.

Catalytic oxidation of naphthalene. T. KUSAMA (Bull. Inst. Phys. Chem. Res., Tokyo, 1928, 7, 1087—1132).—If naphthalene in the gaseous state is oxidised

in presence of vanadium pentoxide as catalyst in order to prepare phthalic anhydride, the heat of reaction is so great that unless it is spread through the whole of the reacting mixture, or the system is cooled, or the catalyst is diluted with some inert substance, the anhydride itself is oxidised. Further, there is a tendency for the pentoxide to be reduced to lower basic oxides, which combine with the phthalic anhydride. This difficulty can be overcome by mixing with the catalyst some acidic substance as promoter, preferably an oxide of molybdenum, tungsten, cerium, chromium, or uranium, or by adding such a substance as sulphur dioxide to the reacting gases. R. CUTHILL.

Oxidation of anthracene by nitrogen oxides. M. A. ILINSKI, B. V. MAKOROV, and N. V. ELAGIN (J. Chem. Ind. Moscow, 1928, 5, 469—473).—An attempt was made to use crude 45—50% anthracene for the manufacture of anthraquinone by oxidation with nitrogen oxides (G.P. 268,049). Nitrogen oxides (from sodium nitrite and sulphuric acid) have no chemical action on anthraquinone or on the solvent nitrobenzene, although the latter, when cooled, absorbs more than 25% of nitrogen oxides. Pure anthracene, when added in small portions to a cooled solution of nitrogen oxides in benzene, gives a practically quantitative yield of anthraquinone. 2-Methylantraquinone is unaffected, and hence, if present in anthracene, will contaminate the product. Carbazole is converted successively into 9-nitroso-, 3-nitroso-, 3-nitro-, 3-nitro-9-nitroso-, 3-nitro-6-nitroso-, and (finally and mainly) 3:6-dinitro-carbazole. Phenanthrene affords resinous oxidation products. From anthracene of 50% purity, 25.2% of anthraquinone was obtained; with anthracene of 87% purity, 80% of the theoretical yield of anthraquinone (93—97% pure) was obtained. With anthracene of 25.63 or 41.9% purity, anthraquinone, with nitrogen and sulphur compounds, was distributed in all fractions. For the separation of anthraquinone from other products of oxidation of impure anthracene, treatment with concentrated sulphuric acid, or partial reduction using alkaline sodium hyposulphite or zinc powder and alkali hydroxide, was unsatisfactory. CHEMICAL ABSTRACTS.

Action of light on anthracene. E. HIBBERT (J. Soc. Dyers and Col., 1928, 44, 377).—Anthracene dissolved in a solvent which does not absorb or dissolve oxygen and exposed to light, undergoes polymerisation, but if the solid material is exposed to light its colour changes to a yellowish-brown. The product on examination was found to give the reactions characteristic of anthraquinone. Anthraquinone is also formed if anthracene is exposed behind glass to the light of the fadeometer. L. G. LAWRIE.

Anthraquinone solutions in sulphuric acid. P. I. SOKOLOV and V. P. DREYING (J. Chem. Ind. Moscow, 1928, 5, 412—416).—The solubility of anthraquinone (99.25% purity) in sulphuric acid (82.5—99.7%) at 26—135° was determined; the increase with rise of temperature is greater for higher than for lower concentrations. When anthraquinone is heated for 6 hrs. at 115—125° with twice its weight of 99.7% sulphuric acid, the loss is 1.2%; with four times its weight of 93.3% acid, the loss is 0.025%. The former product

is the darker. By dilution of the solution of anthraquinone in sulphuric acid with cold water, the product is not easily filterable; the solution is preferably cooled to 30°, when fairly large crystals are obtained. The mixture is diluted to an acid concentration of 90% and heated at 110° to redissolve the second (fine) precipitate, and the whole cooled to 30°. Two further precipitations are similarly effected, the acid concentrations being 87 and 80%, respectively; the final precipitate is easily filtered and washed.

CHEMICAL ABSTRACTS.

Humic acids. SIMEK. **Synthesis of hydrocarbons from water-gas.** SMITH and others. **Utilisation of coal-tar crudes.** JAEGER.—See II. **Determination of hydrogen value of unsaturated compounds.** WATERMAN and others.—See XII. **Phenol in sewage.** DEHE. **Vapour pressures of fumigants.** NELSON.—See XXIII.

PATENTS.

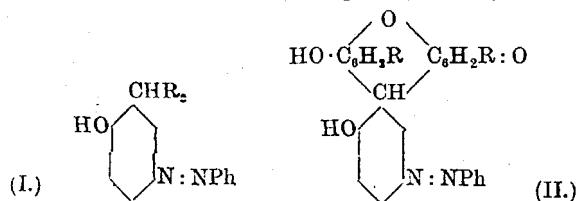
Purification of butaldehyde. C. BOGIN, Assr. to COMMERCIAL SOLVENTS CORP. (Re-issue 17,157, 11.12.28, of U.S.P. 1,556,067, 6.10.25).—See B., 1926, 28.

Manufacture of glycols. G. O. CURME, JUN., Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,695,250, 11.12.28. Appl., 7.1.26).—See B.P. 264,124; B., 1927, 733.

Supplying heat in high-pressure reactions. (B.P. 275,248).—See I.

IV.—DYESTUFFS.

Azo-triphenylmethane and azo-pyrone dyes (meta series). R. N. SEN and B. GHOSH (J. Indian Chem. Soc., 1928, 5, 487–496).—Condensation of benzeneazosalicylaldehyde with (a) dimethylaniline, (b) *o*-cresotic acid, (c) resorcinol, (d) pyrogallol, and (e) *m*-diethylaminophenol gives (a) 4':4''-tetramethyldiamino-2-hydroxy-5-benzeneazotriphenylmethane (in I, R = C₆H₄·NMe₂), (b) the compound I [R = C₆H₂·Me(OH)·CO₂H], (c) the compound II (R = H), (d) the compound II (R = OH), and (e) the compound II (R = H; OH and O = NEt₂), respectively.



The shades of the substances (c), (d), and (e), and of the carbinols from (a) and (b), on wool and silk are recorded. Similar series of dyes have been prepared from naphthaleneazosalicylaldehyde, m.p. 151° (phenylhydrazone, m.p. 96°; oxime, m.p. 201°), diphenyl-4-azosalicylaldehyde-4'-azosalicylic acid, not melted at 300° (oxime, m.p. above 300°), and diphenyl-4:4'-bisdiazosalicylaldehyde, not melted at 300° (dioxime, m.p. above 300°), and (a), (b), (c), (d), and (e), respectively. The introduction of one azo group in the meta-position generally increases the depth of colour, although to a much smaller extent than the corresponding para-derivatives. A second azo group intensi-

fies the colour only slightly, whilst the introduction of a second triphenylcarbinol group into a molecule containing one such group and two azo groups causes a diminution in the intensity of the colour. The yellow shades on wool produced by the leuco-compounds of series *b* change to olive-brown by after-chroming, and are only feebly polygenetic (cf. Green and Sen, B., 1912, 579).

H. BURTON.

Saffron crocus. C. GRIEBEL and F. WEISS (Apoth. Ztg., 1928, 43, 642–644; Chem. Zentr., 1928, ii, 279).—Natural saffron contains ammonium chloride or sulphate; the older specimens contained the smaller quantities. Alkali hydroxide liberates larger quantities than magnesia; volatile bases are therefore considered to arise from the cell contents. Light petroleum extracts small quantities of colouring matter from saffron.

A. A. ELDRIDGE.

Use of *o*-cresol. BOVINI.—See III. **Colour measurements.** DESBLEDS.—See VI. **Dyes in medicine.** VAN URK.—See XX.

PATENT.

Manufacture of dyes from naphthidine. G. LE FLOCH (U.S.P. 1,694,884, 11.12.28. Appl., 13.12.23. Fr., 21.12.22).—See B.P. 208,720; B., 1924, 862.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemical analysis of cotton. Determination of cellulose by oxidation with chromic acid. C. BIRTWELL and B. P. RIDGE (J. Text. Inst., 1928, 19, 341–348 T).—Two methods are described, one gas-volumetric and the other titrimetric, and their application is illustrated by the combustion of bleached cotton, starch, cellulose in cuprammonium solution, and of modified celluloses in sodium hydroxide solution. In the former method, 0.02–0.1 g. of carbohydrate is made to undergo combustion either with a mixture of chromic and phosphoric, or chromic and sulphuric, acids, and the volume of carbon dioxide so obtained is measured in a gas burette. For the analysis of solutions of low cellulose concentration the sulphuric acid mixture is used, and no error due to the formation of sulphur dioxide is introduced if excess of chromic acid is present throughout the whole of the analysis; for the undissolved substances either sulphuric or phosphoric acid may be used. In the titrimetric method the cellulose or cellulose solution is boiled with an excess of standard dichromate solution and sulphuric acid for $\frac{1}{2}$ –1 hr., and the excess dichromate is either determined iodometrically or titrated with standard ferrous ammonium sulphate solution (using ferricyanide as an external indicator). In order to avoid loss of oxygen by direct decomposition of the chromic acid, the reaction mixture must not contain more than 1 vol. of added concentrated sulphuric acid to one of the mixed dichromate and cellulose solutions. In both methods the amount of material taken for analysis is found by assuming that the composition of the carbohydrate is represented with sufficient accuracy by the formula C₆H₁₀O₅, and that complete oxidation to water and carbon dioxide occurs.

B. P. RIDGE.

Chemical analysis of cotton. Action of sodium hydroxide solutions on modified cotton cellulose at the ordinary temperature. C. BIRTWELL, D. A.

CLIBBENS, and A. GEAKE (*J. Text. Inst.*, 1928, **19**, 349—364 T).—The solubility of modified cellulose in sodium hydroxide solution has been determined volumetrically (cf. preceding abstract). Maximum solubility for cotton, modified by means of either acids or oxidising liquids, on treatment with any single solution at 15°, occurs in 3*N*-sodium hydroxide, and the solubility falls rapidly from this point either with increasing or decreasing alkali concentration. If the modified cotton is first treated with a more concentrated solution of sodium hydroxide (6—10*N*) and the latter is subsequently diluted with water, a much larger percentage weight of the cellulose dissolves than when a single undiluted solution is used. Maximum solubility under these conditions occurs at 15° when the material is steeped in 10*N*-sodium hydroxide with subsequent dilution to 2*N*, and by using a standardised procedure on these lines (called the 10*N*—2*N*-treatment) an investigation has been made of the way in which the solubility of modified cotton varies with the extent and nature of the modification. For hydrocelluloses formed by the action of acids on bleached cotton under widely different conditions, the solubility is completely defined when the copper number (Schwalbe-Braidy) is known, but very different relations obtain with oxycelluloses. In any one series of oxidised cottons formed by the action of the same oxidising agent acting under the same conditions but for different times, the copper number of the material defines the solubility, but for different oxidising agents, or for the same oxidising agent acting under different conditions, there is no general relation between solubility and copper number. Although the reducing substances in modified cotton dissolve to a certain extent in the sodium hydroxide, their presence, as such, does not completely account for the loss in weight of the material on extraction. There is a general correlation between the solubility of modified cotton in 10*N*—2*N*-sodium hydroxide and its fluidity in cuprammonium, which is independent of the method of modification, and is not affected by boiling the modified material with dilute alkali. Comparisons have been made between the copper numbers of modified cottons and the reducing properties of their alkali-soluble fractions, the "reducing values" of the latter being determined by a modification of the Schwalbe-Braidy method for the determination of copper number which avoids precipitation and drying of the cellulose extract. For hydrocelluloses extracted with 10*N*—2*N*-sodium hydroxide, the reducing value of the extract is roughly constant at the value 4, irrespective of the nature and extent of the acid attack, whilst similar values for the extracts of oxycelluloses depend greatly on the nature of the oxidising attack and vary from 8 to 20. The relations described do not hold for cotton in the form of cloth unless it is completely disintegrated by fine cutting before extraction, whilst both oxycelluloses and hydrocelluloses of the same copper number prepared from mercerised cotton dissolve to a much smaller extent than do preparations of the same copper number from unmercerised cotton. Diminution in solubility, caused either by the mechanical form of the material or by mercerisation prior to chemical modification, is accompanied by an increase in the reducing value of the material dissolved. B. P. RIDGE.

Copper numbers of cotton. KOEHLER and MARQUEYROL (*Ann. Chim. analyt.*, 1928, [ii], **10**, 349—351).—The so-called Schwalbe-Braidy method for the determination of copper numbers is described in which a mixture of sodium carbonate and bicarbonate is used instead of sodium hydroxide in preparing the alkaline copper sulphate solution, in order to prevent auto-reduction of the latter during the heating. Copper numbers for various materials such as scoured cottons and artificial silk as found by this method are given. It is pointed out in a footnote that this method, which has been ascribed to Braidy, was originated by the authors (cf. B., 1922, 323 A) and not by Braidy, who published an account of it without quoting the authors' names. B. P. RIDGE.

Local strength test for fabrics, textiles, rubber, etc. F. SCHUBERT (*Chem.-Ztg.*, 1928, **52**, 913—915).—The method is based on point penetration by two short parallel cutting edges attached to a rigid support. Measurements are made in directions corresponding to the warp and woof, the pressure being measured by the ordinary dynamometer method. Very small test pieces only are needed, and concordant results are obtained without tedious preparation of the sample, whereas the ordinary tearing test is slow, gives variable results, and at best measures only the strength at the weakest point of the sample. S. I. LEVY.

The cellulose industry and its products. G. K. BERGMAN (*Förh. III nord. kemistmötet*, 1928, 143—166).—An historical account of the rise and development of the cellulose industry in Finland. The nature and properties of commercial cellulose are discussed, and illustrations showing the appearance of different types of cellulose products under the microscope and ultramicroscope are given. Values obtained from Finnish sulphite-cellulose when subjected to the usual standard tests are tabulated. H. F. HARWOOD.

Cellulose cooking with hot water as the heating agent. L. FRIEDLÄNDER (*Papier-Fabr.*, 1928, **26**, 789—791).—The chief disadvantages of the indirect and direct cooking processes in which steam is used as heating agent, which are, respectively, prolonged time of cooking and dilution of the liquor, are avoided if indirect heating by means of hot water is used. The water, at about 164° and 6 atm. pressure, is circulated through the heating chamber or coils by means of a rotary pump, and the cooking process is controlled by altering either the pressure or the rate of flow of the heating water. The time of cooking is about half that required in the indirect steam cooking process, a more uniform and better disintegrated product is obtained in greater yield, and the cooking operation is simplified. B. P. RIDGE.

Tenacity and elongation of artificial silks. P. E. KING and E. N. JOHNSON (*J. Soc. Dyers and Col.*, 1928, **44**, 346—350).—Samples of viscose, cuprammonium, Chardonnet, and cellulose acetate silk yarns of 45—300 denier were examined at constant and at different humidities at 22°, the yarns being conditioned for at least 30 hrs. before being tested. In testing wet yarn it was found sufficient to immerse the yarn in water for 2 min. before testing in the case of cellulose silks, but for not less than 15 min. in the case of cellulose

acetate silk. Accurate average results were obtained by testing 20 lengths of yarn taken indiscriminately from the bundle of yarn lengths obtained by cutting an ordinary hank. Almost all the yarns had a tenacity not exceeding 1.5 g. per denier at R.H. 66%. The tenacity of cellulose acetate silk was smaller than that of viscose silk in the dry condition, but about equal to it in the wet. The average wet tenacity of the silks was about 0.8 g. per denier, but values as low as 0.5 g. per denier were obtained in many cases. The increase of extensibility in the wet state of viscose silk is slight, and of cellulose acetate silks considerable, but the extensibility of cuprammonium and Chardonnet silks is inappreciably affected by wetting. The dry and wet extensibilities of viscose, cuprammonium, Chardonnet, and cellulose acetate silks are, approx., 23, 27%; 11, 11%; 10, 10%; and 25, 30%, respectively. All types of silk suffer a steady decrease of tenacity with increasing humidity.

A. J. HALL.

Metallic complexes of nitrocellulose. J. DUCLAUX (Rev. gén. Colloid., 1928, 6, 222—224).—In the manufacture of artificial silk and other industrial operations involving the filtration of nitrocellulose solutions, it is frequently observed that the filters rapidly become stopped up. This is due to an insoluble gummy substance, which has been found to contain lead in small quantity, but in considerably greater amount than the lead content of the soluble part. The source of the lead is the sulphuric acid used in the manufacture of the nitrocellulose. Other heavy metals produce the same effect to a less extent. The metallic complexes concerned are considered to be similar to those studied by Apard (cf. A., 1928, 276), in which the nitrocellulose appears to behave as a weak acid, and it is suggested that the lead content of nitrocellulose preparations should be carefully controlled in technical operations.

E. S. HEDGES.

Artificial humification of carbohydrates, with particular reference to the formation of so-called humic acids from cellulose. W. FUCHS (Brennstoff-Chem., 1928, 9, 400—402).—Carbohydrates, when energetically treated with certain reagents, give good yields of ulmin-like substances which are, however, relatively sparingly soluble in alkalis. The simple sugars, e.g., dextrose, appear to function as intermediate products, but in nature these compounds would be subject to microbic attack. The artificial ulmins and humic acids present certain similarities to the naturally occurring substances, but also several differences; e.g., the artificial humic acids are soluble in acetone.

W. T. K. BRAUNHOLTZ.

Importance of aluminium sulphate in paper manufacture. H. ROSCHER (Papier-Fabr., 1928, 26, 757—761).—Views expressed by previous workers, and the results of their investigations on the absorption of aluminium from aluminium salt solutions by cellulose, are discussed. The percentage weight of aluminium absorbed by bleached sulphite-cellulose under standard conditions depends on the degree of hydrolysis of the aluminium sulphate, i.e., it increases both with dilution of the liquid and with increase in the sodium hydroxide concentration of the liquid. A considerable increase

in the percentage weight of aluminium absorbed is obtained if a colloidal solution of the hydroxide is used instead of the aluminium sulphate solution. The residual calcium (and other metals such as magnesium) in the cellulose fibres can be almost completely replaced by aluminium from an alum solution. The influence of the anion is shown by the fact that when ferric chloride and sulphate solutions, respectively, are used instead of alum solution, considerably more calcium is displaced from the cellulose in the sulphate than in the chloride solution. It appears that several ions can replace the ash constituents of cellulose under suitable conditions, and that this property of the ions is connected with their electric charges. Conditions affecting the degree of hydrolysis of the aluminium sulphate and reactions between the alum and size in the various stages of paper manufacture are discussed.

B. P. RIDGE.

Beating. III. Direct measurement of the water of imbibition of [paper] stuff. M. NAKANO (J. Cellulose Inst., Tokyo, 1928, 4, 300—304).—No difference in the water content of sheets made of the stuff whether boiled or unboiled, after pressing them at 20° was noted. The water of imbibition is very small, and is therefore difficult to determine by any pressing method.

A. G. POLLARD.

Cellulose extracts for tanning. ANACKER.—See XV.

PATENTS.

Treatment of cellulosic material. EUROMERICAN CELLULOSE PRODUCTS CORP., Assees. of B. DORNER (B.P. 286,211, 31.12.27. U.S., 28.2.27).—Straw-like material, after being shredded and dry-sieved, is washed and disintegrated, the latter process being carried to a point where the fibrous structure is sufficiently opened up to permit access of the wash-water but not so as to produce a pulp fine enough to entrap dirt from the wash-water. It is then subjected to a two-stage alkaline cook, the first with just sufficient alkali to remove the silica present, and the second with a larger quantity to remove other non-cellulose material. F. R. ENNOS.

Neutralising cellulose-bearing material for subsequent saccharification. J. PERL, Assr. to M. M. CORY (U.S.P. 1,687,785, 16.10.28. Appl., 15.6.25).—After treatment of the cellulose material with warm dilute alkali solution, it is washed and mixed with sufficient dilute sulphuric acid for neutralisation of the adsorbed alkali and of the natural mineral bases of the material.

F. R. ENNOS.

Manufacture of artificial silk. J. M. LEAVER, Assr. to PACIFIC LUMBER CO. (U.S.P. 1,685,640, 25.9.28. Appl., 3.9.24).—The viscous solution is discharged vertically downwards in a number of streams which are allowed to fall through the air to reduce their diameter. They are then received on the surface of the coagulating liquid, which moves across their path in a descending incline at a speed which synchronises with that of the falling streams at their point of contact with it.

F. R. ENNOS.

Production of thin films of cellulose derivative. A. EICHENGRÜN, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,688,457, 23.10.28. Appl., 3.8.23. Ger.,

10.8.22).—A solution of the cellulose ether or ester, to which is added a material to diminish the adhesiveness of the film produced, is spread in a very thin layer on to a base consisting of another cellulose derivative which is insoluble in the solvent used for the coating film. After evaporation of the solvent the thin film is removed from the base and rolled in an endless band.

F. R. ENNOS.

Apparatus for manufacture of artificial silk.

(A) N. B. GRILLET, and (B) L. E. G. LAHOUSSE, Assrs. to Soc. FABR. SOIE "RHODIASETA" (U.S.P. 1,695,094 and 1,695,111, 11.12.28. Appl., [A] 24.9.26, [B] 6.5.27. Fr., [A] 1.10.25, [B] 20.5.26).—See B.P. 259,190 and 269,377; B., 1927, 406, 472.

Bisulphite digesting liquor (U.S.P. 1,694,231).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effects produced by the singeing operation on the dyeing properties of cotton. G. E. HOLDEN (J. Soc. Dyers and Col., 1928, 44, 368).—During the singeing of cotton a measurable degree of oxidation takes place and tends to cause a marked modification of the properties of the affected portions of the cotton, one of the most noticeable being the lessened affinity for substantive dyes. Cotton velveteen particularly lends itself to the production of two-tone effects by dyeing after singeing under carefully controlled conditions. It has been found in practice that 5—6 times the amount of dye is required for thoroughly singed cotton as for the non-singed material to produce the same depth of colour.

L. G. LAWRIE.

Auxanin B [for increasing the fastness to light of basic dyes]. P. RABE (Textilber., 1928, 9, 665).—Auxanin B is a violet-black, water-soluble, crystalline powder (cf. B.P. 292,253; B., 1928, 603) which increases considerably the fastness to light of cotton and cellulose silk materials dyed with basic dyes on a tannic acid or Katanol mordant; it similarly increases the fastness of basic dyes on silk not mordanted or weighted, but not on wool. In applying Auxanin B, the dyed material is treated for $\frac{1}{2}$ — $\frac{3}{4}$ hr., in a bath at 25—30° containing 2—5% of Auxanin B, and then dried without rinsing. The absorption of Auxanin B by the dyed material is somewhat slow so that its application by padding methods is not satisfactory. The fastness to light of Malachite Green on non-mordanted Chardonnnet or cellulose acetate silk is greater than its fastness on cotton or cellulose silk mordanted with tannic acid.

A. J. HALL.

Fixation of pigments on textile fibres. G. E. HOLDEN (J. Soc. Dyers and Col., 1928, 44, 364—367).—Experiments to determine the capacity of linseed oil as a fixing agent for various pigments on cotton velveteens and other textile fabrics are described. Prussian blue was used as the pigment for these experiments. In order to obtain even application of the pastes on textile fabrics a very finely engraved metal roller was used for applying the "paint," the cloth being subsequently acted on by a series of revolving brushes. Large-scale experiments were carried out on cotton velveteen in order to determine the amount of linseed oil which could be applied to the fabric without causing appreci-

able stiffness; this amount was found to be 10 g. of linseed oil per sq. yd. It was also found that approx. 1.3 g. of Prussian blue per sq. yd. was the maximum amount which could satisfactorily be fixed on the material, using the above amount of linseed oil. Further experiments showed that by means of suitable mixtures it was possible to fix satisfactorily as much as 4 g. of Prussian blue per sq. yd. on the velveteen. Other substances such as tung oil, gelatin, and various waxes were examined, but none of these substances possessed any advantages over linseed oil.

L. G. LAWRIE.

Influence of the anions of the mordant on the shade of Alizarin-red lake. P. P. VIKTOROV (J. Soc. Dyers and Col., 1928, 44, 336—341).—The causes of the detrimental effects of iron salts (due to the formation of purple iron alizarate) in aluminium mordants containing various organic anions are studied. Alizarin lakes of ferrous iron are unstable, and it is found that reducing agents such as sodium hyposulphite, hydroxylamine, stannous oxide, sodium bisulphite, potassium sulphite, and glucose when present in an alizarin printing colour containing iron hinder the formation of the iron lake in proportion to their reducing power under the conditions which exist in steaming. In producing fast printed alizarin lakes it is essential that lake formation on the fibre should proceed slowly, so that in producing such fast lakes free from iron alizarate it is necessary to have present in the mordant a substance capable of preventing any iron present from combining with the alizarin and also an aluminium mordant which hydrolyses slowly. The beneficial action of potassium thiocyanate in printing is chiefly due to the fact that it resists entrance of iron (from the colour and lint doctors etc.) into the printing colour. During steaming the mordant is hydrolysed with consequent development of free acid, which, if organic, may have sufficient reducing power to prevent the formation of ferric alizarate. It is found that the reducing powers of formic, lactic, tartaric, and acetic acids during prolonged steaming decrease in the order named. The products of acid hydrolysis of the starch thickening present in a printing paste in which free acid has been formed as a result of hydrolysis of the mordant may also act as reducing agents and so favourably influence the resulting shade. Hence, starch would be preferable to gum as a thickening agent were it not for the fact that the inferior reducing properties of the gum are more than counterbalanced by its greater penetration into the cotton; a gum thickening containing aluminium chloride thus shows greater reducing power than a similar starch thickening during steaming. Arsenious acid has a similar action to a thiocyanate in resisting the introduction of iron into a printing colour.

A. J. HALL.

Fading of dyestuffs on textile fabrics. II. Influence of regain. III. Influence of temperature. IV. General. J. J. HEDGES (J. Soc. Dyers and Col., 1928, 44, 341—346; cf. B., 1928, 260).—II. The influence of atmospheric humidity on the fading of dyes on wool as previously described (*loc. cit.*) has been further determined by the same methods in respect of the fading of Brilliant Green C27, Safranine

FF, Brilliant Victoria Blue RB, and Magenta on mercerised cotton, Ponceau RG on silk, and Methyl Violet 10BL and Soluble Pure Blue on wool. The previous conclusions for wool are confirmed, and the loss of colour by cotton is also found to be a linear function of its regain for regains exceeding 5%; for regains of less than 9% the fading-regain curve for silk is not linear, the fading being much more rapid at low than at high regains. The similarity between the fading of wool (regain <8%) and that of silk (regain <9%) is suggested.

III. The rates of fading of Soluble Pure Blue, Acid Green, Ponceau RG, Azo Rubine, and Cyanine B on wool at 10° and 50° each at four different humidities, were found to increase with rise of temperature. The importance of temperature conditions in fading is equal to that of humidity.

IV. The results of various investigations are discussed, and the expression $F = K\sqrt{T}(R + C)$, in which F = loss of colour, R = regain, T = time of exposure, and K , C are constants, for the fading of dyes on wool is obtained. When certain assumptions are made, K and C can be determined, so that the amount of fading under various conditions of time and regain can then be calculated.

A. J. HALL.

Eliminating eye estimates from colour measurements. L. B. DESBLEDS (J. Soc. Dyers and Col., 1928, 44, 327—335).—The colour of any dyed material is defined independently of the human eye but relatively to a surface of standard white plaster by means of a curve having ordinates representing the intensities of the radiations emitted in the violet (400 Å.), blue (450 Å.), green (530 Å.), yellow (580 Å.), orange (620 Å.), and red (700 Å.) zones of the solar spectrum. These intensities are measured by means of a Toussaint photo-electric photo-colorimeter in which radiation from the coloured object is directed through a suitable colour-filter on to a photo-electric cell and thereby produces a variation of electric current in a circuit which includes a galvanometer. Every colour has its characteristic curve, and determination of such curves allows the detection of very small differences between apparently similar colours. The application of these methods in the dyeing and dye-making industries is indicated.

A. J. HALL.

Absorption of acids by wool. II. H. WILKINSON and A. G. TYLER (J. Soc. Dyers and Col., 1928, 44, 369—376).—The results are given of a number of experiments carried out in order to test the accuracy of the available data relating to the absorption of acids by wool. The authors claim that there is perfect agreement between their own data and that of several other workers, and that the equilibrium between the acid absorbed by the wool and the acid remaining in the liquor is represented by the expression $y = a \log bx$, where y is the % of acid absorbed by fibre, x the % concentration of acid left in the bath, and a , b are constants. Other experiments were carried out in which the wool phase was relatively larger and the liquid phase smaller. The absorption of acids by wool is considered to be a chemical reaction.

L. G. LAWRIE.

PATENTS.

Dyeing. A. BODMER, Assr. to HEBERLEIN & Co. A.-G. (U.S.P. 1,694,526, 11.12.28. Appl., 10.7.26. Switz., 14.7.25).—See B.P. 255,453; B., 1927, 103.

Treatment [dyeing] of cellulose acetate. Dyeing, printing, or stencilling of cellulose acetate. G. H. ELLIS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,694,413—4, 11.12.28. Appl., [A] 15.8.25, [B] 10.3.26. U.K., [A] 19.9.24, [B] 18.3.25).—See B.P. 242,393 and 253,978; B., 1926, 87, 741.

Production of metallic effects on fabrics containing organic derivatives of cellulose. R. CLAVEL (U.S.P. 1,694,466, 11.12.28. Appl., 6.4.26).—See B.P. 269,128; B., 1928, 260.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Fineness and available lime content of chemical quicklimes. J. S. ROGERS (Ind. Eng. Chem., 1928, 20, 1355—1356).—The quality of lime as used in industrial chemical processes depends, apart from chemical analysis, on the available alkalinity, and on the degree of fineness when hydrated. Results of screening tests on different samples of chemical quicklime after excess hydration showed a range of 0.7—50.1% retained on 30-mesh and 46—96.1% through 200-mesh. The available alkalinity, as titrated with hydrochloric acid and assuming neutralisation of calcium hydroxide only, showed also varying figures, 49.3—97.1%, with little correlation with the total content of calcium compounds, which in all cases was 92% CaO or more.

C. A. KING.

Action of aluminium, ferrous and ferric iron, and manganese in base-exchange reactions. O. C. MAGISTAD (Ariz. Agric. Exp. Sta. Tech. Bull., 1928, 18, 445—463).—Treatment of artificial zeolites with solutions of aluminium or ferric salts released the replaceable base previously present; the residue usually contained no hydrogen-zeolite, nor was the iron or aluminium subsequently replaceable, and the base-exchange capacity was finally lost. With alcoholic aluminium chloride there was no addition of aluminium or marked loss of aluminium or silicon; with ferric chloride in water, or particularly in ethyl alcohol, iron replaced aluminium, but not in equivalent quantities. Ferrous iron is readily exchangeable for calcium in a zeolite and in soils, and is replaceable by barium; bivalent manganese behaves similarly.

CHEMICAL ABSTRACTS.

The Backman mechanical bleaching powder chamber. G. ANGEL (Chem.-Ztg., 1928, 52, 962—964).—The construction of chambers in reinforced concrete is not in itself new, but failures were not uncommon in the earlier type of chamber. Very careful workmanship is necessary; the inner surfaces must be finished smooth without sharp corners. Several towers are usually built together. The arms and rakes of the stirrers are of iron covered with a special preparation. They last for a year or more according to position. The chlorine feed is introduced on the second or third shelf from the bottom. Mechanically hydrated lime

with not more than 0.5% of excess moisture is preferred. The reaction is controlled by temperature readings on each shelf. All shelves except the top one are provided with cooling coils. The highest temperature of about 35° should be on the third shelf upwards. The capacity of the latest chambers is 5 tons per 24 hrs. with a consumption of 1½ h.p. The cost of construction is about the same as with lead chambers; that of operation is less. They are always under slight vacuum, and no chlorine should be present in the exit gas.

C. IRWIN.

Recovery and use of sulphur dioxide in sulphur extraction. A. GALLETTI and DI S. CATALDO (Atti II Cong. Naz. Chim. pura appl., 1926, 708—718; Chem. Zentr., 1928, ii, 379).—The sulphur mineral is treated with aqueous sulphur dioxide to convert most of the undesirable gangue into calcium hydrogen sulphite; the sulphur content of the washed material is thereby considerably increased. A. A. ELDRIDGE.

Modern methods of sulphur extraction. Heat balance and theoretical proportions by weight. New type of furnace. A. RICEVUTO and G. BUOGO (Atti II Cong. Naz. Chim. pura appl., 1926, 757—789; Chem. Zentr., 1928, ii, 379).—The heat energy employed for the fusion of sulphur is only slightly greater in the Gill furnace than in calcaroni (3.75%, < 3%); in a new type of externally-heated furnace, in which a temperature of about 145° is maintained, 12.5% is utilised.

A. A. ELDRIDGE.

Standards for Gutzeit test [for arsenic]. A. T. HENLEY (J. Inst. Brew., 1928, 34, 608—609).—To obviate the necessity of frequently replacing standard stains produced by the Gutzeit method on account of their impermanence, it is suggested that standards be made by painting strips of drawing paper with "Chrome Lemon" and "Chrome Deep." A full range of standards, which must be checked occasionally, can be obtained by using graduated dilutions of these colours or of a mixture of the colours in equal proportions.

C. RANKEN.

Products from "doctor" and caustic solutions. MECHLIN.—See II. Apatite from phosphatic iron ores. LUYKEN and BIERBRAUER.—See X. Red lead. JUNK. Lead in litharge and red lead. FLUCH. Gold resinate. CHEMNITUS and BARFUSS-KNOCHEN-DÖPFEL.—See XIII. Ferric citrate solutions. FRY and GERWE. Glycerophosphates etc. LETURC.—See XX.

PATENTS.

Contact sulphuric acid process. SELDEN Co., Assees. of A. O. JAEGER (B.P. 286,708, 28.1.28. U.S., 10.3.27).—A catalyst for the manufacture of sulphuric acid comprises a catalytically ineffective zeolite intimately admixed with a catalytically active diluent, e.g., colloidal platinum, or iron, silver, manganese, copper, cobalt, or nickel vanadate. The mixture is made while both constituents are in a gelatinous form, or one constituent may be precipitated on the other so that a coagulated gel is obtained which is mixed with a porous inert material and calcined for use. Of such catalysts, 12 examples are given. A. R. POWELL.

Manufacture of hydrogen chloride and sodium

sulphate. C. S. BENJAMIN, Assr. to GEN. CHEM. CO. (U.S.P. 1,693,217, 27.11.28. Appl., 17.5.27).—The moisture content of a mixture of sodium bisulphate and common salt to be calcined is regulated so that it is between the limits 2—3.5% by wt.

H. ROYAL-DAWSON.

Production of sodium bisulphate. J. N. CAROTHERS and C. F. BOOTH, Assrs. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,693,947, 4.12.28. Appl., 31.12.25).—Water and concentrated sulphuric acid are added to nitre cake to yield a product containing 85% of bisulphate, which is then heated to 150°, cooled to about atmospheric temperature, and disintegrated. H. ROYAL-DAWSON.

Production of iodates. F. A. HENGLEIN, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,693,441, 27.11.28. Appl., 19.4.27. Ger., 23.6.27).—An alkali iodide is brought into contact with oxygen in the presence of an alkali hydroxide at above 100° and under 1 atm.

H. ROYAL-DAWSON.

Manufacture of ammonium vanadate and vanadium pentoxide. J. A. WEIL, H. RAWLINSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 302,129, 3.7.28).—Ammonium vanadate is precipitated from an almost saturated solution of it by the addition, after filtration, of an ammonium salt which is decomposed at a comparatively low temperature with the formation of non-reducing gaseous products, e.g., ammonium nitrate. The precipitate is removed, washed with ammonium nitrate solution, and decomposed by heat, after incorporation with carriers etc., if desired.

L. A. COLES.

Production of pseudo-perphosphates. S. HUSAIN and J. R. PARTINGTON (B.P. 300,946, 20.8.27).—A soluble orthophosphate is dissolved in hydrogen peroxide solution, preferably a 30% solution, and the liquor is evaporated to dryness under reduced pressure in the presence of a dehydrating agent. The product contains hydrogen peroxide of crystallisation, for example, $K_2HPO_4 \cdot 2.5H_2O_2$ and $Na_2HPO_4 \cdot 2H_2O_2$.

A. R. POWELL.

Manufacture of raw calcium bisulphite digesting liquor. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,694,231, 4.12.28. Appl., 23.4.25).—A suspension of finely-divided limestone in water is passed in contact with, and counter-current to, sulphur dioxide through a confined mass of limestone rock. H. ROYAL-DAWSON.

Manufacture of materials of the nature of catalysts for the oxidation of oxidisable gases in admixture with air or oxygen. B. LAMBERT, and NAT. PROCESSES, LTD. (B.P. 301,853, 6.9.27).—Chromium hydroxide precipitated as a hydrogel, e.g., by the addition of cold, concentrated aqueous sodium hydroxide solution to cold 10% chromium nitrate solution, is washed free from soluble salts and treated with a dilute calcium, magnesium, nickel, cobalt, or manganese acetate solution so that it adsorbs the base of the salt. The product, which, after washing, filtering, and drying, contains up to 20% of the adsorbed base, is heated at about 350—400° in a current of air, and can then be used as a catalyst, e.g., in the oxidation of sulphur dioxide to trioxide.

L. A. COLES.

Production of metal carbonyls. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 301,778, 5.9.27).—Materials free from sulphur etc. and containing metals capable of forming carbonyls, or residues inert to carbon monoxide obtained in the production of carbonyls, are oxidised and then reduced with hydrogen, the processes being repeated if necessary, prior to treatment with carbon monoxide. L. A. COLES.

Manufacture of hydrogen. F. C. BLAKE, ASST. to LAZOTE INC. (U.S.P. 1,692,811, 27.11.28. Appl., 20.7.27).—Steam and carbon monoxide are subjected to the action of a non-ferrous catalyst in an apparatus the interior surfaces of which are made of non-ferrous material. H. ROYAL-DAWSON.

Continuous production of pure hydrogen. J. BELLAY (B.P. 284,262, 19.1.28. Belg., 26.1.27).—Water-gas from a gas producer is passed over baffle plates composed of fireclay and granulated charcoal on to which is projected a jet of superheated steam whereby the carbon monoxide is converted into carbon dioxide with the production of an equivalent quantity of hydrogen. The gas stream passes upwards through a tower containing milk of lime to remove carbon dioxide and dust, thence through a tube packed with soda-lime bricks heated at 800° to remove the last of the carbon monoxide. These bricks are made by slaking lime with a solution of sodium carbonate at 95°, screening out the fine material, and mixing it with lime water at 50° in a mould; after the vigorous reaction has subsided, the mass solidifies into bricks. After use in the purifier, the bricks are regenerated by heating at 850–900° until carbon dioxide ceases to be evolved. A. R. POWELL.

Manufacture of carbon dioxide snow. A. P. THURSTON. From DRYICE CORP. OF AMERICA (B.P. 302,070, 17.1.28).—Liquid carbon dioxide at a pressure of about 780 lb./in.² is cooled to about –18°, the pressure is increased to about 1100–1700 lb./in.², and the liquid is then passed through a cooler into a pressure reservoir, whence it escapes through a nozzle into an elongated expansion chamber in which the "snow" is deposited. The escaping gas before its return to the liquefier is used for the final cooling of the liquid carbon dioxide. L. A. COLES.

Utilisation of [solid] carbonic acid [carbon dioxide]. H. DEHOTTA (B.P. 301,764, 30.8.27).—Solid carbon dioxide is charged into pressure vessels capable of being hermetically sealed and constructed of heat-conducting material, so that, under the influence of the absorbed heat and the pressure generated in the vessel, the solid is converted partly into gas and partly into liquid. Outlets, leading into common discharge pipes where two or more of the vessels are used, are fitted at the top and bottom for drawing off the gas and liquid respectively. L. A. COLES.

Recovery of zinc from zinc [chloride]-bearing solutions. N. E. LENANDER, ASST. to ORKLA GRUBE-AKTIEBOLAG (U.S.P. 1,694,710, 11.12.28. Appl., 9.6.26. Norw., 22.5.25).—See B.P. 252,388; B., 1927, 555.

Purification of gases to be used in the production of ammonia. B. F. HALVORSEN, ASST. to NORSK HYDRO-ELEKTRISK KVAELSTOF-A./S. (U.S.P. 1,694,594,

11.12.28. Appl., 3.2.27. Norw., 25.2.26).—See B.P. 266,689; B., 1928, 191.

Production of hydrogen. F. GÜLKER (B.P. 275,273, 2.8.27. Ger., 30.7.26).—See G.P. 446,488; B., 1928, 816.

Heat-responsive material (U.S.P. 1,693,369). Impregnating brake bands etc. (B.P. 279,496).—See I. Vessels with acid-proof linings (B.P. 283,964).—See X. Fertiliser (B.P. 286,282). Fungicide (B.P. 276,033).—See XVI.

VIII.—GLASS; CERAMICS.

Weathering and durability of glass. K. UEDA (Rep. Asahi Glass Co. Res. Lab., 1927, No. 17, 39—55).—The surface of annealed glass is more durable than the interior. A cleaned glass containing SiO₂ 69.43, CaO 11.49, Na₂O 16.46, Fe₂O₃ + Al₂O₃ 1.46, MgO 0.66, SO₃ 0.63% was heated at 600° for 30 min. in a current of acidic gas or vapour, cleaned, and placed in moist air at 60° until permanently dimmed; relative times required for dimming were: untreated 1, air 3, steam 3, coal-gas combustion gases 4, sulphur dioxide 10, hydrogen sulphide 21, chlorine 21, hydrogen chloride 21, sulphuric acid 21, carbon dioxide 22, bromine 23, nitric oxide 25, nitric acid 25, sulphurous acid 35. Times required (days) after treatment with other reagents were: cold nitric acid (10 days) 2, cold fuming sulphuric acid (10 days) 2, boiling *N*-sodium carbonate (3 hrs.) 2, steam (100°, 1 hr.) 6, concentrated sulphuric acid (130°, 3 hrs.) 7, boiling 1% phosphoric acid (3 hrs.) 10, boiling water (3 hrs.) 13, boiling 5*N*-hydrochloric acid (3 hrs.) 14. The following dimming times (days) were observed after heating with the reagent (sealed tube) at 120° for 3 hrs.: carbon dioxide 1 (dry), 6 (moist); sulphur dioxide 2, 12; hydrogen chloride 4, 7; water 13. Progressive losses on boiling with water are recorded. Water penetrates to the interior of the glass on long boiling. CHEMICAL ABSTRACTS.

Ultra-violet transmission of a new window-glass substitute. R. H. CRIST (Ind. Eng. Chem., 1928, 20, 1367).—The transmission of ultra-violet rays through a window-glass substitute, Aldur—a formaldehyde-urea condensation product—showed values of 48% (254 mμ), 56% (270 mμ), 68% (313 mμ), 74% (365 mμ), 80% (436 mμ), and 93% (615 mμ). The material was 3 in. thick, and after exposure to a mercury-vapour lamp at a distance of 8 in. and at 65° the extent of solarisation was within the limits of experimental error, or around 2%. C. A. KING.

Copper ruby glaze. I. T. YOSHIOKA and S. HIRAOKA (J. Jap. Ceram. Assoc., 1927, 35, 608—614).—A study is made of the effect of atmosphere on the copper coloration in soda-silica glazes. Frits were heated for 1 hr. in a current of hydrogen, carbon monoxide, carbon dioxide, nitrogen, or oxygen at 600–1000°. Carbon dioxide and nitrogen are both suitable for the production of reds. Out of 5 types of frits tested, that composed of 0.25 Na₂O, 0.15 K₂O, 0.6 CaO, 0.25 Al₂O₃, 3.0 SiO₂, 0.25 B₂O₃, and containing 1% CuO together with 1% PbO, Fe₂O₃, SnO₂, or ZnO, gave good ruby-reds at 700–800°. The development of colour is best in the case

of tin oxide, and an atmosphere of carbon dioxide gives a brighter glaze than one of nitrogen. J. A. SUGDEN.

Titanium crystal glazes. S. KONDO (J. Jap. Ceram. Assoc., 1927, 35, 431—458).—The results of extensive tests on glazes applied to hard biscuit porcelain are recorded. Good frits containing soda, potash, zinc, and lime, magnesia, manganese, or iron are described which mature at cones 8 to 12. In general, the development is better in potash glazes. Whilst manganese and iron glazes are more crystalline than those containing lime and magnesia, the latter have a better colour and lustre. J. A. SUGDEN.

Cast iron for enamelling purposes. W. N. HARRISON, C. M. SAEGGER, JUN., and A. I. KRYNITSKY (J. Amer. Ceram. Soc., 1928, 11, 595—608).—The influence of the nature of the cast iron on the formation of blisters on the enamelled metal has been investigated. From preliminary experiments it was noted that, in preventing blisters, (1) variations in the enamelling practice greatly influenced the results; (2) no benefit was derived from the repeated melting of the iron, or from additions of sulphur or manganese to the metal to adjust the ratio of these two elements to a definite value; (3) relatively high pouring temperatures appeared to be beneficial. A new test, in which the enamel received a variety of firing treatments of different durations instead of a single one, was developed. Resistance to blistering was adjudicated on the basis of the extent of the range of firing treatments within which blister-free ware was produced. It was shown that the baking of enamels at 500° prior to firing reduced blister formation. Data indicated that the thin superficial layer of the metal was the most prolific source of blisters. The removal of this layer by pickling with 12% hydrochloric acid for 10 min. at 60° by sand-blasting or by machining is effective in eliminating blisters. A. T. GREEN.

Fibrous structure of porcelain bodies. R. SHIGEMUNE (J. Jap. Ceram. Assoc., 1927, 35, 615—621).—A continuation of a study of the fibrous microstructure of certain porcelains. A body containing felspar grains passing 200-mesh showed very little development of fibrous structure. Further experiments were carried out on three admixtures to 30% Hakuho orthoclase of 50—80-mesh, viz., 70% of Korean kaolin (body A), 70% of kaolin-quartz mixture corresponding to $\text{Al}_2\text{O}_3 \cdot 20\text{SiO}_2$ (body B), and 70% of chemically pure alumina (body C). Each of these mixtures was fired at cone 12 for 8, 24, and 67 hrs. The fibrous structure was most developed in body B fired for 67 hrs., and occurred in body A only when fired for 67 hrs., and not at all in body C. The author concludes that the fibrous crystals are probably those of leucite. Its formation depends on the physical nature of the matrix. Long heating and a siliceous matrix (as in body B) both tend to lower the viscosity of the mass and therefore to aid the formation of a fibrous crystalline structure. J. A. SUGDEN.

PATENTS.

Compound glass sheet and its production. CORNING GLASS WORKS, Assees. of W. C. TAYLOR (B.P. 280,196, 27.10.27. U.S. 6.11.26).—Two sheets of unground and unpolished glass are cemented to the opposite sides of a sheet of transparent material having approx-

imately the same refractive index, e.g., "pyralin," and the outer surfaces are subsequently ground and polished.

L. A. COLES.

Ovens or kilns for firing pottery and other ware. F. BENNION, H. J. PLANT, and J. B. CLARKE (B.P. 301,270, 26.4.28).—A circular kiln is heated by four or more fire-mouths, each of which is provided at the back with branch flues extending under the oven floor and communicating with flame distributors projecting inside the oven near the lining and a little above the floor. The kiln is also provided with a central vertical conduit, which may or may not be perforated, extending to or nearly to the crown of the kiln and communicating at its lower end with a passage in the oven floor leading to the chimney stack. The walls and crown of the kiln are lined with fireclay boxes or compartments filled with non-conducting material, e.g., diatomite. L. A. COLES.

Manufacture of glass containing barium. F. ROTHE and H. BRENEK, Assrs. to RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G. (U.S.P. 1,694,831, 11.11.28. Appl., 11.10.26. Ger., 16.10.25).—See B.P. 259,953; B., 1928, 779.

Manufacture of sintered magnesite brick. K. HARR (U.S.P. 1,694,540, 11.12.28. Appl., 10.11.23. Ger., 14.11.22).—See B.P. 207,172; B., 1924, 748.

IX.—BUILDING MATERIALS.

Tensile strength of mixtures of Portland and alumina cements. E. MIKI (J. Jap. Ceram. Assoc., 1927, 35, 622—623).—The setting time and tensile strength of mixtures of Portland cement with "Atlas lumnite" cement or "ciment fondu" have been studied. The early strength of a standard 1:3 mix is greatly reduced if 20% of the Portland cement is replaced by an alumina cement. No such injurious effect is found if 80% is replaced, and in the case of "Atlas lumnite" the high early strength is maintained at greater age. Mixtures containing 30—80% of Portland cement set too rapidly for ordinary purposes.

J. A. SUGDEN.

Early strength of cement. I. S. NAGAI (J. Jap. Ceram. Assoc., 1928, 36, 1—11).—Various Japanese Portland cements have been compared with two imported high early-strength cements, density, sieve, tensile, and compression tests being made. It is concluded that 80—90% of the tensile strength is developed in 18—24 hrs. in most of the cements; the increase in compressive strength is much slower; most of the Japanese cements have high early compressive strength, but are inferior to the imported cements in tensile strength; a 2-day test is more rational than a 3-day test. J. A. SUGDEN.

Tests of frost action on concrete. W. H. BATCHELDER (Eng. News-Rec., 1928, 101, 882).—Results of a series of freezing tests on concrete test bars showed that freezing immediately after casting is the worst condition which might occur, a loss of 31% in strength resulting, and in one case a fall of 69% when frozen after 1 day's set. When the concrete was protected for 7 days the loss in strength was reduced to 0 and 3.5% respectively. Concrete is not harmed greatly by frost after 24—72 hrs.' curing, if subsequently given adequate curing, which is,

however, delayed by the period during which the concrete was frozen.

C. A. KING.

Quicklime. ROGERS.—See VII.

PATENTS.

Extraction of saps, and the impregnation, staining, or drying of timber logs. H. J. C. FORRESTER. From SENTER & Co., G.M.B.H. (B.P. 301,244, 2.2.28).—Steam, gases, or solutions for dyeing, impregnating, etc. are introduced into holes or cuts between the open ends of felled tree trunks or below the open ends of standing trunks, arranged so that all the cells are severed and the liquid etc. is supplied to the entire cross-section of the wood; *e.g.*, two series of parallel holes are drilled in staggered relation, the holes being spaced from one another by their diameter. L. A. COLES.

[Concrete] mixing machines. J. F. BURN and J. LANCASTER (B.P. 299,979, 6.10.27 and 25.5.28).

Cakes of solid matter from fluids (B.P. 280,170).—See I. **Heating by submerged flames** (B.P. 300,819).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Calculation of carbon balance of metallurgical furnaces. R. D. PIKE (Ind. Eng. Chem., 1928, 20, 1356—1361).—A systematic method of determining the carbon balance of a furnace is indicated. The general scheme is to calculate carefully the carbon passing up the stack, based on Pitot tube measurement, stack area, and gas analysis at the Pitot tube station, and to equate this quantity to that introduced in the batch and fuel and also that in the gases which leak out of the furnace. Leakage is a considerable factor in fuel consumption, figures expressed as a percentage of the total flow being obtained as follows: secondary air outwardly from regenerators 23.4, outwardly from hearth 13.2, air inwardly to regenerators 21.9, inwardly through flue, valves, dampers, etc. 26.9.

C. A. KING.

Proposed new criteria of ductility [of metals] from a new law connecting the percentage elongation with size of test piece. D. A. OLIVER (Inst. Mech. Eng., 1929. Advance proof. 24 pp.).—Regarding the total elongation as made up of a general and a local extension, the total extension l and the original gauge length L are connected by the equation: $l = a + bL$, where a is a constant for the local extension. The value of a is approximately constant and independent of the cross-sectional area. The linear relation for each test piece confirms a law of the form kL^a , and if plotted logarithmically a straight line is obtained which indicates that the constant k itself follows the law: $k = \alpha A^\beta$, where α and β are new constants. It is proposed that constants a and α should be adopted as criteria for ductility as they are constant for any grade of material and practically independent of the type and size of test piece.

C. A. KING.

Corrosion and rusting of alloyed and plain cast iron. P. KÖRZSCHKE and E. PIVOVARSKI (Arch. Eisenhüttenw., 1928—9, 2, 333—340; Stahl u. Eisen, 1928,

48, 1716).—The distribution of graphite throughout grey cast iron, the amount of graphite present providing it is within the usual limits, and the physical characteristics of the graphite have no effect on the behaviour of the metal in corroding media. The resistance to corrosion by dilute hydrochloric acid increases with the silicon content to 1.5% Si, and the resistance to acetic acid increases with increasing silicon up to 3%; on the other hand, silicon greatly reduces the resistance of cast iron to alkaline solutions, hence for general purposes this constituent should be kept as low as possible. Nickel up to 6% increases the resistance to alkaline media, but has no effect in improving the acid-resisting properties of the metal. Cast iron with 0.5—1% Cr and 2.5% Ni resists corrosion by alkalis very well and by acids fairly well, but the addition of chromium introduces serious difficulties in working the metal. The rate of oxidation of cast iron in a moist atmosphere is decreased by about 25% by the addition of 0.3—0.4% Cu, but the resistance to acids is not improved by up to 0.9% Cu.

A. R. POWELL.

Mechanical properties of steel wire drawn at high temperatures in relation to the degree of reduction, the temperature of drawing, and the carbon content. A. POMP and W. KNACKSTEDT (Mitt. K.-W. Inst. Eisenforsch., 1928, 10, 117—174; Stahl u. Eisen, 1928, 48, 1705—1712).—The mechanical properties of soft iron (0.03% C) wire and of three mild-steel wires (0.35, 0.70, 0.84% C) have been determined after drawing to varying degrees of reduction at temperatures between 20° and 300°. The soft iron wires had a structure composed of medium-grained ferrite, whilst the structure of the steel wires was purely sorbitic. In all cases an increase in the drawing temperature up to 300° resulted in a considerable increase in the values obtained for the elastic limit, yield point, and breaking strain, but the iron wire rapidly became more brittle in torsion tests with rise in drawing temperature. High-temperature drawing (200—300°) is to be recommended, however, for the steel wires owing to the great increase produced in the elastic limit with only a very slight decrease in the torsion strength. The paper contains a number of graphs showing the results obtained for all the four wires tested together with a full discussion.

A. R. POWELL.

Theory of steel hardening. E. SCHEIL (Arch. Eisenhüttenw., 1928—9, 2, 375—388; Stahl u. Eisen, 1928, 48, 1776—1777).—On quenching a steel with 0.93% C the transformation from austenite to martensite is more complete the greater is the rate of cooling; it may be rendered still more complete by cooling below 0°. It is therefore assumed that tensile stresses accelerate the transformation whereas compressive stresses retard it; this would explain the further transformation that takes place below 0°, as the difference between the coefficients of expansion of austenite and martensite would cause the development of tensile stresses in the residual austenite. Annealing tests on a martensitic steel showed a transformation to take place at 100° and a second change at 300°, whereas the transformation point of austenite in the same steel was 250°. The transformation of austenite into pearlite can take place directly or through a series of intermediate steps;

above 300° direct transformation ensues on annealing, whereas below 250° the second form of martensite is first produced. A third possibility is that separation of carbide from the supersaturated austenite is the first phenomenon to be observed. Which of these changes takes place depends on the number of nuclei present and the rate of crystallisation of the various decomposition products.

A. R. POWELL.

Critical points of pure carbon steels. T. SATO (Tech. Rep. Tôhoku Imp. Univ., 1928, 8, 27—52).—The transformation points of pure electrolytic iron and of pure carbon steels containing up to 1.55% C have been determined by measurements of thermal expansion and by magnetic analysis. The equilibrium diagram of the iron-cementite system in the solid state is given.

C. W. GIBBY.

Copper steel with a high carbon content. A. F. STOGOV and W. S. MESSKIN (Arch. Eisenhüttenw., 1928—9, 2, 321—331; Stahl u. Eisen, 1928, 48, 1743—1744).—The effect on the magnetic and mechanical properties of steel with 0.7—1.2% C of the addition of 1—5% of copper has been determined. In all cases the coercivity and the product of the coercivity and remanence are increased more or less proportionally to the copper content, whereas the Ar₁ point is lowered and the temperature interval between the Ar₁ and Ac₁ points is widened. The tensile strength, hardness, and yield point of a copper steel in the annealed condition increase with increasing copper content, but the elongation, reduction in area, and impact strength reach a maximum with about 3% Cu. In the case of hardened and tempered copper steels the tensile strength increases with the copper content when the structure is hypereutectoidal, and decreases therewith when the structure is hypoeutectoidal. In the quenched and tempered state copper steels have a high yield point and ultimate strength combined with good elongation and reduction in area.

A. R. POWELL.

Recovery of apatite from the residual slimes [of phosphatic iron ores] by flotation. W. LUYKEN and E. BIERBRAUER (Arch. Eisenhüttenw., 1928—9, 2, 355—359; Stahl u. Eisen, 1928, 48, 1775—1776).—By flotation of the non-magnetic residue obtained in the magnetic separation of Swedish magnetite ores a yield of 75% of the phosphorus content was obtained in a concentrate assaying about 15% P, using about 1.8 kg./ton of sodium palmitate, provided that the tailings were dewatered and the water was used repeatedly in the circuit to avoid losses due to precipitation of the flotation agent by the lime in the water or derived from soluble calcium salts in the ore residue.

A. R. POWELL.

Determination of oxygen in iron and steel. O. MEYER (Z. angew. Chem., 1928, 41, 1273—1276, 1295—1298).—A comprehensive review of recent work on this problem.

A. R. POWELL.

Rapid determination of silicon in iron-silicon alloys by density measurements. W. DENECKE (Giesserei-Ztg., 1928, 25, 304—306; Chem. Zentr., 1928, ii, 275).—Schlumberger's method is applied to iron-silicon alloys containing smaller percentages of

silicon, benzene being used as volumeter liquid. Alloys containing less than 21% Si gave a remarkable distribution of results.

A. A. ELDRIDGE.

Age-hardening of beryllium-copper alloys followed by X-ray examination. O. DAHL, E. HOLM, and G. MASING (Z. Metallk., 1928, 20, 431—433).—The α -phase of beryllium-copper alloys is cubic face-centred whilst the β -phase is body-centred; the alloy containing 2.5% Be shows only the α -diffraction pattern in röntgenograms taken after quenching from 800°. Subsequent annealing at 350° produces traces of γ in 10 min. and considerable separation of γ in 6 hrs. At 150° no γ is detectable after 243 hrs., but the lines of the α -structure become somewhat blurred after only 4 hrs. at 150° and more distinctly so at 200° after a very much shorter period. These results indicate that highly dispersed γ separates at temperatures below 300° in a few hours, whereas at higher temperatures the precipitated particles are much coarser. During the separation of the highly dispersed γ , the electrical conductivity falls rapidly and the hardness and tensile strength increase greatly owing to the high internal stresses set up by the lattice distortion accompanying the decomposition of the α -phase.

A. R. POWELL.

Changes of length and of the modulus of elasticity of beryllium-copper alloys during age-hardening. O. DAHL and C. HAASE (Z. Metallk., 1928, 20, 433—436).—The length of a rod of a beryllium-copper alloy with 2.5% Be quenched from 800° increases linearly with the temperature up to 150°. With further rise of temperature the hardness begins to increase and the rate of expansion to diminish until at 280° the rod begins to contract. At 300° another small expansion takes place, but at 350° another contraction occurs if the rod is maintained at this temperature for 1 hr. On cooling again contraction ensues linearly. Annealing the quenched alloy at 250° causes a contraction over a period of 8 hrs., the curve of which is similar to that showing the increase in hardness over the same period; the electrical conductivity falls rapidly to a minimum in 1½ hrs., then increases linearly with time of annealing. Ageing at 400° causes a rapid increase in the torsion modulus and electrical conductivity during 1½ hrs., followed by a very slow increase for a further 16 hrs.; at 150°, however, the conductivity decreases with time of ageing and the torsion modulus slowly rises. These results are considered to support the precipitation theory of age-hardening.

A. R. POWELL.

Effect of inhibitors on the acid dissolution of copper and copper alloys. H. O. FORREST, J. K. ROBERTS, and B. E. ROETHLI (Ind. Eng. Chem., 1928, 20, 1369—1371).—Evolution of hydrogen from copper is quite slow in dilute acids at 75°, but increases rapidly at concentrations of more than 23% HCl. The attack is slow at atmospheric temperature even in concentrated solutions. Inhibitors are not very effective until a high acid concentration is reached, and the rate of corrosion is decreased probably when depolarisation due to molecular hydrogen becomes sufficiently great. The requisite quantity of inhibitor is that which is sufficient to form a film of the inhibitor substance or of reaction products

on the metal, thereby decreasing the area effective for depolarisation; greater quantities are of little value.

C. A. KING.

Absorbability of gases in casting copper and effect of adding cuprosilicon. O. W. ELLIS (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 123, 26 pp.).—Water vapour has a high solubility in copper, and causes formation of oxide. Silicon reduces cuprous oxide, and then removes gaseous oxygen. Copper melted in a reducing atmosphere contained carbon dioxide, nitrogen, and water, but practically no cuprous oxide. Oxidised copper in contact with charcoal absorbed carbon as mon- and di-oxides up to 21.9% of gas. The ratio of occluded di- to mon-oxide increased with rise of temperature, but the occluded gas never contained more than 80.5% by wt. of dioxide. Hydrogen was present in only small quantities, except when oil was used for melting, when it was produced possibly by interaction of cuprous oxide and methane, although carbon dioxide, carbon monoxide, and water are the chief gases occluded.

CHEMICAL ABSTRACTS.

Effect of cadmium on mechanical properties of brass. W. BANNAU (Amer. Met. Market, 1928, 35, 12—14).—Up to 0.5% Cd the structure of 70% and 60% copper-zinc alloys is unaffected; with 1—4.5% Cd, free cadmium is present. Lessened resistance to shock and low elongation are observed. In 55% Cu brasses 3.6% Cd does not diminish the tensile strength, but 1.8% Cd affects the ductility and brittleness.

CHEMICAL ABSTRACTS.

Effect of heat treatment on some mechanical properties of 86:4:6:3:1 copper-nickel-tin-zinc-lead alloy. R. J. ANDERSON (Amer. Met. Market, 1928, 35, 1—3, 14).—The alloy shows wide variations in hardness for a given treatment; it is evidently of the solid solution type.

CHEMICAL ABSTRACTS.

Cadmium as corrosion preventive for light metals. J. DORNAUF (Korrosion u. Metallschutz, 1928, 4, 97—102; Chem. Zentr., 1928, ii, 109).—Cadmium is a satisfactory corrosion preventive for aluminium alloys. Only small potentials are set up between aluminium and cadmium, the latter being the electrode attacked. Cadmium protects aluminium from the action of solutions of mercury salts; it also exerts a protective action on magnesium alloys.

A. A. ELDRIDGE.

Mechanism of rolling, hammering, and drawing zinc and cadmium. G. MASING (Z. Metallk., 1928, 20, 425—427).—The coefficient of linear expansion of zinc and cadmium rods and wires is increased very considerably in a longitudinal direction by rolling, swaging, or drawing. This is ascribed to the formation of twinned crystals which commences to take place simultaneously with the commencement of cold work and continues throughout the working.

A. R. POWELL.

Tensile properties of crystals of aluminium alloys which undergo age-hardening. G. SACHS (Z. Metallk., 1928, 20, 428—430).—Single crystals of 5% copper-aluminium alloy were prepared by annealing drawn wires at 525° for 6 hrs., then at 300° for 1 hr., stretching them 1.5%, and annealing for 6 days at 450—515°. After quenching in cold water and ageing at 100° for 30 min., tensile strengths of 33—45 kg./mm.²

were obtained without reducing the normal ductility. X-Ray examination by the Laue method gave similar patterns for the annealed and age-hardened wires, and both broke along a plane at an angle of 45° to the wire axis when pulled in a tensile machine. The age-hardened wires recrystallised at a lower temperature than annealed wires, and the new crystals produced were larger, otherwise no difference between the two could be detected.

A. R. POWELL.

Gold alloy "750/2." Behaviour after cold-drawing and heating. W. HEIKE and F. WESTERHOLT (Z. anorg. Chem., 1928, 176, 200—204).—The mechanical properties of the alloy of composition 75% Au, 4% Ag, 21% Cu have been studied with special reference to the time and temperature of heating and rate of cooling.

H. F. GILLBE.

Tantalum as a constructional material for chemical apparatus. F. HEINRICH and F. PETZOLD (Chem. Fabr., 1928, 689—691).—The inertness of tantalum towards corrosive solutions, with the exception of those containing hydrofluoric acid or the caustic alkalis, renders the metal particularly valuable for the construction of chemical apparatus, especially such as is required to withstand the action of *aqua regia* or hydrochloric acid. The metal is not attacked by mercury or any salt solution, but bromine dissolves it slowly and fused salts readily destroy it. Tantalum vessels should not be heated above 300° in any gas as rapid embrittlement ensues especially in gas mixtures containing hydrogen or nitrogen.

A. R. POWELL.

Monel metal as tower packing. G. WEISSENBARGER and L. PIATTI (Chem. Fabr., 1928, 703—704).—Monel metal and aluminium gauzes were treated for 1 hr. at 100° with 5% solutions of sulphuric, hydrochloric, acetic, and nitric acids. The loss of the monel metal gauzes did not exceed 0.11% with the first three; with nitric acid it was 2.13%. Aluminium was as resistant to nitric and acetic acids as monel metal, but was attacked by sulphuric acid and particularly by hydrochloric acid.

C. IRWIN.

Nickel anodes and the acidity of the solution. R. G. SUMAN (Metal Ind., 1928, 26, 350).—For nickel plating, a solution of p_H 6.1 is most efficient. The solution used contained 255 g. of single nickel salts and 19.8 g. of ammonium chloride in 3.8 litres of water. The reaction varied with the composition of the anodes.

CHEMICAL ABSTRACTS.

Factors affecting the relative potentials of tin and iron. E. F. KOHMAN and N. H. SANBORN (Ind. Eng. Chem., 1928, 20, 1373—1377).—The electrochemical relation between tin and iron is influenced considerably by the presence of organic acids such as are present in canned fruit. At concentrations equivalent to 0.75% of malic acid, tin is distinctly cathodic in acetic, malonic, and succinic acids, only slightly cathodic in malic acid, and distinctly anodic to iron in citric acid. Addition of apple pomace to acetic, malonic, and succinic acids reverses the condition and the tin becomes anodic, a further protection to iron being also induced by the presence of tin in solution, which raises the cathodic polarisation on iron. In hydrochloric and sulphuric acids of concentrations ranging from 0.05 to

20%, tin becomes increasingly more anodic to iron with increasing concentration of acid even when the metals are not in electrical contact, and corrosion of tin is very greatly increased with metallic contact, due to galvanic action. The potential conditions are influenced chiefly by the hydrogen-ion concentration and negligibly by the conductivity of the solution. The results agree with those of commercial practice in which the more acid fruits produce little perforation of the container, although less acid fruits, *e.g.*, black cherries, in the juice of which tin is only mildly anodic to iron, cannot be canned successfully because of the hazards of corrosion.

C. A. KING.

Influence of bismuth on the mechanical properties of lead. O. BAUER (Giesserei-Ztg., 1928, 25, 297—299; Chem. Zentr., 1928, ii, 286).

Nickel catalyst. BAG.—See II. **Lead pigments for iron protection.** EIBNER and LAUFENBERG.—See XIII.

PATENTS.

Apparatus for the concentration of graphite and other ores by flotation. J. F. M. R. DE ROBILLARD (B.P. 275,673, 9.8.27. Fr., 9.8.26).—The sides of the flotation cell are provided with a number of small apertures covered with fine wire gauze, on to which streams of water are directed from jets of smaller cross-section than that of the apertures so as to cause bubbles of air to be drawn into the cell by the injector action of the water.

A. R. POWELL.

Manufacture of [steel] containers for compressed gases. H. E. STÜRCKE (U.S.P. 1,692,521, 20.11.28. Appl., 8.5.24).—Fully annealed mild steel billets are formed into the required shape of container, these are annealed again, subjected to internal pressure corresponding with the ultimate test pressure, and then heat-treated to remove the mechanical stresses set up by this treatment.

A. R. POWELL.

Separate production of iron, nickel, cobalt, or other metals which form carbonyls, from mixtures containing several such metals. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,099, 25.6.27).—The product from which the metals are to be extracted is treated in a suitable way to obtain the free metals in a finely-divided form, the metals are converted into carbonyls, and the mixture of carbonyls is fractionally distilled in an atmosphere of carbon monoxide. *E.g.*, chloanthite is heated at 550° in air and steam to expel sulphur and arsenic, and the oxides are reduced at 450° in hydrogen. The product is treated at 140° with carbon monoxide under a pressure of 180 atm., the evolved gases are condensed at —20°, and the product is fractionally distilled, giving first nickel then iron carbonyl. The residual cobalt compound is purified by heating it at 150° under 200 atm. in carbon monoxide, and subsequently subliming the cobalt tetracarbonyl. [Stat. ref.]

A. R. POWELL.

Protection of articles made of brass and like alloys against discoloration. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 289,441, 25.4.28. Ger., 27.4.27).—Articles are heated, preferably at 500—600°, in a current of reducing gases, *e.g.*, a mixture

containing 80% of nitrogen and 20% of hydrogen, after being cleaned by pickling.

J. S. G. THOMAS.

Reduction of zinciferous materials. L. MELLERSH-JACKSON. From NEW JERSEY ZINC CO. (B.P. 300,519, 8.8.27).—Agglomerates made by carbonising briquettes of fine oxidised zinc ore and a coking coal bonded with sodium carbonate as an accelerator of reduction are passed through a long, narrow, horizontal, reducing furnace on a moving hearth containing a bed of fine ore on which is a layer of coarser ore. The charge is previously preheated in a recuperator or the coked briquettes may be discharged directly from the coking furnace to the hearth of the reducing furnace. The waste gases from the recuperator are utilised for heating the preheater or in the coking furnace.

A. R. POWELL.

Manufacture of aluminium alloys. A. GEYER (B.P. 284,722, 3.2.28. Fr., 4.2.27).—Cuprosilicon, cupromanganese, and/or iron are added to a bath of molten aluminium at 800—1100° under a layer of coal or other carbonaceous material, a small quantity of wet coal dust is sprinkled on the surface, the bath is heated to 1200° and stirred, then further aluminium is added to lower the temperature to 700°, and the metal is poured as usual. The hydrogen liberated from the wet coal dust is stated to cause a more thorough mixing of the constituents under highly reducing conditions.

A. R. POWELL.

Refining of metals [silver] and alloys. A. PACZ (U.S.P. 1,691,207, 13.11.28. Appl., 11.5.21. Renewed 1.3.26).—The structure of silver alloys is refined by stirring the molten alloy beneath a flux containing sodium fluoride.

A. R. POWELL.

Vessels having an acid-proof lining. I. G. FARBENIND. A.-G. (B.P. 283,964, 20.1.28. Ger., 22.1.27).—The lining consists of cast silicon slabs.

H. ROYAL-DAWSON.

Electrolytic production of light metals. P. L. HULIN (B.P. 284,678, 3.2.28. Fr., 3.2.27).—In a cell for the electrolysis of fused chlorides, the cathode is composed of a number of steel rods dipping into the fused salt and provided with water-cooled copper heads or holders.

A. R. POWELL.

Apparatus for electrolytically producing metal alloys. E. KELSEN (B.P. 296,060, 22.8.28. Austr., 24.8.27).—A cell for the deposition of metal alloys on a rotating cylindrical cathode comprises two parallel, rectangular electrolyte compartments separated from one another by a small gap and each containing one anode and one electrolyte adapted to deposit one of the constituents of the alloy. The cathode rotates between the two cells so that the metals are deposited on it alternately in very thin layers. Means are provided for burnishing the cathode as it rotates between the cells and for catching the leakage from each cell separately, purifying it, and returning it to its appropriate cell.

A. R. POWELL.

Mills (U.S.P. 1,690,493—7).—See I.

XI.—ELECTROTECHNICS.

Recommended equipment of a modern X-ray laboratory for the study of structures of materials. G. L. CLARK (Ind. Eng. Chem., 1928, 20, 1386—1390).—A

description is given of the various types of X-ray apparatus installed in the laboratories of the University of Illinois, including a new type of cassette for the pinhole and oscillation methods to be used with a multiple-diffraction apparatus; the design and arrangement of gas-type, copper-target tubes provided with finely-regulated leakage to adjust the internal air pressure; together with manipulative details and typical diffraction patterns obtained from industrial materials.

C. A. KING.

Insulating oils. BUTKOV. — See II. **Colour measurements.** DESBLEDS. — See VI. **Copper steel.** SROGOV and MESSKIN. — See X.

PATENTS.

[Crucible] **electric induction furnace.** E. F. NORTHROP (B.P. 301,954, 8.9.27).—The effect of stray magnetic fields about a furnace inductor coil free from inter-penetration of transformer iron is reduced by providing an easier return path for the magnetic flux than is afforded by the air, and by surrounding this path with a magnetising winding. J. S. G. THOMAS.

High-frequency induction furnaces. SIEMENS & HALSKE A.-G. (B.P. 291,774, 27.9.27. Ger., 10.6.27).—A primary winding is divided into two or more insulated bands set on edge and so thin that the skin effect is negligible; they are so arranged that the several windings are equal in respect of resistance, impedance and inductive action on the inside of the furnace.

J. S. G. THOMAS.

Electrodes for use in arc welding. IMPERIAL CHEM. INDUSTRIES, LTD., and J. H. PATERSON (B.P. 302,064, 30.12.27).—Strands of flux, e.g., asbestos fibre soaked in sodium carbonate solution, are arranged between a number of parallel wires welded together at intervals.

J. S. G. THOMAS.

Manufacture of gas-filled electric incandescence lamps. GEN. ELECTRIC CO., LTD., and F. J. G. VAN DEN BOSCH (B.P. 301,143, 17.9.27).—For purposes of flashing, the gas-filling, preferably a mixture of argon and nitrogen with a little hydrogen, is mixed with the vapour of white phosphorus heated above 50°.

J. S. G. THOMAS.

Negative electrode for electric accumulators. A. POUCHAIN (B.P. 282,449, 19.12.27. Ger., 20.12.26).

[Electrode holder for] **electric arc-welding, cutting, or soldering.** ALLOY WELDING PROCESSES, LTD., E. H. JONES, and E. J. CLARKE (B.P. 301,660, 2.1. and 11.1.28).

Detection of suspended matter in fluids (B.P. 272,914). **Determining the contents of a gas** (U.S.P. 1,691,138).—See I. **Light metals** (B.P. 284,678.) **Metal alloys** (B.P. 296,060).—See X.

XII.—FATS; OILS; WAXES.

Absorption spectra and fluorescence of fats. W. SPROESSER (Chem. Umschau, 1928, 35, 325—327).—Weak absorption maxima were found in the absorption spectra of cacao butter (between 270 and 280 $\mu\mu$) and some other vegetable butters in chloroform solution. Suggestions are made for the technical photometric

examination of the fluorescence of fats by comparison with a standard object, with the help of colour filters to limit the range of light transmission.

E. LEWKOWITSCH.

Sham cola nuts. D. VAN OS and Y. VAN DER WAL (Pharm. Weekblad, 1928, 65, 1266—1271).—Nuts from a consignment purchased as cola nuts, found to contain no caffeine, but 38% of fat, and differing in structure and physical properties greatly from the true product, were identified as seeds of *Bassia Parkii* (shea nuts).

S. I. LEVY.

Fat from the liver of the sperm whale. M. TSUJIMOTO and K. KIMURA (Chem. Umschau, 1928, 35, 317—318).—By ether extraction of sperm-whale liver, 6.3% of a yellow-brown semi-solid fatty material was obtained, yielding on saponification 69.3% of fatty acids and 19.3% of unsaponifiable matter. The fatty acids formed a yellow-brown, semi-solid crystalline mass with m.p. 32—33.5°, d_4^{20} 0.8877, neutralisation value 194.1, iodine value 141.3, n_D^{20} 1.4610, ether-insoluble bromides 38.5% (blackened without melting at 250°, bromine content 70.47%). The lead salt-ether separation yielded about 75.4% of liquid acids (neutralisation value 188.9, iodine value 175.5, n_D^{20} 1.4720), and about 24.6% of solid acids (m.p. 52—53°, neutralisation value 209.5, iodine value 11.8). By the lithium salt-acetone method there were obtained 23.4% of the highly unsaturated acids with neutralisation value 170, iodine value 311, and n_D^{20} 1.4923. The unsaponifiable matter was an orange-yellow, viscous, semi-solid material, iodine value 260, which gave colour reactions reminiscent of vitamin-A; it contained 48% of cholesterol, and the authors further succeeded in isolating a new unsaturated hydrocarbon ($d_4^{21.5}$ 0.8981, $n_D^{21.5}$ 1.5110, iodine value 379.5, mol. wt. 501) to which they assign the probable formula $C_{35}H_{60}$.

E. LEWKOWITSCH.

Physical properties of pure triglycerides. R. B. JOGLEKAR and H. E. WATSON (J.S.C.I., 1928, 47, 365—368 r).—The physical properties of the synthetic glycerides of capric, lauric, myristic, palmitic, and stearic acids have been ascertained; the examination showed that the m.p. and refractive index afforded little criterion as to purity, but the densities, solidifying points, and especially the viscosities varied appreciably even when a fair degree of purity had been attained. Curves for the solidifying points of mixtures of tripalmitin and tristearin were plotted; for mixtures with from 25 to 50% of tristearin points were obtained characteristic of the two modifications (double m.p.) of this glyceride. The authors were unable to confirm Pascal's values (B., 1914, 602) for the refractive indices of mixtures of tripalmitin and tristearin, but found a linear relation between n_D and composition between 70° and 80°.

E. LEWKOWITSCH.

Swelling [absorption] phenomena of aluminium fatty acid [soaps] in various solvents. I.—III. E. MARKOWICZ (Farben-Ztg., 1928, 34, 326—328, 414—418, 503—505).—I. When dibasic aluminium palmitate, largely used as a flattening medium for paints, is stirred with certain organic media it gradually absorbs the solvent, swelling to form pastes or gels, the character of which depends on the solvent employed. The solvents

are classified under (1) aliphatic hydrocarbons (benzine, white spirit) yielding opaque soft pastes which are easily spread or painted out; (2) aromatic and chlorinated hydrocarbons from which strongly swelling, jelly-like, translucent elastic gels are produced, and (3) crude turpentine etc., in which aluminium palmitate swells to a translucent non-elastic, ropy gel. On drying in air, the pastes with aliphatic solvents become more elastic, but do not flow, and ultimately dry to hard crusts, the process resembling generally the drying of a highly concentrated suspensoid. Benzene gels retain the solvent for very considerable periods; with thick masses a skin is formed and finally a similar horny mass is produced. Terpene gels are sharply distinguished in that the mass flows and retains its tenacious nature for several weeks (cf. aluminium linoleate); no skin-formation or shrinking was observable, although after long exposure the ductility is reduced. In the absence of air the viscosity of the aromatic and terpene gels, which is a maximum shortly after mixing, decreases gradually to a limiting value; in the former case no further change occurs, but terpene gels become more homogeneous, and in greater dilutions may separate into gel and sol phases. The aliphatic pastes, however, are never stable, but always pass after a time into the gel condition, although remaining turbid. The fully dried crusts or gels may be made to swell again in the original or another solvent.

II. The reactions of aluminium palmitate with 25 different dispersion media are discussed in detail. No swelling occurs in water, alcohol or acetone, dioxan or aniline (liquids of high dielectric constant), nor in viscous fluids such as linseed oil or paraffin oil. The other solvents, in which marked absorption occurs, are examples of the groups considered in part I. Intermediate types are represented by decalin and amyl acetate. Measurements were made of the changes of viscosity and time of setting depending on the concentration of the solvent; the decrease of viscosity with time is greatest with the chlorinated hydrocarbons and least marked in the case of turpentine. With the aliphatic pastes the solidity, absorption, and transparency were found to vary with the b.p. of the solvent; with terpene gels the form of the product depends on the age, origin, and purity of the solvent.

III. The difficulties encountered in the technical application of aluminium palmitate to matt varnishes etc. are discussed: care must be taken in the choice of solvent in order to avoid gelatinisation, and the fully-saturated paste should be incorporated as soon as possible. A greater flattening effect is produced with lower-boiling solvents, of which greater absorption occurs. Amyl acetate is the most suitable dispersion medium for use with nitrocellulose lacquers, whilst for waterproofing textiles, for which a high concentration is required, benzene or carbon tetrachloride is recommended. If an impervious, continuous coating is desired, solutions in turpentine or decalin may be used.

E. LEWKOWITSCH.

Drying process of fatty oils. H. WOLFF (Chem. Umschau, 1928, 35, 313—317).—Following a discussion of various theories of the drying of oil films, the author describes experiments on the influence of the base

employed (zinc, iron, glass) on the drying curves (increase in weight/time) of tung oil, linseed oil, and boiled oils. It is confirmed that an actual decrease in the weight of the drying film may occasionally be observed at the beginning of the drying process. E. LEWKOWITSCH.

Determination of unsaponifiable matter in oils and fats. E. L. SMITH (Analyst, 1928, 53, 632—641).—A method for determination of unsaponifiable matter in oils and fats, claimed to be accurate to within 1% of the amount present, consists in adding 40 c.c. of alcohol and 10 c.c. of 40% aqueous sodium hydroxide to 20 g. of oil, and boiling for 1 hr. under reflux. The solution is then transferred to a separating funnel with 150 c.c. of water, shaken with 300 c.c. of redistilled ether, the soap solution extracted twice more with 250 c.c. of ether, the combined extracts washed with 20 c.c. of water, and the bulk of the ether removed. The soap solution is extracted three times more with 250 c.c. portions of ether, the extracts are washed, and the bulk is reduced in the same flask as the first extract. The extract is then rinsed with ether into a separating funnel, and 50—75 c.c. of ether in another funnel are used for extracting all the wash liquors from the first separator. The extract is washed twice with water, then with 2*N*-sodium hydroxide in 10% alcohol, followed by two more water washings, and the whole repeated at least once. Finally it is washed with 0.5*N*-hydrochloric acid and then with water to wash out alcohol from the ether layer. The ethereal extracts are evaporated and the residue is weighed, mixed with 10 c.c. of neutralised alcohol, warmed, and titrated with 0.025*N*-sodium hydroxide (1 c.c. = 7 mg. of fatty acid, as oleic acid). A shorter method involves only four extractions. The first three extracts are united, treated with 20 c.c. portions of wash liquors, using the fourth extract to re-extract the washings.

D. G. HEWER.

Determination of the hydrogen value of unsaturated compounds. H. I. WATERMAN, J. N. J. PERQUIN, and H. A. VAN WESTEN (J.S.C.I., 1928, 47, 363—365 T).—A new method for the determination of the hydrogen value of unsaturated compounds, especially suitable for substances having high vapour pressures, is described in detail. Hydrogenation is performed in the presence of a catalyst, and the volume of gas absorbed is measured directly.

E. LEWKOWITSCH.

High-vacuum grease. F. HEINRICH and F. PETZOLD (Z. angew. Chem., 1928, 41, 692—693).—The value of nine varieties of grease for lubricating stopcocks of apparatus in which it is desired to maintain a high vacuum has been determined, and the results are reproduced in a series of graphs showing on a logarithmic scale the increase of internal pressure with the time.

A. R. POWELL.

Action of air on nickel catalyst for oil hydrogenation. A. BAG (Masloboino-Zhir. Delo, 1928, No. 5, 6).—The loss of activity of a nickel catalyst cooled in carbon dioxide, when exposed to air for one month, is small.

CHEMICAL ABSTRACTS.

Film characteristics of the esters of the component fatty acids of linseed oil. B. H. THURMANN and W. R. CRANDALL (Ind. Eng. Chem., 1928, 20,

1390—1392).—The behaviour of films of the ethyl esters of the fatty acids of both linseed oil and partially hydrogenated linseed oil—ethyl oleate and stearate—singly and in admixture, has been examined. Nitro-cellulose lacquer was used as a fixative and the films, on muslin and tinned iron, were examined frequently over a period of 10 months. The esters of the less unsaturated fatty acids such as oleic acid are very stable in films, but the esters of the more unsaturated acids are not so stable. They rapidly become sticky, odorous, and dark-coloured, and it is therefore necessary to compare films of oleic derivatives or substances having a similar range of iodine value obtained by polymerisation or hydrogenation of linseed oil.

E. H. SHARPLES.

Composition of Chinese wood oil (tung oil). A. STEGER and J. VAN LOON (J.S.C.I., 1928, 47, 361—363 r).—The authors summarise the literature on the constitution of tung oil, and give an account of the preliminary analysis of a typical wood oil, which they believe to consist principally of the glyceride of an isomeride of linolenic acid. Practically only fatty acids with an 18-carbon straight chain are present; the saturated fatty acids appear to consist of a mixture of 75% of palmitic and 25% of stearic acids.

E. LEWKOWITSCH.

Baudouin's sesamé oil reaction. H. HELLER (Allg. Oel- Fett-Ztg., 1926, 25, 315—316; Chem. Zentr., 1928, ii, 301).—Baudouin's test with hydrochloric acid, and the test with furfuraldehyde and hydrochloric acid, are not so trustworthy as is Soltsien's test.

A. A. ELDRIDGE.

Determination of adsorptive power of bleaching earths used to decolorise oils. A. WIBERG (Z. angew. Chem., 1928, 41, 1338—1342).—The bleaching effect on soya-bean and coconut oils of decolorising earths under varying conditions was studied by measuring the intensity of the colour remaining after treatment in a simple apparatus. It is remarkable that the adsorption of the colour increases with increased temperature of reaction (up to 98°). No advantage was gained by increasing the rate of stirring above 200 r.p.m. In order to obtain comparative results, it is essential that adsorption equilibrium be reached (a condition attained in the tests recorded within 15 min. at 98° and 200 r.p.m.).

E. LEWKOWITSCH.

Transformations of fatty acids. STADNIKOV and others. **Fatty acids in petroleum.** TANAKA and KUWATA. **Miscibility of castor oil with hydrocarbons.** TABER and STEVENS. **Oil testing.** FRIEDBACH.—See II. **Availability of nitrogen of oil cakes.** AYYAR.—See XVI. **Irish butter.** ARUP. **Vitamin-D in butter and margarine.** FLURY.—See XIX. **Digestion of sewage grease.** NEAVE and BUSWELL.—See XXIII.

PATENTS.

Apparatus for [solvent] extraction of oils, fats, etc. from substances containing the same. G. W. BRADSHAW and E. R. JACKSON (B.P. 298,675, 15.7.27).—A valve mechanism to be placed between the solvent extractor and the vessel used for solvent recovery from the meal is described. The inlet to the latter vessel stands up above the floor of the valve casing and con-

stitutes the seating of the valve, which consists of a horizontal sliding hollow casing, closed by a perforated plate through which the solution may be drained from the extractor.

E. LEWKOWITSCH.

Lacquers etc. (B.P. 301,133).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oil absorption in paint grinding. A. EIBNER (Farben-Ztg., 1928, 34, 725—729).—A summary of recent investigations on the oil absorption of pigments, special reference being made to the work of Wolff in this field. The modern hypothesis that equal volumes of pigments require approximately equal volumes of oil to produce the "adhesion-cohesion equilibria" constituting paste paints is discussed fully. The very wide variation in oil absorptions based on equal weights of pigments differing appreciably in sp. gr. has obscured the position in the past. Other factors receiving consideration are the relation between oil content of oil pastes and the amounts of oil necessary to convert such pastes into ready mixed paints, and the influence of structure and surface properties of various pigments on oil absorption.

S. S. WOOLF.

Distribution of pigment in paint films. H. WOLFF (Farben-Ztg., 1928, 34, 669—670).—Dry films of white lead- and zinc oxide-oil paints were rubbed down under controlled conditions so that the films were removed in ten equal layers. The pigment content of each of these was determined, and in the case of the white lead paint films the "ether-soluble lead" was also determined in each fraction. It is established that a concentration of pigment occurs in the lowest layers, and in the case of white lead in the uppermost layers also. There is no parallelism between the extent of formation of "lead soaps" and the concentration of lead pigment in a particular layer. The reactions between white lead and linseed oil are speculatively discussed.

S. S. WOOLF.

Decomposition of oil paints by bacteria and fungi. W. VAUBEL (Farben-Ztg., 1928, 34, 505).—The observations of Haag (Arch. Hygiene, 1928, 100) and D'Ans (B., 1928, 935) on the action of fungi etc. on fatty acids, oils, and paints are discussed. E. LEWKOWITSCH.

Types of red lead. A. JUNK (Farben-Ztg., 1928, 34, 671—676).—A number of samples of red lead, both of the "old type" and of the modern "disperse" (or finely-divided, non-hardening) type, have been comprehensively examined. Features of the modern type are low sp. gr., high content of "true red lead" (32% being the minimum possible content of lead peroxide), average particle size of 2.5 μ with no particles greater than 4 μ , relatively high bulking value, oil absorption to give paint consistency 25% (approx.) as against 15% for old-type red lead, and non-hardening on storage. The modern material settles out progressively, but can readily be brought back to normal consistency, whereas the older type reaches an equilibrium in its settling, due to thickening of the vehicle caused by interaction between the "free" litharge in the red lead with the oil and later with liberated glycerol, these reactions leading eventually to the formation of a hard,

cement-like mass. The modern type in paint form gives thinner films and greater spreading power than the old type, but the film is discontinuous and has less protective value than the latter. As an accelerated test, the prepared red lead paints were heated for 4 hrs. at 80°, the results being in satisfactory agreement with period tests. S. S. WOOLF.

Determination of free metallic lead in litharge and red lead. P. FLUCH (Z. anal. Chem., 1928, **75**, 371—390).—The sample (5 g.) is dissolved in 2.5 c.c. of glacial acetic acid or in 30 c.c. of 80% acetic acid containing 0.5—0.6 g. of hydrazine sulphate. In the first case the insoluble residue consists of the free lead and any lead peroxide, and in the second of free lead and lead sulphate together with insoluble impurities. The residue is collected, washed with glacial acetic acid or ammonium acetate, then with hot water, and digested with nitric acid. The resulting nitrate solution is analysed for lead by any suitable method. For highly dispersed preparations of litharge a saturated lead acetate solution may be used as the solvent for the lead oxide.

A. R. POWELL.

Value of lead pigments in iron protection. A. EIBNER and W. LAUFENBERG (Korrosion u. Metallschutz, 1928, **4**, 107—110; Chem. Zentr., 1928, ii, 108—109).—The imperviousness to water of a film of linseed oil increases with its content of lead soap and lead-glycerol compounds; hence lead pigments selected for paints should be those which are able to form the largest quantities of lead soaps. The lead-linseed oil compounds could not be dissolved by treatment with benzene under pressure. The lead film aggregate is an irreversible colloid. The protective action of red lead is examined.

A. A. ELDRIDGE.

Simple accelerated exposure test for varnishes and lacquers. H. V. HANSEN (Ind. Eng. Chem., 1928, **20**, 1384—1385).—An apparatus is described for exposing small test panels to the action of ultra-violet light. The material during exposure can be subjected to wet and dry cycles, and the results obtained agree very satisfactorily with those given by outdoor exposure; it is emphasised, however, that great caution must be used in interpreting results until considerable experience has been gained on materials of well-known stability to outdoor exposure. E. H. SHARPLES.

Constitution of gold resinate. F. CHEMNITZ and R. BARFUSS-KNOCHENDÖPPEL (Chem.-Ztg., 1928, **52**, 857—859).—Gold resinate (used in the ceramic industry) is prepared by precipitating a gold salt solution with "sulphur balsam" (a compound of sulphur and turpentine oils). Previous work on the constitution of the "balsam" and resinate is summarised, and details of the laboratory preparation and examination of these materials are given. The average values obtained on analysis of the precipitate were: Au 63.18%, C 18.31%, H 2.35%, O (diff.) 6.15%, and S 10.01%. S. S. WOOLF.

Saffron crocus. GRIEBEL and WEISS.—See IV. **Window-glass substitute.** CRIST.—See VIII. **Linseed oil films.** THURMANN and CRANDALL.—See XII. **Peru balsam.** TSCHIRCH.—See XX.

PATENT.

Manufacture of lacquers, films, artificial masses, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,133, 6.9.27 and 7.5.28).—The products comprise urea-formaldehyde condensation products, nitro-cellulose, and vegetable or animal oils, *e.g.*, linseed oil, castor oil, or cod-liver oil, which have been treated with oxidising gases at a high temperature to yield a viscous oil but not a jelly, and which may have been deodorised by hydrogenation. Resins, solvents, plasticising agents, etc. may also be added. L. A. COLES.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Importance of temperature and humidity control in rubber testing. I. Stress-strain and tensile properties. REP. PHYSICAL TESTING COMMITTEE OF DIVISION OF RUBBER CHEMISTRY OF THE AMERICAN CHEMICAL SOCIETY (Ind. Eng. Chem., 1928, **20**, 1245—1273).—By several thousand experiments it is shown that natural variations in temperature in a laboratory may affect the physical tests on vulcanised rubber as greatly as would an alteration of 25—40% in the time of vulcanisation, whereas humidity affects the results only to a minor degree. Variation in the absolute humidity of the atmosphere between mixing and vulcanisation, however, can affect the tensile strength and modulus of the products as greatly as does temperature variation after vulcanisation. Recommendations are therefore made that mixed stock prior to vulcanisation, and vulcanised stock prior to testing, be conditioned for 24—28 hrs. at 27.8° at 45% R.H., and that the testing room be at 27.8°. If this temperature cannot be maintained for conditioning prior to vulcanisation, the corresponding absolute humidity, *viz.*, 0.012 g. of water per litre, should still be used. The period between the removal of samples from the conditioning atmosphere and vulcanisation or testing should not exceed 2 hrs. The influence of variation in conditions before mixing is negligible as long as ordinary care is exercised in storing the rubber and compounding ingredients. Before complete standardisation of testing is possible it will be necessary also to specify limits for other variable factors. D. F. TWISS.

Strength test for rubber. SCHUBERT.—See V.

PATENTS.

Treatment of rubber latex. L. MELLERSH-JACKSON. From NAUGATUCK CHEM. Co. (B.P. 282,011, 5.10.27).—Alkali-preserved latex, concentrated and containing compounding ingredients if desired, is brought near to or slightly on the acid side of neutrality. Such treatment facilitates drying and imparts to the dried rubber mass increased plasticity and cohesiveness before vulcanisation, and greater tensile strength, and, frequently, improved resistance to abrasion afterwards.

D. F. TWISS.

Manufacture of transparent or translucent rubber. C. MACINTOSH & Co., LTD., S. A. BRAZIER, and E. H. HURLSTON (B.P. 300,936, 19.8.27).—Rubber is vulcanised with sulphur in the presence of an organic

accelerator such as zinc diethyldithiocarbamate, tetra-ethylthiuram disulphide, or piperidine piperidine-1-carbothionolate and a zinc or cadmium salt of a fatty acid.

D. F. TWISS.

Manufacture of articles of rubber and similar substances from aqueous dispersions containing such substances. DUNLOP RUBBER CO., LTD., and D. F. TWISS (B.P. 301,100, 25.7.27).—Aqueous dispersions such as rubber latex normally have their dispersed particles negatively charged with electricity. The sign of the electric charge is reversible, *e.g.*, by reduction of the value of the p_H to approx. 3; electrophoretic deposition is then effected on a cathode or on a porous diaphragm or mould surrounding the cathode.

D. F. TWISS.

XV.—LEATHER; GLUE.

Manufacture of an improved tannin extract from badan by the Smetkin-Jakimov method.

A. SMETKIN and P. JAKIMOV (Collegium, 1928, 477—480).—Thin badan roots were ground and sifted into fine particles (3—6 mm.) and coarse (6—15 mm. diam.). These were leached with cold and hot water (95°), respectively, in "Neubäcker" vessels. The product obtained in each case by cold-leaching contained an excessive amount of non-tans. Extraction of the materials with water at 95° yielded good liquors, which on concentration to d 1.183 contained (for fine and coarse, respectively): tans 21.6 (21.77), non-tans 11.44 (13.49), and insoluble matter 1.15 (1.41)%; the ratios of tans to non-tans were 65.4 : 34.6 (61.5 : 38.5). The spent material contained 2.88 (6.64)% of tan on the dry weight. The concentrated extract, which was soluble with difficulty in cold water, on being lightly sulphited became readily soluble. The yield of tannin was 70.5%. The extraction of badan roots of poor quality at suitable temperatures yields a valuable tannin extract superior to oakwood extract in its tans : non-tans ratio.

D. WOODROFFE.

Siberian larch bark as raw material for the manufacture of tannin [extract]. P. JAKIMOV and N. WELTISTOVA (Collegium, 1928, 488—495).—It is estimated that there are 500 million tons of larch bark (*Larix decidua*) available in Siberia. The thickest bark is less than 3 m. from the ground and could easily be removed. Samples of bark from trees of different ages were analysed; young trees 47—91 years old contained: tans 10.86—19.30, non-tans 6.0—9.1, difficultly soluble matter 0.8—1.6, sugary matter 0.7—1.4%; the tans : non-tans ratio was 1.5—2.7. The tannin content of these young trees was greater than that of trees 140—200 years old. The higher the bark the greater was the content of non-tans and sugary matter, the lower the ratio of tans : non-tans, and the thinner the bark. The outer layers of the bark contained 50% less of soluble matter and tannin, respectively, than the inner layers. The bark of damaged or diseased trees was inferior in tannin content to that of sound trees.

D. WOODROFFE.

Evaluation of [sulphite-]cellulose extracts [for tanning]. E. ANACKER (Collegium, 1928, 495—499).—Dilute solutions of the extracts do not show the Procter-Hirst reaction, but they give a precipitate with

β -naphthylamine hydrochloride. The analytical data are no guide to the suitability of the material for tanning. The shake method gives 2.5—3.0% less tannin than the filter-bell method on an extract of d 1.263. The tannin content is not important since these extracts are used either for filling leather or for solubilising other extracts. The affinity of sulphite-cellulose extract can be determined by finding the matter absorbed by hide powder from solutions containing 10 g. and 20 g. per litre, respectively. Close approximation of the two results shows high affinity of the extract for pelt. Crude sulphite-cellulose lyes show greater affinity by this test than extracts freed from lime and iron. Dark colour or darkening of colour on exposure to light, high hygroscopicity, bad smell, and high ash content should be avoided. Two extracts showed 77% and 44.3% increase in weight, respectively, at 85% humidity. The test was made with a little of the extract on a filter paper. The ash content of sulphite-cellulose extracts (d 1.263) varies from 5 to 12%. The possible use of lignin-sulphonic acids in tanning is discussed.

D. WOODROFFE.

Tanning leather with badan extract. A. SMETKIN and A. PISSARENKO (Collegium, 1928, 481—487).—Badan tannin particles show greatest dispersion at p_H 3—4. The viscosity of badan solutions containing more than 6% of tannin is comparatively high. Badan solutions are not salted out to a great extent, so that their high viscosity must be due to gummy or resinous matter. A pack of limed hides was completely delimed with sodium bisulphite and hydrochloric acid, treated for 2 hrs. with an acid solution (p_H 3.5), coloured for 1 day in weak tan liquors containing 0.1% of tannin, for 1 day each in 0.3% and 0.6% solutions, then in solutions of d 1.009 and, on the sixth day, of d 1.014. After 3 days in this liquor and 3 days in one having d 1.022, the hides were given 3 layers of 2, 2, and 3 weeks' duration, respectively, in liquors of d 1.037, 1.052, and 1.067. The leather was next drummed for 4 hrs. in extract liquor of d 1.075, then run for 12 hrs. in strengthened liquor (d 1.087), and left in the liquor for 36 hrs. The leather was drum-oiled and finally dried. The product had moisture 18.0, ash 1.12, fat 2.87, water-soluble matter 13.97, hide substance 37.30, combined tan 26.74%, degree of tannage 71.72, yield 268, water absorption (2 hrs.) 25.79%, (24 hrs.) 36.25%. The finished leather could not be distinguished from ordinary sole leather either in appearance or analysis.

D. WOODROFFE.

XVI.—AGRICULTURE.

Alkali soil studies and methods of reclamation.

P. S. BURGESS (Ariz. Agric. Exp. Sta. Bull., 1928, 123, 157—181).—Arid climates with periodical excesses of moisture are necessary conditions for the accumulation of alkali in surface soils. Nitrates and chlorides migrate through moist soils with greatest speed, sulphates and hydrogen carbonates less readily, whilst carbonates and hydroxides move very slowly. The conditions leading to freezing up or puddling during reclamation are described. Intermittent flooding and drying, followed by application of 1—2 tons of calcium sulphate per acre, is employed to displace all the sodium present in the

soil zeolites. Application finally of organic matter (manure) removes sodium hydroxide by combination with the carbon dioxide liberated.

CHEMICAL ABSTRACTS.

Relations of green manure to the nitrogen of a soil. T. L. LYON and B. D. WILSON (Cornell Agric. Exp. Sta. Mem., 1928, No. 115, 1—29).—Experiments in which crops of vetch, field peas, rye, oats, buckwheat, and grass were ploughed under showed that not all legumes are more effective than non-legumes in producing a high content of nitrate nitrogen during the main part of the growing season. Liming the soil increased the accumulation of nitrate nitrogen. The loss of total nitrogen was in inverse order to the nitrate nitrogen content of the soil when the nitrates were highest, but the relation between the percentage of total nitrogen in the soil and the nitrate nitrogen accumulation was less definite.

CHEMICAL ABSTRACTS.

Plant composition as a guide to the availability of soil nutrients. R. M. SALTER and J. W. AMES (J. Amer. Soc. Agron., 1928, 20, 808—836).—The rates at which crop plants take up nitrogen, phosphorus, and potassium are not related to the increase in dry weight. Neubauer's seedling test is criticised. The stalk tests for potassium or nitrogen requirements are untrustworthy.

CHEMICAL ABSTRACTS.

Effect of soil type and fertilisation on the composition of the expressed sap of plants. M. M. MCCOOL and M. D. WELDON (J. Amer. Soc. Agron., 1928, 20, 778—791).—The percentage of nitrogen, phosphorus, potassium, calcium, and magnesium in the juice of the leaves is usually greater than in that of the stems, but in rainy weather potassium may be leached from the leaves more than from the stems. Application of mineral nutrients generally results in increased concentrations of those elements in the juice of the crop. If one element is a limiting factor, slow growth appears to permit the accumulation of high concentrations of other elements.

CHEMICAL ABSTRACTS.

Relative availability of the nitrogen of oil cakes as indicated by pot-culture studies. S. K. AYYAR (Madras Agric. Dep. Yearbook, 1926 (1927), 92—95).—With nitrogen fertilisers alone the increases in yield of grain (ragi) were in the following order: ground-nut, hoongay, steamed horn meal, castor, ammonium sulphate, ganja. With nitrogen fertilisers and superphosphate, the order was: steamed horn meal, ground-nut, castor, ganja, ammonium sulphate, hoongay.

CHEMICAL ABSTRACTS.

Relative nitrifiability of different nitrogenous organic manures in certain soils of the Central Farm, Coimbatore. K. S. V. AYYAR (Madras Agric. Dep. Yearbook, 1926 (1927), 85—91).—The results of experiments on a black cotton soil and a red garden soil are recorded.

CHEMICAL ABSTRACTS.

Fertiliser experiments with cotton. H. M. STEECE (Rep. Agric. Exp. Stat., Washington, 1927, 79—86).—The collected results of fertiliser trials during the period 1919—27 are reviewed and discussed from the point of view of the relative merits of various fertilisers and the manner in which these are applied.

A. G. POLLARD.

Fertilisation of soils poor in lime. Comparative tests with various carbonates. G. RIVIÈRE and G. RICHARD (Compt. rend., 1928, 187, 1068—1070).—Crops of wheat and oats were grown on soil poor in lime, plots of which were previously treated with the carbonates of calcium, magnesium, sodium, and potassium, respectively, in equivalent quantities. The plot treated with sodium carbonate in each case yielded the heaviest crop, the sodium probably supplying an essential plant food as well as facilitating nitrification.

B. W. ANDERSON.

Some effects of fertilisers on the nature of the soil solution, with special reference to phosphorus. C. H. SPURWAY (J. Amer. Soc. Agron., 1928, 20, 802—807).—Generally soils containing 0.5 in 10^5 or less of soluble phosphorus respond to phosphorus fertilisation; with those containing 2 in 10^6 the application of phosphorus is of doubtful profit.

CHEMICAL ABSTRACTS.

Mathematical interpretation of experimental results obtained with fertilisers for sugar cane. Y. KUTSUNAI (Proc. Hawaiian Sugar Planters' Assoc., 1927 (1928), 171—175).—Cane yields calculated from Spillman's formula $Y = M - AR^x$, where Y is the yield with x units of potash fertiliser (applications of nitrogen and phosphate being constant), M and A are constants, and R is the ratio between the increment due to $x + 1$ and that due to x , closely agreed with those obtained using 60—240 lb. of potash (K_2O) per acre.

CHEMICAL ABSTRACTS.

Fertilising value of Baltimore sewage sludge. KEEFER and ARMELING.—See XXIII.

PATENTS.

Production of an alkaline-reacting nitrogenous fertiliser with gradable alkalinity. N. CARO and A. R. FRANK (B.P. 286,282, 30.9.27. Ger., 3.3.27).—The product containing 10—30% of fixed nitrogen obtained by the action of ammonia on calcium carbonate etc. at 500 — 900° , prepared as described in B.P. 279,419 and in B.P. 279,421 and 279,811 (B., 1929, 53, 68), is heated at 900° in a current of a non-reacting gas, e.g., nitrogen, hydrogen, or carbon monoxide, or is heated *in vacuo*, to yield a product containing only free lime in addition to the nitrogen-fixation product for use with acid soils, or is treated at 300 — 600° with carbon dioxide or gases containing it to yield a product containing no free lime. Intermediate products containing any desired proportion of free lime are prepared by the partial application of either method. Alternatively a product containing no free lime is prepared by the addition of carbon monoxide to the ammonia in the initial process.

L. A. COLES.

Manufacture of insecticides. W. H. GROOMBRIDGE, and A. J. DICKINSON, LTD. (B.P. 301,186, 10.11.27).—Solutions particularly adapted for spraying on to the cotton plant for destroying the pink boll-worm contain ammonium fluosilicate and one or more alkali or other soluble soaps, preferably with the addition of free alkalis, the ingredients being mixed below 30° .

L. A. COLES.

Producing a liquid insecticide containing the effective ingredients of *Derris* species. ZAIDAN HOJIN RIKAGAKU KENKYUJO (B.P. 285,797, 20.2.28).

Japan, 18.2.27).—The active ingredients obtained by extracting powdered derris root with, *e.g.*, ether or benzene are dissolved in a mixture of anhydrous material free from alkali, *e.g.*, fish oil, fats, waxes, resins, or higher alcohols, hydrocarbons, or fatty acids, with an approximately equal quantity of an anhydrous solvent, *e.g.*, benzene, carbon tetrachloride, or trichloroethane, and the product is emulsified with soap solution or mixed with gum arabic to form a solid product. (Cf. B.P. 239,483; B., 1926, 614.) L. A. COLES.

Fungicide. I. G. FARBERIND. A.-G. (B.P. 276,033, 16.8.27. Ger., 16.8.26).—A fungicide for seeds consists of a copper salt mixed with a solid caustic alkali and a tartrate, *e.g.*, a mixture of 15 pts. of anhydrous copper sulphate, 21 pts. of anhydrous Seignette salt, 9 pts. of caustic soda, and 5 pts. of sodium chloride.

H. ROYAL-DAWSON.

Weed killer. R. N. CHIPMAN (U.S.P. 1,694,205, 4.12.28. Appl., 15.4.26).—A non-poisonous herbicide consists chiefly of a mixture of calcium chlorate and chloride.

H. ROYAL-DAWSON.

XVII.—SUGARS; STARCHES; GUMS.

Effect of hot molasses on exhausted beet slices. W. KOHN (Z. Zuckerind. Czechoslov., 1928, 53, 157—161).—On treating exhausted beet slices for $\frac{1}{2}$ hr. with molasses having a density of 19.6—25.4° Balling, at 85—95°, the following were found to have passed over from the latter to the former material: sugar 0.79—1.87% (of the dry substance of the molasses), nitrogen 0.07—0.16% (of the polarisation of the molasses solution), ash 0.94—1.71% (of the polarisation of the molasses solution), K_2O 0.60—1.10 and Na_2O 0.1—0.3% (of the carbonated ash of the molasses solution). No transfer of either calcium or magnesium took place.

J. P. OGILVIE.

Acidimetric method of determining dextrose using Fehling's solution. M. D. HADJEFF (Z. Unters. Lebensm., 1928, 55, 615—618).—The reaction of the dextrose with Fehling's solution is carried out by the Meissl-Allihn method. The cuprous oxide is separated and washed by decantation, the finer particles being collected by filtering the liquid through an unweighed Allihn tube. It is dissolved in a measured quantity of a mixture of 0.5*N*-sulphuric acid and 0.25*N*-hydrogen peroxide. The excess acid is titrated by means of 0.5*N*-sodium bicarbonate, 3 drops of methyl orange being added as indicator, and the titration continued until a permanent yellowish-green tint is obtained; the volume of acid used to dissolve the cuprous oxide is multiplied by the empirical factor 1.020 to give the corresponding volume of 0.5*N*-sulphuric acid (1 c.c. 0.5*N*- $H_2SO_4 = 0.015378$ g. Cu). The corresponding weight of dextrose is found from the Meissl-Allihn tables. The quantity of copper sulphate in the 100 c.c. of solution titrated must not be greater than 2 g. and preferably should be less than 1 g.

W. J. BOYD.

Humification of carbohydrates. FUCHS.—See V. **Fertilisers and sugar cane.** KUTSUNAI.—See XVI.

PATENTS.

Cellulose saccharification (U.S.P. 1,687,785).—See V. **Fermentation of cane sugar etc.** (B.P. 295,648).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Water content of the yeast cell. A. HARDEN (J. Inst. Brew., 1928, 34, 607—608).—According to Paine (B., 1912, 38), 100 c.c. of a 0.1*M*-solution of sodium chloride, when added to 50 g. of pressed yeast, produces in 3 hrs. an extrusion of only 3.8 g. of liquid from the yeast cells. Interpreting Paine's experiment according to Beetlestone's theory (B., 1928, 170), which assumes that pressed yeast contains 30% of solids, 40% of interstitial water, and 30% of internal water, it is obvious that 100 g. of salt solution containing 0.58 g. of sodium chloride yield 120 g. of solution + 3.8 g. of liquid = 123.8 g. of solution containing 0.58 g. of sodium chloride, *i.e.*, 0.47% of sodium chloride. The amount actually found is 0.56%, which indicates that the large amount of interstitial water postulated by Beetlestone is not present in yeast cake, and that the internal water content of the yeast cell is much nearer 70%, a conclusion reached by Harman and Oliver, and also by Hopkins.

C. RANKEN.

Influence of sodium chloride on the growth and metabolism of yeast. H. B. SPEAK, A. H. GEE, and J. M. LUCK (J. Bact., 1928, 15, 319—340).—Wort containing up to 10% of sodium chloride is fermentable by *Saccharomyces cerevisiae*, the weight of the yeast crop decreasing with increase in salt concentration. The influence of the salt on the maximum rate of carbon dioxide production depends on the size of the inoculum as well as on the salt concentration. The effect of the salt is on cell division. The nitrogen content of the crop is low up to 6% of sodium chloride. The ratio sugar fermented/yeast crop increases with sodium chloride concentration up to 5%; the ratio carbon dioxide/sugar utilised rises as the concentration of sodium chloride increases up to 2%.

CHEMICAL ABSTRACTS.

Cold and warm malting. H. LÜERS (Woch. Brau., 1928, 45, 569—574).—High-grade Hungarian barley was floor-malted for malt of Pilsener type, heaping and turning being arranged to maintain average temperatures of 14°, 16.5°, and 23°. The times on the floor were 13, 9, and 7 days, respectively, and the malting loss was greatest in the low-temperature experiment owing to excessive respiration. This condition was reversed on growing "cold" and "warm" batches for 10 and 9 days, respectively, when growth and modification were similar and satisfactory, and the "cold-floored" malt gave higher laboratory extract. Malt grown at 14° contained more amino- and soluble nitrogen, sucrose, invert sugar, and soluble phosphoric acid. The diastatic power was greater and, in the brewery, saccharification was more rapid and less extract was lost in the grains. The resulting beers were of excellent flavour and appearance, those from the "warm-floored" malts being thin flavoured and lacking brilliancy. The malt floored at 16.5° gave intermediate results, though nearer to those of the cold-floored malts. A similar experiment on Bavarian barley treated by Mürger's

mechanical-pneumatic system for malt of Munich type was carried out at about 15.5° and 21°. The malt prepared at 15.5° gave better grinding results, higher extract, and more rapid saccharification. Differences in protein breakdown were less marked in this case, but the "warm-prepared" malt contained much more soluble and a lower proportion of inorganic to total phosphoric acid. It also contained more sucrose and invert sugar than the malt prepared at 15.5°, probably on account of the excessive aeration necessary to keep down the temperature of the latter, with resulting increased respiration. The malt grown at 21° gave a beer of good typical Munich flavour, whilst that from malt grown at 15.5° was characterless and rough. It would appear that low temperatures are better for preparing malts of Pilseuer type and high temperatures for Munich malts, though the differences found may be in part due to the use of different malting methods. F. E. DAY.

Defective wines. E. HUGUES (Ann. Falsif., 1928, 21, 519—532).—The results of analysis are recorded for a large number of wines defective from various causes, and figures for equivalent normal wines are given for comparison. The effect of hail may be seen in high values for dry extract, ash, and alkalinity of ash, and moulds may have been present; when the harvest has been retarded the wines may be very green and high in dry extract. Drought caused a low content of alcohol and high acidity, and much the same type of result was found when the sun had caused total or partial drying up of some grapes. In cases of inundation of vineyards damaged grapes may be cautiously used for white wines, but defects in taste caused by deposits of mud and diseases due to various organisms, high dry extracts, and low acidities may be expected. The attacks of insects may cause a drying up of the grapes, but often afford entrance to moulds etc. Such vintages yield wines rich in dry extract and in fixed acidity, and have a high proportion of glycerin and peptic substances; glycuronic acid may also be present. Mildew has a retarding influence on maturity, and predisposes the wine to attack by "tourne," resulting in flatness and turbidity. At the same time total acidity is increased and the cream of tartar disappears. Attack by grey rot results in an increase of alcohol and extract, a lowering of total acidity and cream of tartar, and the presence of dextrorotatory substances. Such wines are frequently attacked by "casse brune." *Mycoderma vini* gradually transforms the alcohol into water and carbon dioxide, and esters and substances giving rise to the bouquet are slowly oxidised. Sour wines indicate the presence of acetic acid. *Tourne* causes disappearance of tartrates and a lowering of fixed acidity. The presence of mannitol, found particularly in hot seasons, gives a peculiar flavour at once sweet and sour; it attacks the *lævulose* of incompletely fermented wines, and owing to transformation of malic and lactic acids the fixed acidity is lowered. "*Vins de lies*" have high dry extract and ash, and low alcohol content, low alkalinity of ash, and high content of phosphates and ammoniacal nitrogen. The amount of pressing has an effect on the wine; a pressed wine has higher dry extract, tannin, ash, nitrogen, phosphoric acids, sulphates, and chlorides than an unpressed one. Intensive cultivation may in

certain seasons produce wines the composition of which simulates fraudulent watering, but may usually be distinguished by the higher values for ash, alkalinity of ash, and total potash. D. G. HEWER.

Distillation of high-percentage alcohol. E. LÜNDER and W. KILP (Chem. Fabr., 1928, 713—715, 726—727).—In two distilleries in which tests were carried out it was found that the mash, treated in column stills of 16 trays, was completely stripped of alcohol on the eighth and tenth trays. This is, however, no more than an adequate safety margin, the mash used containing 7.5—8.0% of alcohol against a normal 9—10% content. A similar series of samples were then taken on the trays of the rectifiers, and the alcohol and fusel oil contents determined. The two columns must be carefully proportioned to each other or the volume of condensate in the second may be excessive. The bottom tray of the rectifier should in normal working contain 50—60% (wt.) alcohol and the reflux for high-strength spirit can be kept down to 15%. The dephlegmator serves mainly as a heat exchanger. The fusel oil contents decrease steadily as the vapour passes up the rectifier. Provided the ethyl alcohol content of an aqueous solution exceeds 42%, amyl alcohol, the principal constituent of fusel oil, can be separated from it by fractionation. The highest fusel oil content (30%) is found in the bottom of the rectifier. C. IRWIN.

Detection of formaldehyde. CHERICI.—See XIX.

PATENTS.

Fermentation of cane sugar, glucose, and fructose. P. LINDNER (B.P. 295,648, 10.7.28. Ger., 16.8.27).—A mixture of lactic acid, alcohol, and carbon dioxide is produced by dissolving sucrose, dextrose, or *lævulose* in milk or other liquid containing casein, and fermenting the whole at 25—35° after inoculation with *Thermobacteria mobile* derived from the aguamiel of the *Agave americana*. C. RANKEN.

Eliminating the unpleasant smell attached to glycerin obtained by fermentation. VEREIN. CHEM. WERKE A.-G. (B.P. 278,703, 30.9.27. Ger., 11.10.26).—Crude glycerin obtained by fermentation is treated, either in the concentrated state or in aqueous solution, with oxidising agents such as sodium hypochlorite, prior to its distillation. C. RANKEN.

Furnaces for malt kilns (B.P. 300,322).—See I.

XIX.—FOODS.

Separation of constituents of frozen milk after fractional fusion. V. V. WINTER (Chem. Listy, 1928, 22, 550—554).—Milk is frozen and allowed to melt gradually, the successive portions of melted milk being analysed. The fat, protein, lactose, and salt contents of the first fraction are approximately twice as great as those of the original milk, whilst in the last fraction the content of fat has fallen from the original value of 2.85% to 0.6%, of protein from 3.38% to 0.22%, of lactose from 5.11% to 0.53%, and of salts from 0.69% to 0.01%, and the sp. gr. from 1.0333 to 0.9989. The content of these substances falls regularly in the successive fractions, with the exception of lactose, the relative concentration of which rises in the last fraction.

R. TRUSZKOWSKI.

"Chromiform" as a preservative for milk samples. FONZES-DIACON and LAFORCE (Ann. Falsif., 1928, 21, 536—539).—The addition of one pastille, containing 0.25 g. of potassium dichromate and 0.25 g. of trioxymethylene, to a 250 c.c. sample of milk preserves the milk so that after 2 months' exposure to light the figures of analysis are comparable with those of the fresh milk. When this reagent ("chromiform") is used, the acidity of the milk is very little raised, the attack of the lactose by ferments is negligible, the casein is but very slightly hydrolysed, the fat can be determined by Gerber's method, the determination of chlorides is not influenced, and the constants still permit the determination of 5% of added water.

D. G. HEWER.

Composition of Irish butter. Distribution of the volatile acid groups among the glycerides of butter fat. P. ARUP (Analyst, 1928, 53, 641—644).—Quantities of butter fat were prepared from composite samples of butter representing three samplings of 114—205 samples each time, taken in the Irish Free State. The fats were slowly crystallised in incubators at 27°, and separated by filtration into solid and liquid portions, and the process was continued to obtain fractions liquid at 10° as the lower limit, and those crystallising at 37° as the upper extreme, with four intermediate fractions. The Reichert-Meissl, Polenske, and Kirschner values and the iodine values of the fractions were determined, and the results are interpreted as indicating an approximately even distribution of the various volatile acid groups among the liquid and solid glycerides, and as giving support to the theory of impartial, mutual distribution of the acid groups among the different glycerides. The iodine values indicate an approximation to the mono-olein type in the fraction crystallised at 37°, and to the diolein type in the fraction liquid at 10°.

D. G. HEWER.

Identification substances in margarine. L. HORON (Ann. Falsif., 1928, 21, 532—534).—The compulsory addition of sesame oil and potato starch to margarines has had the effect in Belgium of confining examination of such products to a search for these substances, and an extension of the system to France is deprecated. It is pointed out that as a result butter is now adulterated with small quantities of coconut oil, lard, or a mixture of the two. The study of the inter-relationships of the various analytical constants is advocated. (Cf. following abstract.) D. G. HEWER.

Determination of coconut oil in butter. L. HORON (Ann. Falsif., 1928, 21, 534).—The Polenske determination is repeated twice. A small quantity of the supernatant insoluble volatile acids is removed, and the refractive index determined at 20° or 25°. This value is divided by the number of c.c. of 0.1N-caustic soda required for the neutralisation of the insoluble volatile acids. Pure butters give values 6 to 7 units greater than those containing 10% of coconut oil. D. G. HEWER.

Standardisation of vitamin-D in butter and margarine containing vitamin. F. FLURY (Biochem. Z., 1928, 203, 14—21).—In feeding experiments with rats, the antirachitic action of a margarine containing different amounts of irradiated ergosterol (radiostol) was tested. Animals receiving a diet free from vitamin-

D to which was added 0.1—0.2 g. of such a margarine did not develop rickets, and rats which had developed rickets showed considerable improvement in condition when they received 0.2 g. per day.

P. W. CLUTTERBUCK.

Possible transference of mercury from grain cauterisers into the harvested corn and flour. A. STOCK and W. ZIMMERMANN (Z. angew. Chem., 1928, 41, 1336—1337).—Traces of mercury are to be found in the plants and harvest-corn grown from seed treated with solid cauterisers containing mercury, whence mercury may be introduced in minute quantities into flour, bread, etc. The mercury is not wholly removed by washing from such cauterised seed-corn, which should therefore not be admixed with food or fodder.

E. LEWKOWITSCH.

Honey ash. F. E. NOTTBOHM (Arch. Bienenkunde, 1928, 8, 32—52; Chem. Zentr., 1928, ii, 115).—The ash of blossom- and honeydew-honey contains, respectively, K₂O 30.50—50.78, 52.59—57.16; Na₂O 5.54—10.03, 3.16—4.31; CaO 2.12—8.00, 0.52—1.30; MgO 1.50—2.17, 0.71—2.31; P₂O₅ 1.65—12.50, 6.64—9.51%.

A. A. ELDRIDGE.

Determination of honey in honey cake. R. T. A. MEES (Chem. Weekblad, 1928, 25, 674—676).—It is assumed that the honey content may be measured by the invert sugar content, and therefore by the percentage of laevulose present. The laevulose is determined by the iodometric method of Kolthoff (cf. B., 1923, 467 A), with the modification that N-solutions are used instead of 0.1N-solutions, since the aqueous extract is already dilute. The quantities so found agreed with the laevulose content of the sugar mixture employed in batches of cake specially prepared.

S. I. LEVY.

Detection of formaldehyde in foods. E. CHIERICI (Annali Chim. Appl., 1928, 18, 504—508).—To 10 c.c. of concentrated sulphuric acid in a test-tube are carefully added a certain amount of the substance to be tested, treated as described below, and 0.2 c.c. of alcoholic 5% gallic acid, the whole being partially mixed. In presence of formaldehyde, the solution turns emerald-green with blue edges below and exhibits two characteristic absorption bands in the red. Under these conditions sugars give a hay-green coloration, but this does not interfere with the test if the reaction is carried out in the cold. The materials to be tested are prepared as follows—Milk: 20 c.c. are mixed with a few drops of concentrated sulphuric acid and the liquid is filtered through a double filter. Wine: 20 c.c. are completely decolorised by animal charcoal without heating and filtered; the use of hexamethylenetetramine for desulphiting the wine may lead to the formation of formaldehyde. Beer may be tested directly. Canned meat: about 30 g. are shaken with an equal volume of boiling water and filtered through a moistened filter. Syrup, mustard: the separated ethereal extract is added dropwise to the reagent. Jam: 50 g. are mixed with 50 c.c. of boiling water and filtered through a moist double filter, the ethereal extract of the filtrate being tested. Butter etc.: 20 g. are melted by mixing with sufficient boiling water, the cooled mass being filtered through a moist double filter, and the filtrate extracted

with ether. Vinegar etc.: this is first decolorised with animal charcoal. Tomato extract: this responds to the reaction, in spite of the presence of dextrose and glutamic acid.

T. H. POPE.

Determination of caffeine in tea. S. GOBERT (Ann. Falsif., 1928, 21, 517–518).—3 g. of the very finely-powdered tea are moistened with 4 c.c. of ammonia to swell the cells and liberate the combined caffeine. After 30 min. 4 extractions are made, each with 25 c.c. of ethyl acetate. On evaporation, taking up the residue with water, re-evaporation, and treatment with chloroform a residue of 2–3 mg. results. Purification is then carried out by treating with 4 c.c. of ammonia (*d* 1.18), and after 30 min. with occasional shaking, 4 extractions, each with 25 c.c. of ethyl acetate, are made, the liquid is centrifuged for 5–7 min., and the ethyl acetate distilled from the decanted liquid. The dried residue is extracted twice with 50 c.c. and once with 25 c.c. of boiling water; 15 c.c. of 1% potassium permanganate are added to the extract, and after 15 min. the manganese is precipitated by 12-vol. hydrogen peroxide containing 1% of glacial acetic acid. After keeping, filtering, and evaporating, the residue is dried, extracted 3 times with 25 c.c. of chloroform, and after filtering and washing with chloroform, the residue is dried and weighed. Moisture is determined at the same time. Concordant duplicate results were obtained on samples of Ceylon tea.

D. G. HEWER.

Potentials of tin and iron [in fruit canning]. KOHMAN and SANBORN.—See X.

PATENTS.

Manufacture of evaporated milk. H. WADE. From BORDEN Co. (B.P. 301,220, 19.12.27).—The filling and sealing of the cans is carried out in a sterilising atmosphere of saturated steam at 127° at a pressure sufficient to prevent ebullition. The cans are cooled to 43° before being exposed to atmospheric pressure. By this process the usual practice of shaking the cans to relieve the milk is unnecessary.

W. J. BOYD.

Manufacture of alimentary fish powders. F. BILLON (B.P. 286,709, 2.2.28. Fr., 11.3.27).—Whole fish or fish offal is crushed to a paste, with which chloropierin is mixed in the proportion of 1–3 pts. per 1000. The paste is rapidly dried in a fan-dryer at a temperature not exceeding 70°, and the resulting powder is de-oiled by means of a solvent.

W. J. BOYD.

Manufacture of phosphatides. "PHARMAGANS" PHARM. INST. L. W. GANS A.-G. (B.P. 285,417, 17.8.27. Ger., 16.2.27).—Plant or animal tissues are subjected, without destruction of their cellular structure, to dialysis with pure water or an aqueous solution, such as a 15% solution of alcohol, at a temperature not above 37°. The cell walls act as the dialysing diaphragms. The process of dialysis may be accelerated by producing a *P.D.* in the dialysing liquid. Separation of the phosphatides from solution may be achieved, *e.g.*, by adding organic solvents or albuminous substances to the dialysing liquid so that the phosphatides will be precipitated when the added substances reach a certain concentration, or by adding adsorptive agents such as aluminium or iron hydroxide. The phosphatides obtained by this

process are soluble in water, unlike the phosphatide products hitherto known. They contain besides the lecithin nucleus, carbohydrates, anthocyanins, and inorganic compounds with calcium and iron, whereby their value for nutritive, therapeutic, and bacteriological purposes is enhanced.

W. J. BOYD.

Purification of cereal products [from dust and light particles]. H. SIMON, LTD., and H. J. DENHAM (B.P. 301,765, 1.9.27).

Nutrient medium (B.P. 271,120).—See XX.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Action of ultra-violet light on ferric citrate solutions. H. S. FRY and E. G. GERWE (Ind. Eng. Chem., 1928, 20, 1392–1394).—When citric acid solutions containing ferric salts are exposed to ultra-violet light, 3 mols. of carbon dioxide are liberated for every mol. of ferric sulphate present. It is assumed that the photochemical change involves the oxidation of citric acid, with the liberation of 1 mol. of carbon dioxide, to the unstable acetonedicarboxylic acid. This also loses 1 mol. of carbon dioxide, thereby giving acetoacetic acid, which decomposes with the formation of acetone and a third molecule of carbon dioxide. The quantitative data obtained confirm these assumptions.

E. H. SHARPLES.

Examination of pharmaceutical preparations by the extended capillary diagram and the analytical quartz lamp. RAPP (Pharm. Ztg., 1928, 73, 1585–1589).—The following development on the methods of Kunz-Krause and Goppelsröder is described. A glass tube 60 cm. long and 3 cm. in diam. stands in a small cylindrical glass or porcelain vessel of about 20 c.c. capacity and only just wide enough to fit outside the tube. A piece of fluted filter-paper 56 cm. long and 2 cm. wide is folded lengthways and placed in the tube with the bottom resting in the small vessel, and only the top of the paper touching the walls of the tube. 3–5 c.c. of the liquid to be examined are placed in the small vessel and the top of the tube is loosely corked. After remaining thus for a definite time the paper is removed, completely dried, and examined by ultra-violet light. *E.g.*, a mixture of 0.1% solutions of the hydrochlorides of quinine, narceine, apomorphine, papaverine, and pilocarpine gave a "capillary strip" 25.5 cm. long which, after drying and examination with the quartz lamp, showed the first 14.7 cm. marine-blue (apomorphine), then 3.7 cm. bluish-black (apomorphine and narceine), 2 cm. bluish-grey (quinine and papaverine), 4.1 cm. yellow (papaverine and pilocarpine), and, finally, 1 cm. fluorescent-blue (quinine). In other cases the bands vary in size, colour, and structure, and the method has been successfully applied to the study of the following: (a) deterioration of alkaloid solutions on keeping; differences in capillary behaviour were noticed with solutions of apomorphine, narcophine, physostigmine, pilocarpine, and scopolamine after keeping for 6 months; (b) stability of "Ysat" preparations; (c) differentiation of pure chemical substances and their proprietary equivalents; thus, differences were noticed between aspirin and acetylsalicylic acid in ether

solution, and between veronal and diethylbarbituric acid in both alcohol and ether solution; (d) marked variations in colour and appearance which occur in the capillary values of 10 commercial digitalis preparations and 5 ergot preparations; (e) comparison between preparations from plants and drugs and commercial extracts and galenical preparations having similar names. Application of the method for detection of adulteration, for routine pharmaceutical control, and other possible uses is referred to, together with an outline of future work. E. H. SHARPLES.

Determination of calcium carbonate in calcium glycerophosphate, lactophosphate, and the mono- and di-acid phosphates. E. LETURC (Ann. Falsif., 1928, 21, 534—536).—About 0.5 g. of the dried glycerophosphate, thinly spread over a porcelain crucible, is calcined to a slightly grey ash, and 0.4 c.c. of hydrochloric acid ("official") and 3 c.c. of water are added. After dissolution the liquid is filtered hot. If there is any black residue, the filter must be ashed again. 30 c.c. of water and 20 c.c. of 50% acetic acid, followed by 1 g. of sodium acetate, are then added, and after dissolution 1 g. of oxalic acid is added. After heating and rotating the liquid, the calcium oxalate formed sinks to the bottom and is removed by filtration, dried, and weighed. Calcium lactophosphate is treated similarly. For the mono- and di-acid phosphates the French Codex method is followed at first, and, after the washing by decantation, the residue is dried, calcined, taken up with 0.5 c.c. of hydrochloric acid (official) and 3 c.c. of water, dissolved, and filtered, and the method described for the glycerophosphate then followed. D. G. HEWER.

Preparation of liquor aluminii acetici. A. WØHLK (Dansk Tidsskr. Farm., 1928, 2, 320—323).—In order that the resulting solution shall be stable on keeping, it is necessary that exactly equivalent quantities of aluminium sulphate and calcium carbonate be used in its preparation, and it is recommended that the sulphate content of the former and the purity of the latter should both be determined volumetrically before use. H. F. HARWOOD.

Manufacture of argentum proteinicum, argentum colloidal, and other organic silver compounds. J. SCHWYZER (Pharm. Ztg., 1928, 73, 1549—1553, 1568—1570).—The preparation from casein of lysalbinic and protalbinic acids, the conversion of the latter into silver sodium anhydroprotalbinates, and the final preparation of argentum proteinicum are described in detail, with special reference to the methods of control. The specifications of the pharmacopœias of different countries are discussed, and amendments and additions suggested. An apparatus and method for dialysis on the large scale are described, but no really satisfactory system is yet known. The manufacture of argentum colloidal is discussed, and the view is advanced that this is, as regards almost the whole of the silver content, a definite silver compound, the characteristic opalescence being due probably to only 1 or 2% of the silver content actually present as colloidal metal. Tests for alkalinity (determined on the ash) and solubility in water are described and suggested for inclusion in the pharmacopœias. A brief description is

also given of several less important preparations of silver with protein acids. S. I. LEVY.

Assay of ointments of mercuric oxide and ammoniated mercury. N. L. ALLPORT (Quart. J. Pharm., 1928, 1, 23—27).—Mercuric oxide ointment (yellow, 5 g., or red, 1 g.) is warmed at 50° with 100 c.c. of a mixture of benzene 13, acetic acid 2, and 90% ethyl alcohol 5 pts. The resulting clear solution is saturated with hydrogen sulphide, the mixture heated at 50°, the precipitate collected in a Gooch crucible, washed with hot benzene and a little alcohol, and dried at 100°. Ammoniated mercury ointment (2.5 g., or 1.5 g. of B.P. 1898 preparation) is heated at 70° with 100 c.c. of a mixture of benzene 9, acetic acid 10, 90% ethyl alcohol 1 pt., the resulting clear solution being then treated as above. CHEMICAL ABSTRACTS.

Evaluation of rhubarb. A. TSCHIRCH and P. SCHMITZ (Pharm. Acta Helv., 1928, 3, 88—92; Chem. Zentr., 1928, ii, 375—376).—Determinations of chrysophanic acid showed no difference between Chinese rhubarb and rhubarb of the *Palmatum* group cultivated by the authors. When kept for a long period the material gives lower values, whilst the rhizomes of old plants give higher values than those of young ones.

A. A. ELDRIDGE.

Evaluation of Peru balsam. A. TSCHIRCH [with H. ROSENTHAL and G. FRIEDLÄNDER] (Pharm. Acta Helv., 1928, 3, 85—88; Chem. Zentr., 1928, ii, 375).—The analytical characteristics of Peru balsam are described. The material has d_{25}^{25} 1.145—1.167, and gives a clear mixture with 1 vol. of 90% alcohol, but turbidity on addition of a further 7 vols. of alcohol; 3 g. of balsam give a clear mixture with 1 g. of carbon disulphide, and yield a brown resin on addition of 9 g. of carbon disulphide. The characters of the portions soluble or insoluble in ether and light petroleum are described. These and other analytical tests are preferred to determinations of the acid and saponif. values of the crude product. A. A. ELDRIDGE.

Volumetric process for the determination of phenazone. J. RAE (Pharm. J., 1928, 121, 575).—Excess of picric acid is added, the solution filtered after 10 min., and the excess of acid determined by titration with 0.1N-sodium hydroxide solution. S. I. LEVY.

Action of Schiff's reagent on pyrimidone. A. VALDIGUÉ (J. Pharm. Chim., 1928, [viii], 8, 506—510).—By the addition of Schiff's reagent to an aqueous or alcoholic solution of pyrimidone the solution is coloured red, more or less intense according to the proportions of the reactants. Antipyrine does not give the reaction, but many samples of pyrimidone of varied origin, the camphorate, salicylate, hydrochloride, and sulphate, all form the red colour. 2 mg. of pyrimidone in 10 c.c. of water give a very distinct rose colour with 3 or 4 drops of the reagent. The colour is stable to air and light, is intensified on heating, and persists on cooling, but it is destroyed by strong acids, alkalis, and reducing agents. It is not due to aldehydic impurity. E. H. SHARPLES.

Carvacrol. A. WAGNER (Pharm. Zentr., 1928, 60, 757—763, 773—777).—An account is given of the history, constitution, occurrence, formation, preparation,

properties, reactions, detection, and determination of the product, with an exhaustive bibliography.

S. I. LEVY.

Colours of some newer dyestuffs used in medicine at different hydrogen-ion concentrations.

H. W. VAN URK (Pharm. Weekblad, 1928, 65, 1227—1230).—Tables are given showing the colours for the pH range 1—12 of the acridine derivatives tryptaflavine and rivanol, of the sodium compound of tetraiodophenolphthalein ("Iodotetragnost"), and of the sodium salt of the mercury compound of dibromofluorescein ("mercurochrome 220").

S. I. LEVY.

Solutions of quinine in ethylurethane. II. M. GIORDANI (Annali Chim. Appl., 1928, 18, 479—485; cf. A., 1928, 907).—Spectrographic measurements in the ultra-violet show that dissolution of quinine hydrochloride in presence of ethylurethane is accompanied by the formation of molecular complexes, which cause a marked change in the absorption spectrum, and confirm the view that the product of the transformation is quinotoxin (cf. Schmidt, Physikal. Z., 1900, 1, 466; Fischer, "Die ultravioletten Absorptionsspektren der China-alkaloide," 1925).

T. H. POPE.

Stability of solutions of cocaine and ψ -cocaine.

E. SADOLIN (Dansk Tidsskr. Farm., 1928, 2, 309—315; cf. A., 1927, 264).—A 1% solution of cocaine hydrochloride undergoes hydrolysis to the extent of only 10% on keeping for a month at the ordinary temperature, and no further hydrolysis takes place within a year; the results are the same if either water or 0.0001*N*-hydrochloric acid is employed as solvent. Cocaine is more resistant to alkalis than ψ -cocaine. The latter is completely hydrolysed on shaking with 0.1*N*-sodium hydroxide for 5 hrs. at 20°, whilst cocaine requires 9—10 hrs. under similar conditions. Using 1% sodium bicarbonate solution in place of sodium hydroxide, 20% of the cocaine and 30% of ψ -cocaine is hydrolysed at the end of the above periods.

H. F. HARWOOD.

Strychnine hydrochloride. J. E. DRIVER and S. P. THOMPSON (Quart. J. Pharm., 1928, 1, 37—43).—Strychnine hydrochloride crystals, in equilibrium with moist air at the ordinary temperature, have the composition $C_{21}H_{22}O_2N_2 \cdot HCl \cdot 1.75H_2O$; commercial samples contain $> 1.5 < 1.75 H_2O$. The solubility of the salt is greatly diminished by the presence of hydrochloric acid or metallic chlorides. The solubilities in water and hydrochloric acid solutions have been determined.

CHEMICAL ABSTRACTS.

Microchemical reactions of scopolamine. M. WAGENAAR (Pharm. Weekblad, 1928, 65, 1226—1227).

—The most sensitive reactions are those with bromine and gold chloride. The crystals obtained are described.

S. I. LEVY.

Pharmacological assay of digitalis. J. W. TREVAN, E. BOOCK, J. H. BURN, and J. H. GADDUM (Quart. J. Pharm., 1928, 1, 6—22).—Agreement between the results obtained by various methods was fairly good with a strong leaf, but unsatisfactory with a weak leaf. The international standard powder is a practical standard for digitalis leaf and tincture in Great Britain.

CHEMICAL ABSTRACTS.

Lemon oil. J. PRITZKER and R. JUNGKUNZ

(Pharm. Acta Helv., 1928, 3, 79—83; Chem. Zentr., 1928, ii, 193).—Fresh or suitably preserved lemon oil has n_D^{20} 70—76; values above 77 indicate that the oil is old and altered, unless it is free from terpenes. Old lemon oil changes colour and deposits a brown mass; with hydrochloric acid, d 1.19, the acid layer becomes dark brown or black, and the oil layer brownish. A residue on steam distillation of over 4% is abnormal. The normal Fehner-Hue value is 0.6—1.2.

A. A. ELDRIDGE.

Coumarin and umbelliferone methyl ether in lavender products. A. ELLMER (Riechstoffind., 1927, 206—210, 220—222; Chem. Zentr., 1928, i, 268).

—An extracted lavender oil, on extraction with a concentrated aqueous barium hydroxide solution, gave 0.9% of umbelliferone methyl ether and 3.4% of coumarin, whereas a distilled oil on similar treatment gave only 0.8% of coumarin. Probably the difference is due to the destruction of enzymes by steam-distillation, and the coumarin present in the distilled oil represents that originally formed together with that liberated by the boiling water. In the extracted oil further action of the enzymes on the glucoside is still possible, thus accounting for the higher yield of coumarin. A method for the isolation and determination of coumarin by means of concentrated aqueous barium hydroxide solution is described.

E. H. SHARPLES.

Caucasian bay leaf oil. B. RUTOVSKI and N. MAKAROVA-SEMLJANSKAJA (Riechstoffind., 1927, 218—219; Chem. Zentr., 1928, i, 268).—An average sample of the oil, obtained in a yield of 0.915%, had d^{20} 0.9167, α_D —19.6°, n_D^{20} 1.4690, acid value 1.1, ester value 47.6, ester value after acetylation 67.3, solubility in 90% alcohol 1 in 0.4 vol., in 80% alcohol 1 in 2 vols. It contained cineole (50%), eugenol (benzoyl derivative, m.p. 69°), acetyleugenol, α -pinene (nitrochloride, m.p. 99°; semicarbazone of the pinonic acid, m.p. 201°), l - α -terpineol (nitrochloride, m.p. 99°; nitropiperidide, m.p. 151°; phenylurethane, m.p. 110°), and a sesquiterpene, b.p. 118—122°/12 mm., d^{20} 0.9335, α_D up to +16.8°, n_D^{20} 1.4902 (nitrosochloride, m.p. 113°).

E. H. SHARPLES.

Essential oil of *Cachrys alpina*, MB. B. RUTOVSKI and J. VINOGRADOVA (Riechstoffind., 1927, 194—195; Chem. Zentr., 1928, i, 268).—Steam-distillation of the fresh material gave 0.98% (2.7% on dry plants) of oil, having d^{20} 0.8456, α_D +4.04°, n_D^{20} 1.4868, acid value 0.26, ester value 3.9, ester value after acetylation 15.23, and was insoluble in 90% alcohol. The half-dried roots of the plant gave 0.4% of oil having d^{20} 0.8671, α_D +6.64°, n_D^{20} 1.4972, acid value 1.6, ester value 4.56, ester value after acetylation 67.62, solubility in 90% alcohol 1 in 0.1 vol. Oil was obtained from the above-ground part of the plant in a yield of 0.8—1.52% on the dried material; it had d^{20} 0.8618, α_D 4.16°, n_D^{20} 1.4880, acid value 0.79, ester value 11.88, ester value after acetylation 29.87, and was insoluble in 90% alcohol. It contained pinene, limonene, terpinolene, cymol, and a terpene and sesquiterpene of unknown constitutions.

E. G. SHARPLES.

Determination of caffeine in tea. GOBERT.—See XIX.

PATENTS.

Preparation of a nutrient medium [for absorption through the skin]. K. STEJSKAL (B.P. 271,120, 17.5.27. Austr., 17.5.26).—The product consists of a non-aqueous emulsion containing a fat or oil, *e.g.*, olive oil, and a water-binding liquid capable of being consumed by the body, *e.g.*, glycerol, together with carbohydrates and/or proteins or their decomposition products.

L. A. COLES.

Production of trimethylamineglycol monoborate. K. LÜDECKE (B.P. 293,438, 9.2.28. Ger., 8.7.27).—An aqueous solution containing 1 mol. of trimethylamineglycol and 1 mol., or between 1 and 3 mols., of boric acid is concentrated to a syrup at about 30–60° *in vacuo*, and trimethylamineglycol monoborate is deposited in a crystalline form (+1H₂O), m.p. above 300°, by the addition of a liquid in which it is insoluble, *e.g.*, acetone. Alternatively, the syrup is dissolved in alcohol before the addition of the acetone, or the original constituents are brought to reaction in alcohol instead of in water. The product is of therapeutic value.

L. A. COLES.

Phosphatides (B.P. 285,417).—See XIX.

XXII.—EXPLOSIVES; MATCHES.

Effect of compression on the explosive properties of explosive gas mixtures. A. HAD and A. SCHMIDT (Z. angew. Chem., 1928, 41, 1309–1312).—The explosion pressures and brisance values for mixtures of oxygen with hydrogen (1:2), methane (2:1), ethylene (3:1), and acetylene (2.5:1) at ordinary pressure, and also when compressed at 600 atm., show that the compressed mixtures are as powerful as the ordinary solid explosives. Their brisance is comparable with that of liquid air explosives. Any practical application is, however, excluded by the difficult and dangerous technique. The uncompressed gases are very inferior in brisance to the solid explosive.

S. BINNING.

Stabilisation of nitroglycerin powders by diethyldiphenylurea. LÉCORCHÉ and JOVINET (Compt. rend., 1928, 187, 1147–1148).—Notes from a paper already abstracted (B., 1928, 656).

S. BINNING.

Determination of stability of smokeless powder and guncotton by measurements of hydrogen-ion concentrations. N. L. HANSEN (Förh. III nord. kemistmötet, 1928, 227–230).—A method for determining the stability of explosives of the nitrocellulose class has been devised based on the formation of acid when the explosive undergoes slow decomposition. The material (in the case of smokeless powder, after sifting to obtain grains of 0.35–0.8 mm. diam.) is dried *in vacuo* at 40° for 8 hrs., or, in the case of guncotton, until the water content is less than 1%. 5 g. of smokeless powder or 2.5 g. of guncotton are weighed out into each of nine glass cylinders, closed by rubber corks carrying a capillary tube and stopcock. Eight of the cylinders are placed in a calcium chloride bath heated at 110°, and after immersion for 15 min. any condensed moisture is removed. One cylinder is removed each hour, filled with distilled water (free from carbon dioxide) after cooling, and the p_H value of the solution

determined at once without previous filtration, using a quinhydrone electrode; the p_H value of the ninth (unheated) sample is similarly determined. The results are plotted, using p_H values as ordinates and the times of heating in hours as abscissæ, the curves obtained indicating the relative stability of the material under examination. Experiments show that in respect to the decomposition of the explosive, 8 hrs.' heating under the above conditions corresponds to upwards of 30 years' magazine storage at 10°. H. F. HARWOOD.

Determination of the friction sensitivity of ignition materials. H. RATHSBURG (Z. angew. Chem., 1928, 41, 1284–1286).—The apparatus consists of a mechanically rotated pestle of steel or porcelain, which turns in a mortar, and is held down by a load of lead weights variable at will. The rate of rotation being kept constant, the friction is maintained for constant short periods under successively increasing loads until detonation occurs. The load required is found to be constant for the same material. With variable rates of rotation, the load required varies; curves are constructed which are characteristic for each material.

S. I. LEVY.

Flame movement in gaseous explosive mixtures. ELLIS.—See II.

PATENTS.

Digestion of nitrocellulose. HERCULES POWDER Co., Assees. of M. G. MILLIKEN (B.P. 301,267, 20.4.28. U.S., 23.2.28).—Nitrocellulose is digested by being forced under pressure in aqueous suspension through a long pipe of small diameter. The suspension contains 1 pt. of nitrocellulose to 25 pts. of water, the pressure is 80 lb./in.², and the pipe is 1600 ft. long and 3½ in. diam. The temperature of the suspension just after entering the pipe may be raised to 160° by injecting steam into a steam jacket surrounding the pipe.

S. BINNING.

Manufacture of nitroglycerin and the like. F. E. SMITH, A. P. H. DESBOROUGH, W. T. THOMSON, W. LEDBURY, and E. W. BLAIR (B.P. 301,112, 26.8.27).—The nitration of polyhydric alcohols such as glycerin, glycol, or their ether derivatives is carried out in two stages either by adding the alcohol to pure nitric acid and then separating the nitrated compound by adding in one or more portions the correct amount of sulphuric acid, or by dissolving the alcohol in sulphuric acid and then precipitating the nitrated compound by adding nitric acid.

S. BINNING.

Mixing of liquids (B.P. 299,942).—See I.

XXIII.—SANITATION; WATER PURIFICATION.

Alkaline digestion of sewage grease. S. L. NEAVE and A. M. BUSWELL (Ind. Eng. Chem., 1928, 20, 1368–1369).—The lower fatty acids present in sewage sludge ferment readily under anaerobic conditions over the p_H range 7.2–7.6. With the exception of formic and acetic acids, 1 g. of fatty acid yields 1 g. of gas. The destruction of 1 g. of fatty acid is accompanied by the liquefaction of 25–40 mg. of solid nitrogenous matter to supply the metabolic needs of the organisms. This amount is ample for their needs, as the further addition of nitrogen in various forms has an inhibitory

effect. The increased rate of evolution of gas obtained when the temperature is raised to 37° is due mainly to the accelerated fermentation of sewage solids other than grease. C. JEPSON.

Causes and prevention of hydrogen sulphide in abattoir sewage. A. B. PORTER and J. A. CRESSWICK (J.S.C.I., 1928, 47, 380—382 r).—The presence of hydrogen sulphide in abattoir wastes, consisting of a dilute saline solution of blood together with protein matter in solution and suspension, is due to the action of *B. coli communis*. The formation of the gas may be minimised by (a) discharge to the sewer of all wastes in a fresh condition and as cold as possible, (b) removal of the coarse material obtained from the stomach and colon before discharge to the sewer, and (c) reduction of inoculating agencies by sterilisation of plant when out of operation. C. JEPSON.

Fertilising value of Baltimore sewage sludge. C. E. KEEFER and G. K. ARMELING (Eng. News-Rec., 1928, 101, 837—840).—Digested sewage sludge is shown by field experiments to have a definite manurial value though inferior to artificial or farmyard manure. The effect persists into the second year, suggesting the slow availability of the nitrogen. Analysis of the dry material indicates nitrogen (as NH_3) 2.2%, phosphoric acid (as P_2O_5) 0.5%, potash (as K_2O) 0.2%, fats 9.8%. C. JEPSON.

Determination of phenol in sewage. H. DEHE (Chem.-Ztg., 1928, 52, 983—985).—Two new methods are described. To 200 c.c. of the sample are added 10 c.c. of saturated zinc acetate solution, 40 c.c. of silver nitrate solution (5%), and 10 c.c. of sulphuric acid (1:3). After being kept in a closed flask with occasional vigorous shaking for 3—5 hrs., one half of the total volume is filtered and acidified with sulphuric acid; a few drops of hydrogen peroxide are added, and the mixture is steam-distilled until the distillate shows no trace of phenol. The distillate is made alkaline with caustic soda and concentrated, and when cold is made up to 500 c.c. The phenol present may be determined (a) iodometrically, or (b) by fractional titration. In (a) 10 c.c. of the alkaline distillate are diluted, warmed to 50—60°, treated with 15 c.c. of 0.1*N*-iodine, and when cool made acid with 15 c.c. of sulphuric acid (1:3); the excess of iodine is titrated back with 0.1*N*-thiosulphate. Each c.c. of iodine absorbed is equivalent to 1.567 mg. of phenol. In (b) 55—60 c.c. of the alkaline distillate are treated with a little solid baryta and after several minutes 25 c.c. of clear liquid are obtained, to which 10 drops of sulphuric acid are added and the barium sulphate is collected on a filter, the paper being washed two or three times with the least possible quantity of water (free from carbon dioxide). Exactly 0.2 c.c. of alizarine-yellow (0.1%) is added and the mixture brought to p_{H} 11.04 with the aid of a suitable buffer solution. Exactly 0.1 c.c. of phenolphthalein (1%) is added and the solution is titrated with 0.1*N*-sulphuric acid to p_{H} 8.4, again using a suitable buffer solution. From the amount of sulphuric acid required, less that required for a blank determination using distilled water, the quantity of phenol present (mg.) can be obtained by multiplying by 9.4. C. JEPSON.

Vapour pressure of fumigants. I. Methyl, ethyl, isopropyl, and *sec*.-butyl chloroacetates. II. Methyl, ethyl, *n*-propyl, *n*-butyl, *sec*.-butyl, and isobutyl formates. O. A. NELSON (Ind. Eng. Chem., 1928, 20, 1380—1382, 1382—1384).—The vapour pressures, and vapour-pressure curves and equations, of methyl, ethyl, isopropyl, and *sec*.-butyl monochloroacetates, and of methyl (b.p. 31.6°), ethyl (b.p. 53.8°), *n*-propyl (b.p. 80.4°), isopropyl (b.p. 68.4°), *n*-butyl (b.p. 105.6°), *sec*.-butyl (b.p. 93.6°), and isobutyl (b.p. 99.9°) formates have been determined by the Smith and Menzies method. The weights of each substance mentioned necessary to saturate a 1000-litre or 1000-cub. ft. chamber at temperatures from 20° to 50° are also given. E. H. SHARPLES.

Rapid determination of total hardness [of water] by separate determination of lime and magnesia hardness. K. V. LUCK and H. J. MEYER (Z. angew. Chem., 1928, 41, 1281—1284).—Calcium is precipitated by excess of potassium oxalate in neutral solution, and the precipitate decomposed by sulphuric acid and titrated in the usual way with permanganate. The procedure described is found to involve no appreciable error as compared with the usual precipitation from ammoniacal solution containing ammonium salts, and the excess of oxalate in the filtrate does not interfere with the subsequent determination of magnesium. This is carried out by pouring on to the surface of the cooled filtrate an alcoholic 8-hydroxyquinoline solution, then raising to b.p., separating the precipitate, dissolving in hydrochloric acid, and determining the quinoline compound with bromide-bromate solution in the usual manner. S. I. LEVY.

Effect of sunlight on the determination of chlorine [in water] by the *o*-tolidine method. F. R. McCURMB and W. R. KENNY (J. New England Water Works' Assoc., 1928, 42, 410—413).—Direct sunlight causes a rapid production of colour in this test and later a fading effect, so that if the usual fixed period of 5 min. elapses before the colour is read the results are low. Direct sunlight should therefore be avoided and the colour read at the time of maximum intensity. It is advisable to keep samples of chlorinated water in brown glass bottles so as to avoid reduction of chlorine concentration due to exposure to light. C. JEPSON.

PATENTS.

Apparatus for purifying water and subjecting it to radium emanations. F. L. MAIN (B.P. 302,095, 19.3.28).—A cylindrical stoneware vessel contains a perforated cup filled with filtering material resting upon shoulders about half way down the walls, and a porous jar sliding inside the vessel and resting upon the cup. The jar contains layers of filtering material separated by porous plates. A porous container filled with radioactive material rests in the lower part of the vessel, where the filtered water collects. The jar, plates, and container are made by compressing and heating at about 1370° a mixture of 3 pts. of diatomaceous earth and 1 pt. each of powdered silica and pumice stone. L. A. COLES.

Liquid insecticide (B.P. 285,797).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEBRUARY 15, 1929.

I.—GENERAL; PLANT; MACHINERY.

Nomography. V. O. LIESCHE (Chem. Fabr., 1928, 583—584, 595—597, 621—623; cf. A., 1928, 983).—Nomograms on two parallel scales and an intermediate scale inclined at an angle to the other two effect graphical solutions of equations of the type $u/v = w(s-w)$, e.g., for bimolecular reactions. By the use of an unevenly subdivided middle scale, equations of the type $u/v = q$, e.g., gas equations, may be solved, and if one of the parallel scales is logarithmic a solution is afforded of equations of the type $\delta = e^{-t/r}$, (radioactive disintegration). Examples of the use of logarithmic scales in determining the reaction velocity of unimolecular reactions and of nomograms for reducing the volume of a gas to N.T.P. are given. A. R. POWELL.

Nomography. IX, X, XI. O. LIESCHE (Chem. Fabr., 1928, 478, 490—492). XIII—XVI. R. KEMPF (*Ibid.*, 560—561)—IX. The diameter in mm. of a wrought-iron shaft transmitting H horse power at n r.p.m. is given by $d = 120 \sqrt[3]{H/n}$. This equation is represented as a nomogram of three parallel scales.

X—XI. The temperature-time relations in the heating or cooling of a system are given by $\log(1-d) = -\log 2z/h = -\log ez/t$, where d is the proportion of heat transmitted, z is the time, t the temperature, and h is the time in which half the temperature difference has been transmitted. By means of a nomogram h can be determined. The case of a liquid brought to the b.p. is slightly different, requiring two intermediate points. A similar treatment is suitable for numerous other processes.

XIII—XVI. Rectilinear nomograms are given for the determination of the correction to be applied to thermometric readings for the exposed thread of mercury, and to b.p. determinations for variation in the atmospheric pressure. C. IRWIN.

Nomography. XII. O. LIESCHE (Chem. Fabr., 1928, 531).—The construction of a nomogram is described for determining the gas velocity V in a chimney stack from the equation V (m./sec.) $= \phi \sqrt{[2gH(t_2-t_1)/(t_1+273)]}$, where H is the height of the stack in m., t_2 is the temperature of the flue gases, and t_1 the temperature of the air. A. R. POWELL.

Recrystallisation. H. DANNEEL (Chem. Apparatur, 1927, 14, 193—194; Chem. Zentr., 1927, II, 2623).—An apparatus for the continuous recrystallisation of a salt or organic compound comprises two containers connected with one another at the top and at the bottom with siphons. Both vessels are filled with a saturated solution of the salt, and into one vessel con-

tinuous supplies of the crude salt are introduced; this vessel is heated while the other is cooled. In this way a circulation of the liquid is set up, such that cold saturated liquor is continually returning to the hot vessel and hot, saturated liquor passing to the cold vessel, where it is cooled with the deposition of the purified salt. By varying the difference in temperature between the two vessels the size of the crystals can be regulated, large crystals being obtained when the temperature difference is small. A. R. POWELL.

Measurement of gas temperatures up to 1500° in radiation fields of varying anisotropy. H. SCHMIDT (Arch. Eisenhüttenw., 1928—9, 2, 293—299; Stahl u. Eisen, 1928, 48, 1825—1826).—A modified head for a radiation gas pyrometer is described briefly and illustrated, and some examples are given of its use in the Siemens-Martin furnace. A. R. POWELL.

Chemical and physical properties of chemical warfare materials. E. ENGEL (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 321—325, 365—367, 402—407, 438—442).—A summary is given of the physical properties on which the effective utilisation of chemical warfare materials depends. The data given for some of the more important include b.p., sp. wt. and vol., coefficient of expansion, vapour pressure, volatility, and heat of evaporation. The physics of cloud and fog formation is discussed, and a short account is given of the absorption of toxic vapours by activated charcoal. S. BINNING.

See also A., Jan., 26, Colloidal solutions and sedimentation (SVEDBERG). 44, Toy balloons and filtration (GARRETT and HURD). Washing by decantation (MARSH). Viscosimeter (LASZLO).

PATENTS.

Retort construction. R. B. PARKER (B.P. 282,418, 14.12.27. U.S., 14.12.26).—A vertical retort is provided with openings through the upper part of the walls for withdrawal of gaseous products; the openings at each side are adjustable separately by means of gates, so that when channelling takes place up one side the gates at that side may be closed and those at the other side may be opened more. Ducts for heating medium are provided in the thickness of the lower parts of the walls. B. M. VENABLES.

Regenerative heat-exchange apparatus. AKTIEBOLAGET LJUNGSTRÖMS ÅNGTURBIN (B.P. 291,402, 26.5.28. Swed., 1.6.27).—In a regenerator, preferably of the rotating type, the regenerative mass comprises strips or pipes or a number of corrugated plates which are constructed of different materials according to their position in the regenerator. B. M. VENABLES.

Heat-exchange apparatus. A.-G. BROWN, BOVERI & Co. (B.P. 300,543, 5.7.28. Ger., 14.11.27).—The tubes of a heat interchanger are placed at an angle of less than 45° to the axis of the inlet and outlet of the outer fluid.

B. M. VENABLES.

Balanced ball mill. W. M. BAKER (U.S.P. 1,692,974, 27.11.28. Appl., 1.9.26).—The mill is cylindrical and runs on tyres. The grinding chamber is annular and contains loose balls or the like, and may be subdivided into several chambers at about equal distances from the axis. Nearer to the axis are a feed chamber at one end and discharge chamber at the other end, divided from each other by a transverse wall. Ports permit passage of material outwards from the feed chamber to the annular grinding chamber or chambers, and from it or them inwards to the outlet chamber. Auxiliary inlets for air are provided direct through the end wall to the grinding chamber. The use of balls etc. in the feed and discharge chambers is not excluded.

B. M. VENABLES.

Ring mill. J. MOLZ, Assr. to BABCOCK & WILCOX Co. (U.S.P. 1,693,247, 27.11.28. Appl., 6.7.23. Ger., 10.7.22).—A stationary grinding ring is provided with an inlet port for material through the grinding surface. The grinding rollers are slidably journaled in a positively-driven frame.

B. M. VENABLES.

Grinding mills. A. E. G. MACCALLUM, and S. SMITH & BLYTH, LTD. (B.P. 301,348, 26.8.27 and 5.4.28).—Various forms of reciprocating scraper are described for use in a paint mill or other type of roller mill.

B. M. VENABLES.

Grinding mills. A. E. G. MACCALLUM (B.P. 301,781, 5.9.27).—In a roller mill for paint and similar materials, a method of holding the grinding bar so that it is absolutely parallel to the roller and easily detachable is described.

B. M. VENABLES.

Dryer. E. P. DECKER (U.S.P. 1,693,747, 4.12.28. Appl., 17.11.27).—The deck or table supporting the material comprises heated imperforate sections alternating with unheated screen sections. Both are inclined, co-planar, and kept in vibration.

B. M. VENABLES.

Apparatus for purifying crude salts by recrystallisation. GES. F. LICHT, WASSER, U. HEIZANLAGEN M.B.H. (G.P. 450,809, 5.8.26).—The apparatus comprises a series of vertical cylinders standing above a collecting tank. The hot solution from the saturators is passed through small pipes into the inner cylinders so that it swirls round the walls in a helical manner and finally passes into the tank where the crystals settle out. The cold mother-liquor is pumped back to the saturators upwards through the annular space between the two cylinders, thus serving as the cooling medium for the hot liquor and being preheated itself. A collapsible scraper removes the incrustation of salt from the inner walls of the inner cylinder during its downward movement, but folds like an umbrella in its upward movement.

A. R. POWELL.

Distillation. INTERNAT. COMBUSTION ENGINEERING CORP., Assees. of W. RUNGE (B.P. 277,952, 6.9.27. U.S., 21.9.26).—The heating means of a still for tar and other materials comprises a hollow member, through which

the heating medium is passed, rotating in the still. The hollow member may be star-shaped and covered on the outside with removable plates. The material to be distilled is preferably sprayed on the heater in such a way that distillation takes place during 180° of rotation, whilst the non-volatile residue runs off during the remainder of a revolution. The heating gases which have passed through the still may be utilised to preheat the material coming in and/or to control the temperature of a number of condensers in series with the outlet of the still.

B. M. VENABLES.

[Rotary drum] filters. H. NOTZ (B.P. 301,541, 1.9.27).—A form of construction is described involving the use of parts of stamped sheet metal welded together, whereby the filter may be homogeneously lead-covered or constructed of aluminium, stainless iron, etc.

B. M. VENABLES.

Rotating filter drum without cells. P. PAPE (G.P. 450,664, 21.11.24).—The filter comprises a horizontal, rotary, perforated cylinder enclosing a hollow suction cylinder for collecting the filtrate. This cylinder occupies almost the whole of the space inside the perforated cylinder. It is provided with an opening at one end through which suction is applied, and with a lever rod with interchangeable loading plates, so as to counterbalance the weight of the suction cylinder and its liquid contents and to keep the cylinder tight against the walls of the outer perforated drum.

Filter for gases. J. WANGER (G.P. 450,912, 5.4.24).—The apparatus comprises a long, narrow, vertical flue with permeable side walls through which the gas stream passes from side to side alternately in opposite directions from the bottom to the top, the area of the gas conduit decreasing from the bottom upwards. A constant stream of moist packing material is slowly passed down the vertical flue so that the gas stream in its passage back and forth through the flue meets material of gradually increasing adsorptive power for the impurities.

A. R. POWELL.

Separation and liquefaction of gases. W. S. BOWEN (U.S.P. 1,694,308, 4.12.28. Appl., 11.1.27).—A column of gaseous mixture is expanded through a small aperture, whereby one constituent is liquefied, the aperture is kept drowned in the liquefied constituent, and the incoming column of gases cooled by heat-exchange with both the liquefied portion and the gas evolved therefrom.

B. M. VENABLES.

Colloid mill. A. FRASER (U.S.P. 1,696,083, 18.12.28. Appl., 7.8.25. U.K., 16.8.24).—See B.P. 243,052; B., 1926, 80.

Cooling brines (B.P. 294,595).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Composition of coal: the active-decomposition point of coal. R. HOLROYD and R. V. WHEELER (J.C.S., 1928, 3197—3203).—Finely-ground, 250 g. samples of various coals have been heated at different temperatures for 120 hrs. under a vacuum, and the solid, liquid, and gaseous products of decomposition examined. As distinct from the decomposition of minor ingredients of the coal conglomerate, an extensive decomposition

of the major constituent of bituminous coals (the ulmin compounds) can be recognised. The temperature at which this active decomposition begins varies with different coals, but is constant for a given coal within $\pm 2^\circ$ under standard conditions of heating. Fundamental changes in the properties of the coals are produced when the active decomposition point, which marks the breakdown of the nuclear structure of the ulmin compounds, has been passed. In general, the active decomposition point varies with the carbon content of the coal, being higher the higher is the carbon content. Thus, with a coal containing 77% C the decomposition point was $290\text{--}300^\circ$, whilst with one containing 89.8% C it was $360\text{--}365^\circ$. Results are given for thirteen coals. F. J. DENT.

Modification of Thomson's mixture for determining the calorific value of coal. M. COUTURE (Atti II Cong. Naz. Chim. pura appl., 1926, 1374—1375; Chem. Zentr., 1928, ii, 613).—A mixture (18 g.) of potassium chlorate and nitrate with ammonium nitrate (1 g.) is used for 1 g. of coal. A. A. ELDRIDGE.

Efficient dehydration of [coal] gas at low cost. G. A. BRAGG (Chem. Met. Eng., 1928, 35, 731—733).—The cost of the drying of coal gas to a dew point of 13° in summer and 0° in winter by various methods is calculated. The cost by the calcium chloride method is 0.67 cent/1000 cub. ft., and by direct cooling with refrigerated water about 0.90 cent using a compression-type refrigerating machine. In this latter case complete removal of naphthalene is necessary, and is not included in the cost, and the lowest dew point is about 5° . Where compressors already exist for high-pressure distribution, dehydration by additional compression will average 0.80 cent/1000 cub. ft. Such compressors, unlike other dehydration plant, will be located after the holder, and must therefore be large enough to deal with peak loads. C. IRWIN.

Comparative costs of gas production in the retort house. E. W. SMITH (Gas J., 1928, 184, 845—848).—The influence of carbonising conditions, capital, labour, and fuel costs, and market prices for coal, coke, and tar on the net cost of a therm of gas is summarised. The most important factors are the value of coal and coke, fuel requirements for producers, and distribution charges. R. H. GRIFFITH.

Tar from peat of Ostashkov district. B. V. MAXAROV (J. Chem. Ind. Moscow, 1927, 4, 755—758).—The tar has d_{45}^{15} 0.9554, contains C 77.85, H 10.07, N 2.95, S 2.64, O 6.49%, and differs from ordinary peat tar by its strongly alkaline reaction and abundance of nitrogen compounds. Fractionation affords oil 65.9, residue 26.1, gas and water 8%. Of the oil, 1.3% has b.p. $<150^\circ$; the fraction b.p. $190\text{--}200^\circ$ consists of cresols and xylenols; the heavy fraction affords crystals of paraffin, m.p. 62.1° , d_{20}^{20} 0.917. The tar oil contains phenols 11.1, organic acids 1.6, organic bases 7.6, neutral oxygen and sulphur compounds 3.7, pyrroles, indoles, etc. 6.4, solid paraffin 5.9, liquid paraffin hydrocarbons 27.2, cyclic hydrocarbons 18.6, unsaturated hydrocarbons 17.9%.

CHEMICAL ABSTRACTS.

Occurrence of higher saturated fatty acids in

natural petroleum and origin of petroleum. Y. TANAKA and T. KUWATA (J. Fac. Eng. Tokyo Imp. Univ., 1928, 17, 293—303).—The high-boiling fraction of the crude petroleum acids (0.135% of distillate) obtained during the refining of a gas-oil distillate of Ishikari (Hokkaido) origin contains in addition to naphthenic acids a mixture (7.7% of total acids) of palmitic (4.5 pts.), stearic (2 pts.), myristic (2 pts.), and arachidic acids (traces), separated through the lithium salts and subsequently the methyl esters. Similar fatty acids occur in Kurokawa, Katsurane, Nishiyama, Kubiki, California, and Borneo petroleums. The occurrence of fatty acids in petroleum favours the animal origin theory, and it is suggested that the source is from whales and sharks of the Mesozoic and Tertiary periods. H. BURTON.

Choice of cracking process [for petroleum oils]. E. OWEN (Chem. Met. Eng., 1928, 35, 737—740).—The Cross, Holmes-Manley, and tank-and-tube cracking processes, which all operate in the liquid phase, are described and details of yields given. The pressure, temperature, and details of the apparatus show variations, but in all cases the cracking coil is followed by a reaction chamber or "soaking drum," in which the hot compressed oil is retained for some time to allow the reaction to proceed. Coke and tar are withdrawn and the product is fractionated, the gas oil being returned to process. The gas produced is usually used for heating the cracking coil. Present-day cracked gasoline is fully equal to straight-run gasoline in all respects, and, owing to its greater content of unsaturated hydrocarbons, has better anti-knock properties. C. IRWIN.

Action of aluminium chloride on olefinic hydrocarbons. A. SZAYNA (Przemysl Chem., 1928, 12, 637—647).—Ethylene, propylene, isobutylene, *n*-octene, and diamylene were heated with anhydrous aluminium chloride at 100° and at 150° . The gaseous product is butane, with the admixture of small quantities of propane in the cases of propylene and diamylene at 150° . The liquid products are low b.p. paraffins and higher b.p. naphthenes in various proportions, and a compound $(\text{AlCl}_3, \text{C}_4\text{H}_8)_n$, from which an oil, $(\text{C}_8\text{H}_{12})_x$ or $(\text{C}_8\text{H}_{10})_x$, is obtained. This oil readily absorbs atmospheric oxygen, and consists of unsaturated cyclic hydrocarbons. R. TRUSZKOWSKI.

Benzine for precipitation of asphaltenes from mineral oil. II. H. BURSTIN and J. WINKLER (Przemysl Chem., 1928, 12, 581—583).—The necessity of superseding Holde's standards for benzine for the precipitation of asphaltenes by those previously described (B., 1929, 6) is demonstrated by showing that it is possible to prepare three different reagents which conform to Holde's standards, each of which has a different precipitating power. R. TRUSZKOWSKI.

Working-up turbine oil distillate for white oil. S. A. NAZAROV (Neft. Choz., 1928, 14, 772—774).—By twelve successive treatments of turbine oil distillate at a low temperature with small portions of sulphuric acid containing 20% SO_3 (free), neutralisation with sodium hydroxide (d 1.032), treatment with fuller's earth, and pressing at 70° , d_{15}^{15} is decreased from 0.894 to 0.873, the flash point raised from 184° to 187° , the iodine value

decreased from 1.643 to 0, the sludge from 4.8% to 0, and the absolute viscosity from 0.563 to 0.

CHEMICAL ABSTRACTS.

Maikop oils. A. N. SAKHANOV and M. A. BESTUZHEV (Neft. Choz., 1927, 12, 402—406).—Characteristics of four crude oils from the Maikop district (Shirvani, Khaduizhinski) are recorded. CHEMICAL ABSTRACTS.

Analysis of light gasolines. J. GIRZEJOWSKI (Przemysl Chem., 1928, 12, 584—586).—The volatile fractions of gasoline which do not undergo condensation in Engler's method are adsorbed by charcoal, and the gain in weight of the latter is divided by the sp. gr. of the gasoline to give the volume adsorbed. This fraction amounts to about 2% for gasolines beginning to boil at 50°, to 13.7% at 40°, to about 50% at 20°, and to 68% at -10°. The discrepancy observed between the volume of gasoline taken and that accounted for amounts to less than 1%.

R. TRUSZKOWSKI.

Refractometry of liquid mixtures with the Zeiss butyrorefractometer; investigations on certain motor spirits. P. FUCHS (Chem.-Ztg., 1928, 52, 921—922, 942—944).—The Zeiss butyrorefractometer, in which the scale is divided into 100 pts., is a suitable instrument for determining the proportions of benzene and benzol in their mixtures. The refractions for the pure constituents are outside the scale readings of the instrument, but may be obtained by extrapolation from the curve showing the increase of scale reading with increase of benzol in the mixture. In this way a curve is obtained for the complete range of mixtures of any benzene with benzol, by means of which variations of as little as 2% of the latter may be detected. Examination of a number of commercial motor spirits shows that they fall into two classes, those with an admixture of benzol and those without.

F. R. ENNS.

Crystallisation of paraffin. I. Crystal forms of paraffin from shale oil. II. Crystal system of paraffin from shale oil. III. Crystal forms and system of petroleum paraffin. Y. TANAKA, R. KOBAYASHI, and S. OHNO (J. Fac. Eng. Tokyo, Imp. Univ., 1928, 17, 275—292).—I. Paraffin, purified by washing with alcohol-ether the crude shale paraffin derived from Fushun oil shale, crystallised in plates and had d_{25}^{20} 0.7719, n_D^{25} 1.4292, and m.p. 60°. By fractional sweating a series of fractions consisting of normal hydrocarbons, m.p. range 57—64°, were obtained. Crystal forms obtained from alcoholic solution of shale paraffin showed interlaced needles to be generally produced at high concentration and high cooling velocity, whereas plate crystals are obtained at lower concentration and lower cooling velocity, and are considered as the result of the most stable arrangement of paraffin molecules. Low viscosity of the medium in which paraffin is dissolved favours the formation of plate crystals and high viscosity needle crystals. Thus by controlling either the viscosity of the paraffin distillate or its cooling velocity the mutual transformation of the two crystal types may be effected. The viscosity of paraffin distillate from Fushun shale oil is extremely low, hence the resultant paraffin crystals are naturally laminar. Of the various forms of shale paraffin crystallised from alcoholic solution, the simplest form is a perfect rhombic plate

having angles of 110° ($\pm 1^\circ$) and 70° ($\pm 1^\circ$). The more complicated plate and needle forms are formed by parallel growths of the rhomb, in the direction of the angle 110° in the case of plate forms and in the direction of the angle 70° in the case of needle forms.

II. A study of the optical properties and crystal system of crystals of shale paraffin shows the fundamental form to be a rhombic plate with each pair of angles 110° and 70°. Other complicated forms also belong to the rhombic system crystallographically and optically, and are produced by parallel growths of the fundamental rhomb. Needle crystals of paraffin are formed by successive parallel growths of the orthorhomb in the direction of the 70° angle, and show the same optical properties as the plate; it differs, however, in the angle of the optical axes.

III. Paraffins contained in petroleum crystallise generally in needle form from paraffin distillates except that from Formosa petroleum, which crystallises in plates. The difference in types depends on the viscosity of the distillates and not on the chemical nature of the paraffins. Increasing viscosity favours needle formation, decreasing viscosity plate formation, and by altering the viscosity of the distillates the two types are transformable. From the crystal form the suitability of the pressed oil for the preparation of viscous lubricating oils may be suggested. The forms of petroleum paraffin crystallised from alcoholic solution are similar to those from shale paraffin, both belonging to the rhombic system, the fundamental form being the rhombic plate with angles of 110° and 70°.

H. S. GARLICK.

Technique and economics of the Edeleanu process for refining mine al oils. G. CATTANEO (Petroleum, 1928, 24, 1634—1641).—A history of the development of the Edeleanu process for refining kerosene or lubricating oil, from earlier plants operated discontinuously to more recent continuous or semi-continuous plants based on the principles of counter-current extraction and multiple-stage evaporation of the sulphur dioxide solution of the extract.

W. S. NORRIS.

Lubricating oils. J. TAUSZ and M. DREIFUSS (Petroleum, 1928, 24, 1383—1412).—It is not possible to determine the angle of contact of lubricating oils with metals by the direct or indirect methods of von Dallwitz-Wegener (Petroleum, 1920, 16, 249). The surface tensions of a number of lubricating oils were determined from (a) the weight of a detached drop, (b) the total weight of a drop, and (c) the capillary rise. Methods (a) and (b) do not lead to correct results, since the viscosity of the oil tends to interfere with the formation of the drop. From 20° to 100° the surface tensions by method (c) are linear functions of temperature. It is impossible to determine the mol. wt. of lubricating oils from their surface tension, because K_E in the Eötvös-Ramsay-Shields equation is not constant. Given the mol. wt., it is, however, possible to evaluate K_E , and thus obtain an approximate value for the critical temperature and b.p. of oils, the b.p. of which can only be determined in the ordinary way with difficulty or not at all. With increasing mol. wt. (M) of the series of oils tested, the ratio γ_{20}/M decreases,

whilst the dielectric constant increases (cf. Walden, A., 1910, ii, 254). Surface tension and viscosity vary in the same sense with temperature (cf. Faust, A., 1926, 674), but the relationship found by Sharman (A., 1926, 464), $\log \gamma = A \log \eta + B$, was not applicable. Determinations of viscosity are best conducted by measuring the rate of outflow of oil from between highly polished, optically plane, metallic surfaces under a known load; the system oil-metal-oil forms an electrical condenser of which the alteration in capacity gives a measure of the outflow of oil during a given time interval. The time required for a given quantity of oil to be expressed increases with the viscosity, and is inversely proportional to the load. The Poiseuille equation is still valid for the oils investigated when the oil film is only 0.003 mm. thick. The velocity of sliding of a bronze plate over a steel plate lubricated by an electrically measured thickness of castor oil or a steam cylinder oil is more than doubled if the tractive load is doubled; the equation of Reiner (A., 1926, 678) is verified. The method gives a means for measuring the thickness of the adsorbed oil layer on a metal surface. A mineral lubricating oil is the more valuable the smaller is the value of $(\sqrt{K} - n_D)$ for the highest possible viscosity, K and n being dielectric constant and refractive index, respectively. W. S. NORRIS.

Insulating oils. N. A. BUTKOV (Izvestia Thermo-Tech. Inst. [Moscow], 1928, 6, 3—6).—The requirements of transformer oils are discussed. Although the presence in these oils of sulphonic acids is undesirable, since they favour the formation of sludge and increase the action on the metallic parts of the machines, tests in which an oil was heated for 5 hrs. at 120–150° in a bomb with an oxygen pressure of 11–13 atm. alone and with 1% of sulphonic acid show that the absorption of oxygen is negligible in both cases. Badly washed oils are hygroscopic, and rapidly absorb atmospheric moisture, with fall in the breakdown voltage of the oil; this voltage depends almost solely on the proportion of moisture present, and decreases rapidly as the percentage rises from 0 to 0.01, and afterwards more slowly. Results which are in the same order for a series of oils as those obtained by the ordinary oxidation test are furnished by the bomb test (as above), in which copper is introduced as an accelerator. After the oil has been heated with oxygen under pressure, it is diluted with the tenfold quantity of light petroleum; after 24 hrs. the precipitate formed is filtered off and weighed and the acidity of the filtrate is determined.

T. H. POPE.

The Conradson number [of lubricating oils]. W. J. PIOTROWSKI and J. WINKLER (Przemysl Chem., 1928, 12, 573–580).—This value for lubricating oils is never greater than 1% where the given oil is a distillate; for residues it varies from 1 to 7% for various Galician oils. The presence of fatty acids up to 10% does not affect the values obtained. The Conradson number of an oil is greater the greater the degree of carbonisation of the cylinders using this oil. Oils composed of naphthenes or aromatic hydrocarbons as well as those obtained by distillation in a high vacuum (3 mg.) have a lower Conradson number than those containing unsaturated aliphatic hydrocarbons. R. TRUSZKOWSKI.

See also A., Jan., 33, Ignition temperature of hydrogen-air mixtures (PRETTE and LAFFITTE).

Effects of coal ash on refractories. McVAY and HURSH.—See VIII. Corrosion of metal pipes by electric currents. BECK.—See X.

PATENTS.

Retorts for the distillation of solid carbonaceous substances. LOW TEMPERATURE CARBONISATION, LTD., and C. H. PARKER (B.P. 301,361, 27.5.27).—The combustion chambers of the retort setting described in B.P. 299,795 (B., 1929, 44) consist of a vertical, central wall, preferably perforated, and lateral walls, with spaces between the latter and the central wall for the passage of the flames and hot gases. The lateral walls, which separate the combustion chambers from the retort chambers, are constructed of box bricks, the cavities of which are disposed on the side of the wall facing the retort. At the base of the walls is a horizontal air-inlet passage, and lying immediately above this is a gas-inlet passage. These passages extend the length of the combustion chamber and are provided with outlets to the chamber at suitable intervals.

A. B. MANNING.

Furnace for the low-temperature carbonisation of coal. C. A. VEDENSKI (F.P. 626,847, 29.12.26).—The furnace consists of two chambers one above the other, the upper chamber serving to preheat the coal to its softening point and the lower to effect its destructive distillation.

A. R. POWELL.

Low-temperature carbonisation of fuels. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 301,975, 19.9. and 6.10.27).—Hot inert gases or vapours, which may or may not contain oxygen, are blown through the fuel in such quantity as to keep the charge in active movement on its bed at a temperature (400–600°) suitable for carbonisation; the conditions are such as to avoid combustion or gasification of the fuel to a greater extent than necessary for the carbonisation. Any dust produced is removed from the gases while they are still hot, e.g., by an electric filter in the upper part of the carbonisation shaft. The process is suitable for the preliminary carbonisation of fuel which is subsequently to be gasified, the carbonised product being then conveyed directly to the producer without cooling. The hot producer gases may be used, after suitable temperature adjustment, as the heating gases in the carbonisation process.

A. B. MANNING.

Treatment of coal and like carbonaceous material. C. LANDER, F. S. SINNATT, J. G. KING, and A. CRAWFORD (B.P. 301,720, 4.8.27).—Anthracite, bituminous coal, lignite, peat, or other carbonaceous material is subjected to the action of hydrogen at high pressures and elevated temperatures, but under conditions which result only in a partial hydrogenation of the material. By this treatment a feebly-coking or non-coking coal can be converted into a solid product which when carbonised yields a hard, non-swollen, or a highly swollen coke; by modifying the treatment any desired coking qualities may be imparted to the initial material. By carrying the process a stage further the coal may be converted into a mass which is plastic or fluid at higher temperatures, but which forms a solid pitch on cooling.

This product may be used as a binder in briquetting, or may be added to other carbonaceous material to confer caking power thereon; or the plastic material may be introduced, either at a temperature at which it will flow or admixed in a suitably divided condition with a liquid vehicle, into an apparatus in which it is further hydrogenated for the production of oils. The quantities of hydrogen absorbed in these processes vary from 0.4 to 1.5% of the ash-free dry material.

A. B. MANNING.

Processes for hydrogenating and splitting hydrocarbons, the distillation products of various coals and their constituents, as also the coals themselves.

A. L. H. SPILKER, C. ZERBE, and GES. F. TEERVERWERTUNG M.B.H. (B.P. 277,974 and Addn. B.P. 279,055 and 279,410, 21.9.27. Ger., [A] 23.9., [B] 18.10., [C] 20.10.26).—(A) Coal, pitch, or other coal distillation products are heated with hydrogen under pressures of 50–100 atm. at 300–500° in the presence of a small quantity (*e.g.*, 0.01% or less) of iodine, or of a compound splitting off iodine or hydrogen iodide at the temperature employed. Liquid hydrocarbons are formed which on fractionation yield motor spirits, Diesel engine oils, and lubricating oils. (B) In place of iodine or its readily dissociable compound the alkali or alkaline-earth iodides may be used in conjunction with metallic salts, *e.g.*, iron alum, ferric chloride, etc., which bring about the dissociation of the iodides at the temperatures employed. (C) Other halogen salts, *e.g.*, ammonium chloride, potassium bromide, etc., and especially mixtures of these, may also be used in the process.

A. B. MANNING.

Production of gases rich in olefines from bituminous coals. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,775, 1.9.27, 5.4. and 22.5.28).—The pulverised coal, preferably predried, is heated rapidly to about 800°, and the coke and gas formed are then quickly cooled again. The material may be allowed to fall through an externally-heated vertical tube, or may be carried through a suitable reaction chamber with the aid of superheated steam or other gases. A Cowper apparatus also forms an advantageous reaction vessel. To prevent the deposition of coal or coke within the heated chamber an inert solid substance, *e.g.*, sand, may be mixed with the fuel. Gases containing 20% or more of olefines are produced from brown coals by the process.

A. B. MANNING.

Manufacture of coal tar. F. COOKE (B.P. 301,645, 2.12.27).—Road tar is prepared directly in the coke-oven plant by drawing it from the air-cooled main at a point between the ovens and the extractors, the gases passing that point being maintained at a temperature between 160° and 190°. The crude tar is steam-washed, separated from ammonia liquor, and dried. Part of the crude tar, and also part of the lighter tar subsequently extracted from the gas, may be recirculated through the coke-oven gas mains. The naphthas and benzols are distilled from the oils separated from the tar by the steam-washing, and, if desired, part of the remaining creosote oil is added to the road tar.

A. B. MANNING.

Cracking of mineral oil. A. E. PEW, JUN., and H. THOMAS, Assrs. to SUN OIL Co. (U.S.P. 1,692,786,

20.11.28. Appl., 25.1.26).—Mercury vapour, under a pressure which maintains it in vapour form at a temperature above that at which substantial cracking of oil occurs, flows in heat-exchange relation with a continuous stream of oil at a lower temperature, so that cracking is effected by condensation of mercury vapour and transfer of its latent heat to the oil. Condensed mercury is returned to the vapour generator, and oil is removed continuously from the zone of heat exchange. The rate of flow of mercury vapour is restricted so that it is less where heat exchange occurs than where heat is generated.

H. S. GARLICK.

Cracking of hydrocarbons. GASOLINE PRODUCTS Co., Asses. of G. C. HARGROVE and W. B. MONTGOMERY (B.P. 290,996, 20.12.27. U.S., 23.5.27).—Heavy stock is continuously diluted with overhead distillate and passed through a cracking apparatus under such conditions of temperature, pressure, and time that not more than 25% is converted into light constituents in a single passage through the cracking system. A portion of the stock to be cracked is subjected to preliminary heating in one or more of the fractionating stages of the system, and is then made to assist in maintaining there the desired temperature. Light fractions are stripped from the cracking stock during a preliminary heating, and the fractions are led direct to the fractionating stages of the plant.

H. S. GARLICK.

Cracking and rectifying petroleum oils and the like. J. W. WADSWORTH (U.S.P. 1,692,476, 20.11.28. Appl., 12.3.26).—Gasoline is separated from the mixture of hydrocarbons and residue resulting from the cracking of petroleum oils under heat and pressure, and passed to a second stage where the pressure resulting from the cracking stage is utilised to effect rectification at super-atmospheric pressures and temperatures.

H. S. GARLICK.

Distillation of petroleum. H. E. HEWETSON, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,694,259, 4.12.28. Appl., 18.6.23).—A series of charges of petroleum are maintained at successively higher temperatures, and the vapours are passed to a condenser. A continuous stream of petroleum flows from each charge to that at the next higher temperature, each stream having a down-flowing and an up-flowing section into which a quantity of liquid of lower b.p. than that of the stream is introduced in order to produce a vapour commingling with the liquid in the up-flowing section.

H. S. GARLICK.

Apparatus for the catalytic conversion of hydrocarbons [into hydrogen and carbon monoxide]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,969, 15.9.27).—Hydrocarbons together with water vapour or carbon dioxide, with or without the further addition of air or oxygen, are passed over a catalyst, *e.g.*, nickel activated with alumina, arranged in a number of parallel tubes of highly alloyed steel, maintained at 600–1000° by a heating medium which does not come into direct contact with the catalyst. If necessary the hydrocarbons are previously freed from organic sulphur compounds. The process may be carried out in two stages if desired (*cf.* B.P. 288,662; B., 1928, 437).

A. B. MANNING.

Retorts for the treatment of oil shale and the like. T. M. DAVIDSON (B.P. 301,407, 25.8.27).—A rotary retort comprises an inclined cylinder supported upon rollers in a retort setting, the ends of the retort being respectively connected to charging and discharging boxes or chambers. A number of scraping devices moving circumferentially engage the inner surface of the retort, and are operated by a reciprocating member moving in a direction parallel to the axis of the retort. A gas off-take, consisting of a fixed tapering tube open at the inner end and provided with a number of inlet openings along its length, extends approximately to the middle of the retort from the discharge end. The feeding hopper inclines into the mouth of the retort and feeds through an adjustable valve on to the lower surface of the retort. Closure members are applied against the ends of the rotating retort in a resilient manner and are rendered gas-tight by means of a solid lubricant.

H. S. GARLICK.

Motor fuel [antidetants]. S. P. MARLEY and W. A. GRUSE, Assrs. to GULF REFINING Co. (U.S.P. 1,690,988, 6.11.28. Appl., 25.8.24).—A saturated alcoholic or benzene solution of two or more organic antidetants, having solubilities of the order of 0.5% in petrol, at least one of which is an amine, are added to petrol; e.g., 0.5% of hexamethylenetetramine in 5 pts. of alcohol and 0.1% of *m*-phenylenediamine in 5 pts. of benzene, or 0.5 pt. of *p*-iodoaniline and 0.5 pt. of *p*-anisidine in 5 pts. of benzene and 5 pts. of alcohol, are added to 90 pts. of petrol.

R. BRIGHTMAN.

Washing of coal, ores, and similar materials. L. HOVOIS (U.S.P. 1,696,767, 25.12.28. Appl., 18.2.27. Belg., 25.2.26).—See B.P. 266,723; B., 1928, 218.

Resolution of emulsions of tar or tar oils. H. W. ROBINSON and D. W. PARKER (U.S.P. 1,696,057, 18.12.28. Appl., 15.9.27. U.K., 24.12.26).—See B.P. 280,059; B., 1928, 81.

Fuller's earth treating process. R. R. ROSENBAUM (B.P. 284,327, 23.1.28. U.S., 29.1.27).—See U.S.P. 1,649,193; B., 1928, 45.

Retorts (B.P. 282,418). Distillation (B.P. 277,952).—See I. Catalyst (B.P. 301,402). **Halogenated hydrocarbons (B.P. 301,009). Alcohols (B.P. 301,000).**—See III.

III.—ORGANIC INTERMEDIATES.

Microchemical detection of some substances which yield volatile aldehyde or ketone with certain reagents. C. GRIEBEL and F. WEISS (Z. Unters. Lebensm., 1928, 56, 158—167).—The detection of acetaldehyde, acraldehyde, and acetone by the microbeaker methods of Griebel (B., 1924, 922; 1925, 470) and Griebel and Weiss (A., 1928, 82), using nitrophenylhydrazines, is described. The production of acetaldehyde from chloral hydrate by reduction with nascent hydrogen, of acetaldehyde from lactic and malic acids by oxidation with permanganate, of acraldehyde from glycerol by heating with potassium bisulphate, and of acetone from citric acid by oxidation with permanganate, is used for the detection of minute quantities of chloral hydrate,

lactic acid, malic acid, glycerol, and citric acid, respectively.

W. J. BOYD.

Chlorination of acetic acid. O. Y. MAGIDSON, I. G. ZILBERG, and N. A. PREOBRSCHENSKI (J. Chem. Ind., Moscow, 1928, 5, 528—529).—The acetic acid contained a little acetic anhydride; artificial illumination was not employed. Brückner's catalyst containing phosphorus, phosphorus pentachloride, and iodine was unsatisfactory; a 50—60% yield of chloroacetic acid required 20—23 hrs., and was contaminated with iodine which was difficult to remove. Activated carbon gave 30—50% yields of pure product in 23—37 hrs. When acetic acid vapour and chlorine were passed through activated carbon at 190—200° very little chloroacetic acid was formed. Powdered antimony has no catalytic action. Sulphur (1—2% of the acid), converted by chlorine into the monochloride, gave a yield amounting to 83% of the theoretical in 17 hrs. The product contained small quantities of sulphur compounds. By using two chlorination vessels in series the loss of acetic acid was diminished and the speed of chlorination doubled.

CHEMICAL ABSTRACTS.

Determination of glycerol by means of potassium permanganate. A. RAVENNA (Zymologica, 1928, 3, 174—176).—The crude glycerol or glycerol liquor is treated with silver carbonate and basic lead acetate and filtered, and the filtrate heated with excess of acid permanganate solution, the residual permanganate being afterwards determined by titration with oxalic acid solution.

T. H. POPE.

Analysis of glycerin according to the I.S.M. 1911. W. PRAGER (Chem.-Ztg., 1928, 52, 903—904).—The determination of glycerin by the International Standard Method, 1911, is considered unsatisfactory on account of the large blank due to the presence of carbonate in the sodium hydroxide employed. Objection is also raised to the use of a standard glycerin as recommended in Supplement No. 1 of the British Executive Committee, since it is very hygroscopic and the moisture content at time of use is difficult to determine quickly and accurately. The need of a really accurate method of international application is emphasised.

F. R. ENNOS.

See also A., Jan., 35, **Behaviour of catalysts in decomposition and synthesis of methyl alcohol (STORCH).** 42, **Determination of carbon and hydrogen in organic substances (BATESCU).** 54, **Chromium powder in organic synthesis (CHAKRABARTY and DUTT).** **Titanium tetrachloride in organic synthesis (STADNIKOV and KASCHTANOV).** 3:4-Dichloronitrobenzene (McMASTER and MAGILL). 56, **Sulphonation of 1-chloronaphthalene (FERRERO and BOLLINGER).** **Electrochemical oxidation of α -methyl-naphthalene (FICHTER and HERSZBEIN).** **Perylene and its derivatives (ZINKE and others).** 58, **Substitution products of azobenzene (BURNS, McCOMBIE, and SCARBOROUGH).** 59, **Alkaline fusion of anthracenemonosulphonic acids (FERRERO and CONZETTI).** 67, **"Oxidising" action of alkalis (LOCK).** 70, **Derivatives of hydroxyaminomethyl-anthraquinones and dihydroxydianthraquinonyl-ethylenes (DE DIESBACH and others).** **Munjistin (MITTER and SEN).** 74, **Acridone derivatives (WEISS).**

76, Synthesis of methyl 2:4-dihydroxybenz-1:10-naphthyridine-3-carboxylate (KELLER and STRANG).
85, Determination of nitrogen in nitro-compounds (WALLERIUS).

PATENTS.

Manufacture of aqueous solutions of carbon disulphide. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,561, 6.9.27).—Clear aqueous solutions of carbon disulphide of any desired dilution are made with the aid of an alcohol, such as butyl or amyl alcohol, and soap. In an example a 5% solution of carbon disulphide is stabilised by means of 4.2% of butyl alcohol and 2.3% of soap. E. HOLMES.

Manufacture of hydrocarbons poor in hydrogen. I. G. FARBENIND. A.-G. (B.P. 283,105, 12.12.27. Ger., 3.1.27).—Dehydrogenation of liquid hydrocarbons obtained by the polymerisation of lower ethylenic hydrocarbons, or by the destructive hydrogenation of tars and like products, to give aromatic or non-aromatic hydrocarbons poor in hydrogen (cf. B.P. 261,393 and 293,887; B., 1928, 440, 702), may be greatly facilitated by the use of chlorine, which immediately combines with the hydrogen liberated. The operation may be carried out by passing the hydrocarbon under treatment, either in the vapour or liquid phase, together with chlorine with or without a diluent gas, over various catalysts. The process may be expedited in some cases by the employment of reduced pressure or actinic rays. Temperatures of 300° are suitable, and cooling may be necessary to prevent undue carbonisation. E. HOLMES.

[Catalyst for] manufacture of unsaturated aliphatic hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,402, 27.6.27).—A catalyst for conversion of paraffins, especially C₂ to C₈, into the corresponding olefines, *e.g.*, pentane into amylene, is active charcoal such as that obtained by passing steam over brown-coal coke at 800–900°. C. HOLLINS.

Manufacture of halogenated hydrocarbon products and analogous substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,009, 18.3.27).—Halogenated hydrocarbons, halogenohydrins, nitriles, carbalamines, halogenated acid chlorides, *etc.* are obtained from the products of destructive hydrogenation of coal, mineral oils, *etc.*, if necessary after preliminary dehydrogenation, or splitting, or cracking, by addition of halogen, halogen hydride, hypochlorous acid, hydrogen cyanide, or carbonyl chloride. The addition may be effected in liquid or vapour phase, in the latter case preferably at high temperatures, *e.g.*, 400–500°, and in presence of catalysts. As example, chlorine is passed, while cooling, into the hydrocarbon mixture, b.p. 60–100°, obtained by hydrogenation of mineral oil, affording after distillation of unchanged hydrocarbons, chiefly dichloro-derivatives. R. BRIGHTMAN.

Manufacture of $\alpha\alpha\beta$ -trichloroethane. I. G. FARBENIND. A.-G. (B.P. 298,084, 1.10.28. Ger., 30.9.27).—A mixture of chlorine and vinyl chloride (10% excess) gives 93% yield of $\alpha\alpha\beta$ -trichloroethane when heated in the dark at 148–153°, or when warmed at 52–56° and at the same time exposed to actinic light. No solvent is used. C. HOLLINS.

[Catalysts for] hydrogenation or reduction of organic compounds. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 301,577, 19.9.27).—Catalysts for hydrogenation processes are prepared by precipitating finely-divided nickel, copper, *etc.* from their salts (*e.g.*, acetates) by means of "oxydisilin" or other silicon-oxygen compound of a lower stage of oxidation than silica, a carrier being present if desired. C. HOLLINS.

Manufacture of alcohols. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,000, 18.6.27).—Products obtained by the destructive hydrogenation of mineral oils, coal, *etc.*, if necessary after preliminary dehydrogenation, or splitting, or cracking, are treated, *e.g.*, with moderately dilute sulphuric acid and the resulting sulphuric esters are decomposed with excess of water. As example, the gas mixture obtained in the destructive distillation of coal, after releasing the pressure, cooling, and compressing to 40 atm., gives a liquid which when released at 5 atm. yields a gas from which isopropyl alcohol, butyl alcohol, and small amounts of higher alcohols may be obtained by way of their sulphuric esters. R. BRIGHTMAN.

Manufacture of acetic anhydride from acetic acid. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,562, 6.9.27).—Acetic anhydride resulting from the thermal decomposition of acetic acid may be freed from the greater part of the water simultaneously produced, and concentrated to about 80%, by taking advantage of the different rates of diffusion of these vapours through graphite. E. HOLMES.

Manufacture of carboxylic acid anhydrides. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 288,549, 5.4.28. Ger., 11.4.27).—Vinyl esters react with carboxylic acids to give the anhydrides of the latter and acetaldehyde. A catalyst which converts paraldehyde into acetaldehyde (*e.g.*, sulphuric, phosphoric, or benzene-sulphonic acid) is preferably present. Vinyl acetate, boiled under fractionating reflux with acetic acid and a little sulphuric acid, gives acetaldehyde and acetic anhydride in about 70% yield. A continuous process is described. Mixed anhydrides may be prepared. C. HOLLINS.

Production of esters. IMPERIAL CHEM. INDUSTRIES, LTD., and G. F. HORSLEY (B.P. 301,523, 3.8.27).—A continuous process is described for the production of esters which form with the alcohol and water azeotropic ternary mixtures containing more water than corresponds with the ester entrained; water (or aqueous condensate) is continuously added to the reaction mixture in sufficient amount to carry over all the ester in the form of ternary mixture. The latter is condensed and the layer rich in ester is fractionated, the aqueous layer being re-used as above. A method for continuous fractionation is described. The example given is isobutyl acetate. C. HOLLINS.

Manufacture of water-soluble esters of dicarboxylic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,041, 28.11.27 and 6.7.28).—Dibasic organic acids are treated with 2 or more mols. of an ethylene oxide with or without a solvent and/or sodium chloride or the sodium salt of the organic acid. Products

from ethylene oxide and phthalic and succinic acids are described; they are solvents and plasticisers.

C. HOLLINS.

Manufacture of *o*- and *p*-xylenes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,940, 20.8.27).—Toluene is treated with formaldehyde in presence of hydrochloric acid or hydrogen chloride and a condensing agent, e.g., zinc chloride, and the resulting mixture of about equal proportions of *o*- and *p*-methoxybenzyl chlorides is reduced catalytically, electrolytically, or with zinc dust and sodium hydroxide at 100° to the corresponding *o*- and *p*-xylenes. The latter are separated by fractional distillation or by sulphonation and hydrolysis.

R. BRIGHTMAN.

Manufacture of sulphonic acids of 6-chloro-2-amino-1-methylbenzene [6-chloro-*o*-toluidine].

I. G. FARBENIND. A.-G. (B.P. 290,229, 10.5.28. Ger., 10.5.27).—Sulphonation of 6-chloro-*o*-toluidine gives by the "bake" method 6-chloro-*o*-toluidine-5-sulphonic acid, or by means of weak oleum at 70–90° the 3-sulphonic acid. Disulphonation with 12–13% oleum at 100–140° yields the 3:5-disulphonic acid. The acids are characterised by conversion into 2-chlorotoluene-3-sulphonic acid (chloride, m.p. 33°; amide, m.p. 183°; anilide, m.p. 180°), 2-chlorotoluene-5-sulphonic acid (chloride, m.p. 63°; amide, m.p. 130°; anilide, m.p. 87°), and 2-chlorotoluene-3:5-disulphonic acid (chloride, m.p. 79°; amide, m.p. 237°; anilide, m.p. 188°), respectively; or into 2:6-dichlorotoluene-3-sulphonic acid (chloride, m.p. 57°; amide, m.p. 210°; anilide, m.p. 163°) and 2:6-dichlorotoluene-3:5-disulphonic acid (chloride, m.p. 165°; amide, m.p. 290°; anilide, m.p. 265°), respectively.

C. HOLLINS.

Production of dibenzanthrone. I. B. ANDERSON, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 301,403, 22.7.27).—In the conversion of 3:3'-dibenzanthronyl into dibenzanthrone by means of alcoholic potassium hydroxide, quantitative yields are obtained by using an inert solvent (kerosene, xylene, benzene, chlorobenzene) preferably at 120°; conversion also occurs in 12 hrs. at 80° or 48 hrs. at 15–20°.

C. HOLLINS.

Manufacture of monobenzoyldiaminoanthraquinones. I. G. FARBENIND. A.-G. (B.P. 276,692, 26.8.27. Ger., 30.8.26).—Diaminoanthraquinones are treated with benzoyl chloride in an organic solvent in presence of an acid-binding agent, fractional separation of mixtures of benzoyl derivatives being effected by the addition of an aliphatic alcohol when required. 1-Amino-4-benzamidoanthraquinone is obtained in nitrobenzene and pyridine at 80°, and the 1:5-isomeride in nitrobenzene in presence of sodium carbonate and alcohol.

R. BRIGHTMAN.

Manufacture of alkylpyrazolanthrone. I. G. FARBENIND. A.-G. (B.P. 282,375, 3.12.27. Ger., 14.12.26. Addn. to B.P. 264,503; B., 1928, 441).—Alkylation of pyrazolanthrone is effected in absence of acid condensing agents, but preferably in presence of acid-binding agents. Mixtures of alkylation products may thus be obtained. Thus sodium pyrazolanthrone and ethyl bromide at 150° in presence of copper acetate

give ethylpyrazolanthrone, m.p. 183–186°. Methyl sulphate in presence of sodium hydroxide at 40° gives methylpyrazolanthrone, m.p. 161°, and ethyl sulphate similarly yields a mixture of two ethylpyrazolanthrone, separable with benzene.

R. BRIGHTMAN.

Manufacture of valuable products from sorbitol.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,655, 19.12.27).—Sorbitol, when heated alone or with water at 300° or with dehydrating agents (sulphuric acid, phosphoric acid, etc.) or catalysts (alumina, active silica, or carbon), loses 1 or 2 mols. of water, yielding products, b.p. 220°/0.1 mm. and 157°/3 mm., respectively, which are valuable plasticisers.

C. HOLLINS.

Catalyst for synthetic production of methyl alcohol. J. C. WOODRUFF, G. BLOOMFIELD, and W. J. BANNISTER, ASSTS. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,695,447, 18.12.28. Appl., 25.10.26).—See B.P. 279,377; B., 1928, 844.

Manufacture of acetaldehyde. C. N. HAND, ASST. to RUBBER SERVICE LABS. CO. (U.S.P. 1,696,479, 25.12.28. Appl., 12.4.26).—See B.P. 269,556; B., 1928, 82.

Purification of acetic anhydride. W. P. SKERTCHLY, ASST. to CELANESE CORP. OF AMERICA (U.S.P. 1,696,363, 25.12.28. Appl., 25.3.26. U.K., 13.2.26).—See B.P. 270,809; B., 1927, 541.

Concentrating lower aliphatic acid solutions. H. DREYFUS and C. I. HANEY, ASSTS. to CELANESE CORP. OF AMERICA (U.S.P. 1,696,432, 25.12.28. Appl., 22.1.27. U.K., 30.11.26).—See B.P. 283,702; B., 1928, 223.

Separation of mono- and di-alkyl derivatives of aromatic amines. W. FLEMMING and H. KLEIN, ASSTS. to SILESIA VER. CHEM. FABR. IDA- U. MARIENHUETTE (U.S.P. 1,695,372, 18.12.28. Appl., 3.11.27. Ger., 19.11.26).—See B.P. 280,877; B., 1928, 361.

Diazo compound (B.P. 295,050).—See IV.

IV.—DYESTUFFS.

Condensation of badan extract with *p*-nitrosodimethylaniline. S. N. GONDEV (J. Chem. Ind. Moscow, 1928, 5, 78–79).—Condensation of badan extract with *p*-nitrosodimethylaniline in presence of methyl or ethyl alcohol produces a resinous substance and affords a water-soluble dye in 8% yield. The dye is formed by interaction of tannins with the *p*-nitrosodimethylaniline. Wool and cotton are directly dyed black, brown, and dark grey; acid dyeing of wool yields dark brown colours, whilst alkaline dyeing of cotton gives greyish-blue colours.

CHEMICAL ABSTRACTS.

See also A., Jan., 26, [Absorption of dyes by] alumina gels (BONNELL), 49. Plant colouring matters (KARRER, HELFENSTEIN, and WIDMER). 62. Compounds of type of malachite-green (CHAMBERLAIN and DULL). 66. Effect of sulphur on colour (DEY and DUTT). 71. Orcein-like dyes (HENRICH and HEROLD). 75. Dyes derived from quinoline-2-aldehyde (DEY and DUTT). 76. Red quinoline dye (WIELAND, HETTCHER, and HOSHINO). 86. Determination of colouring matter by separation of fluid layers (HOFMAN).

PATENTS.

Manufacture of dye preparations. BRIT. DYE-STUFFS CORP., LTD., H. ADAMS, and A. SHEPHERDSON (B.P. 301,549, 3.8.27).—A dispersed dye paste is mixed with the product obtained by the action of hot alkali on ligninsulphonic acid. Such a preparation retains its power of dispersing readily in water. C. HOLLINS.

Dye preparations. IMPERIAL CHEM. INDUSTRIES, LTD., W. GIBSON, A. J. HAILWOOD, J. B. PAYMAN, and A. SHEPHERDSON (B.P. 300,800, 29.8.27).—Polychloronaphthalenes, containing 3–4.5 atoms of chlorine, are sulphonated, e.g., with 22% oleum at 150–180°, and the sulphonic acids or salts are mixed with substantially insoluble dyes, giving aqueous pastes or suspensions which have valuable wetting-out properties and are not liable to deterioration by fungus growths.

R. BRIGHTMAN.

Manufacture of dyes. I. G. FARBENIND. A.-G. (B.P. 288,214, 4.4.28. Ger., 4.4.27).—Basic dyes for silk and tannin-mordanted cotton are produced by coupling β -diketones, β -ketonic acid esters or arylamides, or malonic esters with diazotised safranines. Tolusafraanine gives claret-red, diethylsafaanine various shades of black.

C. HOLLINS.

Production of [acid] dyes. [Anthraquinone-acridinesulphonic acids.] L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 301,769, 1.6.27).— α -Arylaminoanthraquinones, except α -carboxy- or α -amino-arylamino-compounds, are acridinated and sulphonated by treatment with concentrated sulphuric acid or oleum above 130°. 1:4-Dianilino-2-hydroxyanthraquinone or its sulphonation product gives a bluish-red acid dye.

C. HOLLINS.

Manufacture of dyes of the pyrone series. K. and K. S. CARPMAEL, and I. G. FARBENIND. A.-G. (B.P. 300,594, 16.6.27. Addn. to B.P. 247,003; B., 1926, 234).—The pyrone dye acids of the prior patent are converted into suitable salts, and the difficultly soluble salts of the pyrone dyes are separated from the more soluble salts of the secondary products.

R. BRIGHTMAN.

Manufacture of vat [benzanthrone] dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,197, 21.11.27. Addn. to B.P. 289,890; B., 1928, 517).—The process of the prior patent is extended to 8-, 9-, 10-, and 11-aminobenzanthrones. 8-Aminobenzanthrone (obtained from 8-chlorobenzanthrone, m.p. 183°) gives a blue vat dye; 9-aminobenzanthrone at 170° a grey-violet dye, probably identical with that described in B.P. 254,742 (B., 1927, 550), and at 230° a greenish-grey dye; 10-aminobenzanthrone a violet dye; and 11-aminobenzanthrone a greyish-blue vat dye. 8-Chlorobenzanthrone is obtained from 6-chlorobenzanthrone, m.p. 152°, and 11-chlorobenzanthrone, m.p. 174°, from 1-chloroanthraquinone.

R. BRIGHTMAN.

Manufacture of green vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,683, 17.8.27).—Aroylamino-1:2:5:6-diphthaloylacridones (B.P. 295,645; B., 1928, 781) when treated with hydrazine give intense green vat

dyes. The dyes from benzamido-, *o*-chlorobenzamido-, and *m*-methoxybenzamido-1:2:5:6-diphthaloylacridone are described. Alternatively, amino-1:2:5:6-diphthaloylacridone may be condensed with hydrazine hydrate and the product treated with benzoyl chloride in nitrobenzene.

R. BRIGHTMAN.

Manufacture of preparations of leuco-compounds of vat dyes. J. R. GEIGY A.-G. (B.P. 276,023, 15.8.27. Ger., 16.8.26).—Dry, stable, and readily soluble preparations of leuco-compounds of vat dyes are obtained by intimately mixing the vat dye with a dihydric alcohol, e.g., ethylene glycol, propylene glycol, ethylene thiodiglycol, or with an aldehyde other than a carbohydrate such as dextrose, reducing the dye by means of hyposulphite in presence of an alkali, and drying the product.

R. BRIGHTMAN.

Manufacture of a new diazo compound and of new [wool] dyes therefrom. COMP. NAT. MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES ÉTABL. KUHLMANN (B.P. 295,050, 3.8.28. Fr., 5.8.27).—4-Chloro-6-sulphobenzene-1:2-diazo-oxide, made by adding sodium acetate to diazotised 4-chloro-2-nitroaniline-6-sulphonic acid, is coupled especially with unsulphonated phenols or naphthols having a free *ortho*-position, e.g., resorcinol (yellow; violet-brown when chromed, reddish-brown when coppered), 1-*p*-sulphophenyl-5-pyrazolone-3-carboxylic acid (yellow; bluish-red, chromed), α - or β -naphthol (brown; dull violet, chromed). The dyes may be pre-chromed.

C. HOLLINS.

Manufacture of azo dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 301,410, 29.8.27).—Dyes fast to kier-boiling and to light are obtained by coupling, in substance or on the fibre, a diazotised cyanoarylamine with a suitable end-component. Examples are: 4-cyano-*o*-toluidine \rightarrow 2:3-hydroxynaphthoic 4-chloro-*o*-anisidide (red), or α -naphthylamide (bluish-red), or bis-acetoacetylolidine (reddish-yellow); 6-cyano-*o*-toluidine \rightarrow 2:3-hydroxynaphthoic 5-chloro-*o*-toluidide (scarlet); 4-chloro-2-cyanoaniline (from 3-chloro-6-nitroaniline) \rightarrow 2:3-hydroxynaphthoic α -naphthylamide (claret), anilide (reddish-orange), *o*-toluidide (yellowish-scarlet), or 5-chloro-*o*-toluidide (yellowish-scarlet); 6-cyano-*m*-4-xylylidine (from 6-nitro-*m*-4-xylylidine) \rightarrow Naphthol AS (scarlet); 6-chloro-*m*-cyanoaniline (from 4-chloro-*m*-nitroaniline) \rightarrow 2:3-hydroxynaphthoic 5-chloro-*o*-toluidide, *p*-anisidide, *o*-toluidide, or anilide (all yellowish-scarlet); 4-cyano-*m*-toluidine \rightarrow 2:3-hydroxynaphthoic 5-chloro-*o*-toluidide (reddish-orange), anilide (scarlet), or 4-chloro-*o*-anisidide (orange); 5-chloro-*o*-cyanoaniline (from 4-chloro-*o*-nitroaniline) \rightarrow 2:3-hydroxynaphthoic 5-chloro-*o*-toluidide (reddish-orange).

C. HOLLINS.

Manufacture of bluish sulphurised indophenol dyes. H. BOTS and A. CATINEAU, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,695,756, 18.12.28. Appl., 9.11.25. Switz., 28.11.24).—See B.P. 243,739; B., 1926, 866.

Preparation of monoaminodiphthaloylacridones. W. MIEG and A. JOB, ASSRS. to GRASELLI DYE STUFF CORP. (U.S.P. 1,696,499, 25.12.28. Appl., 23.5.27. Ger., 27.5.26).—See B.P. 295,645; B., 1928, 781.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Effluent water of cellulose manufacture. A. HEIDUSCHKA and E. MUNDS (Z. angew. Chem., 1929, 42, 11—15).—A study of the growth of *Leptomitius lacteus* and *Sphaerotilus natans* in the waste sulphite liquor from cellulose manufacture. Typical analyses of the concentrated liquor show that at most only 1% of the cellulose is converted into sugars during the heating. The waste liquor contains 0.1—0.8% of sulphur dioxide (depending on the duration of heating and the raw material used), 0.16—0.81% of acetic acid, 0.009—0.018% of formic acid, and only a trace of sulphur trioxide. The effect of various concentrations of the individual constituents and the whole liquor on the growth of a pure culture of the fungi has been studied *in vitro*. Sulphur dioxide in concentrations up to 0.8% has no effect, but at higher concentrations growth is retarded and ultimately the organism is destroyed. With acetic acid, formic acid, sodium hydroxide, potassium hydroxide, and ammonia growth occurs at a concentration of 0.3, 0.1, 0.05, 0.07, and 0.06%, respectively, whilst the organism can exist up to concentrations of 1.4, 0.9, 0.20, 0.30, and 0.20—0.25%, respectively. Aeration of the water is necessary for growth, and the organism is destroyed at about 31°. With the sulphite waste liquor growth just occurs at a concentration of 0.0001%, the optimum concentration being about 0.1%. With the alcoholic sulphite waste liquor no growth occurs below a concentration of 0.01%, the optimum concentration being about 1.0%. Thus, since growth of the organism is not possible in the higher concentrations of waste liquor, the nuisance may be partly remedied by walling up the outlet to concentrate the effluent. J. W. BAKER.

Regeneration and use of cellulose liquors. U. POMILIO (Atti II Cong. Naz. Chim. pura appl., 1926, 748—756; Chem. Zentr., 1928, ii, 406).—Liquors from the chlorine process, concentrated by repeated use, are less complex in composition than those obtained from the sulphite, sulphate, and soda processes. Their catalytic pyrogenetic treatment and electrolytic regeneration are being investigated. A. A. ELDRIDGE.

Viscose. XIX. Pretreatment of alkali-cellulose and the spinning properties, viscosity, and surface tension of viscose prepared therefrom. XX. Comparison of the properties of steeped and pressed alkali-cellulose. G. KITA, J. ONOHARA, and K. SAKURADA (J. Cellulose Inst., Tokyo, 1928, 4, 293—297, 298—300).—XIX. The effect of varying the periods of steeping and maturation of the alkali-cellulose on the physical properties of the resulting viscose are examined. Spinning properties and viscosity were closely related. Viscose filaments from alkali-cellulose matured for short periods in closed vessels appear as satisfactory as those from alkali-cellulose steeped and matured for longer periods in air or in an atmosphere of hydrogen.

XX. Maturing alkali-cellulose in the pressed-out condition resulted in slightly higher viscosity of the viscose prepared from it than when the cellulose was steeped for a long period, pressed out, and used immediately. Definite differences in spinning properties and in the physical properties of the spun filaments were also observed. A. G. POLLARD.

Acetylation of cellulose. I. SAKURADA (J. Cellulose Inst., Tokyo, 1928, 4, 304—305).—Cellulose is acetylated by treating alkali-cellulose with acetic anhydride in benzene solution at room temperature. The product approximates to a combination of 1 mol. of acetic acid + 2 mols. of cellulose. A. G. POLLARD.

Plastic masses. F. SCHMIDT (Kolloid-Z., 1928, 46, 324—329).—A review of the preparation and properties of commercially important plastic materials, including those derived from nitrocellulose, acetylcellulose, cellulose ethers, condensation products of phenol and urea with formaldehyde, and casein products. E. S. HEDGES.

Alteration in colloid structure of native cellulose by dissolution and spinning. O. FAUST (Kolloid-Z., 1928, 46, 329—336).—A lecture on the structure of cellulose under various conditions. E. S. HEDGES.

Influence of temperature on sulphite[-cellulose] cooking. E. HÄGGLUND (Pulp & Paper Mag., 1928, 26, 1775—1778).—See B., 1928, 924.

Microscopical investigation of artificial silk fibres. L. G. LAWRIE (Rev. gén. Colloid., 1928, 6, 252—257).—See B., 1928, 294.

See also A., Jan., 101, **Decomposition of cellulose by aërobic bacteria** (DUBOS).

Washing agents. WALTHER.—See XII: **Nature of nitrocellulose.** BRUNSWIG.—See XXII.

PATENTS.

Manufacture of cellulose. VICKERS, LTD., and O. D. LUCAS (B.P. 298,800, 29.11.27).—After digesting vegetable material with water at 180° under pressure for 1 hr., the furfuraldehyde is distilled off, and the residue is treated with caustic soda solution in the usual manner. F. R. ENNOS.

Manufacture of artificial silk. K. and K. S. CARPMAEL. FROM I. G. FARBERIND. A.-G. (B.P. 301,563, 6.9.27. Addn. to B.P. 280,628; B., 1928, 154).—A modification of the vertical-path stretch-spinning process in which cuprammonium solutions of cellulose prepared from wood pulp, straw, grasses, or cotton linters are precipitated in water at below 30°, the vertical length of immersion being 50—80 cm. F. R. ENNOS.

Manufacture of artificial silk. A. E. GULL (B.P. 301,141, 14.9.27).—In the manufacture of artificial silk by the dry-spinning process the extruded filaments pass downwards through a drying chamber consisting of a tube of gradually decreasing cross-section, while a current of heated air or gas passes through the tube in the same direction, its velocity being consequently greater at the exit than at the entrance of the drying chamber. F. R. ENNOS.

Manufacture of artificial silk and the like from viscose. K. LEUCHS (B.P. 298,688, 18.6.27).—The artificial silk is spun on to a foraminated spool made of aluminium, on which, after being washed free from acid, it is desulphurised by hot solutions of salts of weak acids, e.g., alkali or ammonium salts of acetic, carbonic, boric, silicic, or phosphoric acids, or sodium sulphite, which are made to flow through the material by differential pressure; finally it is subjected to the necessary

finishing processes, the formation of the thread into a skein being dispensed with. F. R. ENNOS.

Manufacture of viscose silk. I. G. FARBENIND. A.-G. (B.P. 276,679, 25.8.27. Ger., 25.8.26).—In order to increase the wetting capacity of viscose silk, addition is made to the material, before or after spinning or to the spinning bath, of the following: an alkylated or aralkylated naphthalenesulphonic acid, an amino-benzene- or aminonaphthalene-sulphonic acid alkylated or aralkylated in the amino-group, or a product obtainable by sulphonation of brown-coal tar oils by means of chlorosulphonic acid or fuming sulphuric acid (cf. B.P. 271,474; B., 1928, 326). F. R. ENNOS.

Spinning of viscose silk. H. KINDERMANN (B.P. 301,811, 6.8.27).—Soft, lustrous fibres are made from unripe viscose by coagulation first in an acid bath and finally in an aqueous solution of chloride, nitrate, or sulphate of copper, cadmium, lead, tin, mercury, bismuth, antimony, selenium, or tellurium. F. R. ENNOS.

Treatment of organic derivatives of cellulose. BRIT. CELANESE, LTD. (B.P. 279,502, 22.10.27. U.S., 23.10.26).—Materials made of organic derivatives of cellulose are weighted by treatment with a solution of a metallic salt, e.g., stannic chloride, of such concentration that it acts also as a gelling agent; alternatively, a more dilute solution of the salt may be used if the material is first subjected to a gelling agent such as formic acid, phenol, thiocyanates, etc. The weighting material is fixed by washing with water or a dilute solution of alkali or soap. F. R. ENNOS.

Treatment of threads or fabrics containing cellulose acetate silk. G. B. ELLIS. From SOC. FABR. SOIE "RHODIASETA" (B.P. 301,335, 22.6.27).—Cellulose acetate silk is delustred with perfect regularity without deleteriously affecting the strength, elasticity, and other qualities of the fibre by soaking the yarn or fabric for 2–24 hrs. at the ordinary temperature in a 10–30% aqueous solution of calcium, lithium, or magnesium chloride and treating the impregnated fabric, after squeezing, for 1–20 min. in boiling, dilute (0.1–0.01N) acid. Suitable acids include 0.1N-hydrochloric or -boric acid, 0.02N-benzoic acid, and 0.01N-salicylic or -β-naphthalenesulphonic acid. D. J. NORMAN.

Preparation of artificial textile products with reduced lustre from acetylcellulose. N. V. NEDERL. KUNSTZIJDEFABR. (B.P. 291,067, 6.9.27. Holl., 28.5.27).—The spinning process is carried out in the presence of water vapour which is introduced into the spinning cell; or after spinning in the usual way the material, while still retaining some of the solvent, is passed to a second chamber into which water vapour is introduced.

F. R. ENNOS.

Manufacture of cellulose acetate. J. W. BULMER (B.P. 298,819, 2.1.28).—The liquid product obtained by the acetylation of cellulose is treated with sufficient water to produce mild hydrolysis. After being kept for 24–30 hrs. at room temperature an excess of water is added to precipitate the acetate, which is washed and immersed in dilute acid until sufficient hydrolysis has occurred to render it insoluble in chloroform while retaining its solubility in acetone. F. R. ENNOS.

Manufacture of cellulose esters. FABR. VAN CHEM. PROD. (B.P. 287,540, 23.8.27. Fr., 24.3.27).—Cellulose is partially esterified by highly concentrated formic acid, using zinc chloride or a similar salt as catalyst, and the product, after removal of the excess of formic acid centrifugally, is then, without washing or drying, completely esterified at a low temperature, preferably below 0°, by formic acid in the presence of an active catalyst, e.g., gaseous hydrogen chloride alone or in admixture with salts such as zinc chloride. The partly formylated product may also be used as the parent material for the preparation of other esters. By adopting a two-stage process it is possible to eliminate about half the water formed during esterification before the addition of the active catalyst necessary to complete the reaction, and by operating at a low temperature the decomposing action of water formed during this final stage is minimised. Completely esterified, stable products are obtained.

D. J. NORMAN.

Manufacture of mixed acid esters of cellulose or esters of cellulose ethers. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 300,942, 12.5.27).—Cellulose derivatives, other than a nitrocellulose or an ether soluble in alkali, which still contain one or more free reactive hydroxyl groups, are swollen by or dissolved in an organic solvent and treated with an inorganic acid chloride in presence of an acid-binding agent, or with an organic acid chloride in presence of an acid-binding agent other than pyridine, or with a simple or mixed acid anhydride (except acetic anhydride) in the absence of a reaction promoter, or with an acid amide or an acid ester. Thus cellulose acetate, containing 54.5% of combined acetic acid, with phosphorus trichloride in pyridine and acetone gives a product containing 0.5% P; with monophenyl phosphate in pyridine a cellulose acetate phosphate containing 10.8–11% PO₄; and with molten stearic anhydride a cellulose acetate stearate containing 28–30% of stearic acid. Ethylcellulose in xylene solution with silicon tetrachloride in chloroform and pyridine affords a product containing 3.8% of combined silica; with myristic anhydride at 140° an ethylcellulose myristate; and with molten benzamide at 130° ethylcellulose benzoate. A cellulose laurate phosphate containing 65.5% of lauric acid and 4.2% of phosphoric acid is obtained by heating cotton at 110–145° in a mixture of xylene, pyridine, and lauryl chloride, and subsequently adding phosphorus oxychloride at 110–120°. Ethylcellulose obtained, e.g., by heating alkali cellulose at 120° with ethyl chloride, benzene, and sodium hydroxide, when treated without isolation with phosphorus oxychloride and further benzene, yields an ethylcellulose phosphate containing 5.5% PO₄. R. BRIGHTMAN.

Treatment of agave waste (B.P. 301,284).—See XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

See A., Jan., 41, Quantitative spectral analysis (KNOP).

Washing agents. WALTHER.—See XII.

PATENTS.

Dyeing with vat dyes sensitive to calcium compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,166, 15.10.27).—Addition of phosphoric acid or a soluble phosphate to the dye-bath or the dye enables ordinary hard water to be used in the dye-bath.

R. BRIGHTMAN.

Dyeing or otherwise colouring of materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 300,929, 20.7.27).—Acetate silk is dyed by coupling an acetoacetic arylamide with a diazo compound on the fibre, either component being applied first; or by developing with coupling components a diazotised diamine or diaminoazo compound in which one amino-group carries an acetoacetyl group. Examples are: bisacetoacetyltolidine padded on acetate silk and coupled with diazotised *p*-nitroaniline (yellow); *p*-nitroaniline \rightarrow *p*-xylylidine diazotised on the fibre and coupled with bisacetoacetyltolidine (golden-brown); 4-amino-4'-acetoacetamidoazobenzene diazotised on the fibre and coupled with dimethylaniline (orange).

C. HOLLINS.

Dyeing of artificial silk comprising acyl derivatives or ethers of cellulose or of its conversion products. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,564, 6.9.27).—Acetate silk etc. is dyed with azo dyes obtained by coupling 1:3-diketohydrindene with diazotised arylamine-monosulphonic or mono-carboxylic acids, e.g., sulphanic acid (yellow), anthranilic acid (yellow), or 2-amino-6-naphthoic acid.

C. HOLLINS.

Dyeing and printing. POLLOPAS, LTD., and G. SPENCER (B.P. 301,365, 24.6.27 and 24.4.28).—The affinity of textiles for dyes is varied by a previous impregnation or printing with an aqueous solution of a water-soluble formaldehyde-urea condensation product.

C. HOLLINS.

Mercerisation of loose cotton. C. AHNERT (B.P. 301,591, 3.10.27).—The fibres during mercerisation and subsequent washing are subjected to tension sufficient to give them a high lustre. A centrifugal hydro-extractor suitable for the purpose is described.

A. J. HALL.

Production of pattern effects on textile materials composed of or containing cellulose esters. BLEACHERS' ASSOC., LTD., W. KERSHAW, F. L. BARRETT, and R. GAUNT (B.P. 301,567—8, 7.9.27).—The lustre of delustred cellulose acetate silk yarns and fabrics is restored by application of (A) ethyl lactate or (B) ethylene glycol monoethyl ether and drying. For the production of pattern effects, cellulose acetate silk is completely delustred by padding with a solution of a soap and steaming, then printing with a thickened paste containing ethyl lactate or ethylene glycol monoethyl ether, drying, and afterwards washing; the printed parts have a higher lustre than the non-printed parts. Suitable dyes may be added to the printing pastes so that the silk is dyed simultaneously.

A. J. HALL.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Treatment of finely-divided caliche in factories "T," "U," and "V." C. M. AUTY, E. B. DONALD, W. H. CLAYTON, and L. L. MALM (Caliche, 1928, 10, 343—360).—A critical report on the methods adopted in three works for treatment of finely-ground caliche siftings ("finos") separately from the main plant. Analyses are given of the raw material and of the various liquors, and the factors influencing efficiency are discussed. In factory "T" the Trent system (cf. Caliche, 1924, 5, 433) is used, in which the finely-divided caliche is stirred and heated with a series of liquors of diminishing concentration, the first extract going to the main plant and subsequent extracts to other tanks in the subsidiary series. In the systems used in "U" and "V" one lixiviation only is carried out in the tank, and the mixture is then filtered. In "U" filtration takes place under pressure through Burt gyratory filters. The extraction is completed by washing in the filter. In "V" Butters box filters are used, the liquor being sucked through the filter by a vacuum pump. The efficiency of lixiviation is somewhat higher in "U" than in "T." It is low in "V." Filtration under pressure has several advantages over filtration by suction. Details are given of the determination of coarse and fine sand and sludge in caliche, from the results of which may be derived an "index of ease of treatment" = (% total sand/% sludge) \times (% coarse sand/% fine sand).

R. K. CALLOW.

Manufacture of potassium chloride from Solikamsk sylvite. F. F. VOLF and V. S. YATLOV (J. Chem. Ind. Moscow, 1928, 5, 274—279).—Fairly pure potassium chloride is obtained from the sylvite, of low magnesium content, by crystallisation from water. Increase in the magnesium chloride content of the liquors up to 120 g./kg. of water does not influence the speed of dissolution of potassium and sodium chlorides. The solubility of sodium chloride in saturated solutions of potassium chloride containing a little magnesium chloride decreases with time, but increases with rise of temperature when the magnesium chloride content is >100 g./kg. of water. With hot solutions containing more magnesium chloride, even if saturated with potassium chloride, sodium chloride is precipitated on cooling. At this magnesium chloride content, pure potassium chloride cannot be obtained.

CHEMICAL ABSTRACTS.

Determination of water in calcium nitrate. R. LUCAS (Z. angew. Chem., 1928, 41, 1367).—For the determination of combined water in technical calcium nitrate 20 g. of the salt are quickly weighed into a short-necked flask (250 c.c.) and covered with 200 c.c. of freshly-distilled xylene. The mixture is distilled gently for 6 hrs., the distillate being caught in a graduated cylinder, where the water and xylene separate into two conjugate layers. The volume of water is measured directly and calculated to percentage. A. R. POWELL.

Manufacture of copper sulphate from brass turnings. I. B. EVTUSHENKO (J. Chem. Ind. Moscow, 1928, 5, 522—523).—In the method whereby the zinc

content of the brass is reduced to 0.25–0.5% by the prolonged action of hot sulphuric acid without access of air, the treatment with acid being repeated with aeration to dissolve the copper, the latter reaction is accelerated to 4–5 times its former speed if the acid contains 0.4% of chromium. The first reaction is greatly accelerated by using hydrochloric (0.15 g. per c.c.) instead of sulphuric acid. A suitable apparatus has been designed.

CHEMICAL ABSTRACTS.

Determination of copper in commercial copper sulphate. V. MORANI (Atti II Cong. Naz. Chim. pura appl., 1926, 1376–1380; Chem. Zentr., 1928, ii, 590).—Biazzo's method (B., 1925, 314) is untrustworthy, since the reaction $\text{HSCN} + 3\text{O} + \text{H}_2\text{O} = \text{HCN} + \text{H}_2\text{SO}_4$ does not quantitatively express the change.

A. A. ELDRIDGE.

Sulphuryl chloride and its preparation. E. TERLINCK (Chem.-Ztg., 1928, 52, 901–903, 944–946).—The reaction $\text{SO}_2 + \text{Cl}_2 = \text{SO}_2\text{Cl}_2$ is reversible, the formation of sulphuryl chloride being favoured by low temperatures and its decomposition by higher temperatures; catalysts such as camphor, acetic anhydride, and absorbent carbons, which are commonly used in the preparation, accelerate both reactions. For the efficient preparation of the substance the dry gases are combined in the presence of a catalyst, e.g., norit, at normal temperatures. The whole is then cooled to -10° to effect complete combination of the constituent gases, filtered from the catalyst, and then carefully distilled, the liquid being in contact with the source of heat for as short a time as possible. F. R. ENNOS.

See also A., Jan., 25, **Behaviour of helium towards glass and palladium** (PANETH and PETERS). 35, **Catalytic preparation of copper sulphate** (ABEL and REDLICH). **Nitrite-nitrate formation from ammonia and oxygen at alkaline surfaces** (HOFFMANN). 36, **Catalytic properties of arsenic compounds** (MOUREU, DUFRAISSE, and BADOCHÉ). **Electrolysis of copper sulphate solutions** (CHANOZ). **Preparation of persulphates** (ESSIN). **Electrolytic preparation of potassium permanganate** (RAPIN). 38, **Hydrates of beryllium halides** (ČUPR and SALANSKÝ). **New fluoboric acid and its salts** (TRAVERS and MALAPRADE). 40, **Reaction between nitrogen peroxide and sulphur dioxide** (BRINER, LUNGE, and VAN DER WIJK). 42, **Rapid determination of selenium** (BENESCH). **Determination of nitrate nitrogen** (ŠIMEK). **Volumetric determination of phosphoric acid in phosphates** (DRACHOUSSE and DOUCHY). 43, **Determination of chromium** (RIPAN). 54, **Titanium tetrachloride in organic synthesis** (STADNIKOV and KASCHTANOV). 90, **Determination of silica in tissues** (KING). 98, **Toxicity of thallium sulphate** (MUNCH). 110, **Micro-determination of iodine in organic products** (PFEIFFER). **Micro-determination of iodide in presence of chloride** (KIEFERLE and ERBACHER).

Stainless iron in nitric acid industry. MITCHELL. —See X. **Chrome tanning compounds.** STIASNY. —See XV. **Retention of phosphate by hydrated alumina.** MILLER. **Precipitation of calcium oxalate.** CHAPMAN. —See XVI. **Assay of mercury oxycyanide**

and mercury cyanide pastilles. SCHULEK and STASIAK. —See XX.

PATENTS.

Recovery of sodium salts [carbonates] from brines [containing them]. W. A. KUHNERT (U.S.P. 1,693,237, 27.11.28. Appl., 29.3.27).—Sodium carbonate brines are treated with gases containing carbon dioxide at such a rate that the temperature does not exceed 45° whereby coarsely crystalline sodium sesquicarbonate is precipitated; after this has been removed, carbonation is continued to precipitate sodium bicarbonate.

L. A. COLES.

Apparatus for the preparation of cooling brines. O. DAHL and E. A. H. KJÖRSTAD (B.P. 294,595, 28.12.27. Fr., 27.7.27).—Sea water etc. is pumped through a vessel containing a mixture of ice and salt, or successively through vessels containing ice and salt, the vessels being provided with upright pipes at the top in which the brine rises to provide a suitable head and which also serve for the escape of air bubbles from the vessel. When the ice has melted, the brine is passed into a receptacle below, whence it may be drawn off for use or returned to the vessels after they have been recharged.

L. A. COLES.

Electrolysis of brine. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,603, 16.8.27).—Calcium and magnesium impurities are precipitated and removed from brine (saturated with chlorine for electrolytic purposes) by adding the calculated quantity of sodium fluoride. Not more than 10 mg. of magnesium hydroxide nor 12 mg. of calcium hydroxide per litre of brine are left after treatment, whilst the small amount of sodium fluoride (about 0.1%) which enters the solution has no deleterious effect.

P. E. L. FARINA.

Preparation of large sal ammoniac crystals. CHEM. FABR. GROSS-WEISSANDT G.M.B.H., and P. SEIDLER (B.P. 275,991, 9.8.27. Ger., 10.8.26).—Large crystals are obtained by crystallisation from solutions containing small quantities of aqueous extracts of plants or plant parts obtained, e.g., from woody grasses, weeds, leaves, stalks, foliage, barks, roots, fruits, seeds, etc. The vegetable matter need not be fresh; thus meadow hay, dry shreds of sugar beet, dried malt germs, etc. give good extracts. Large crystals of ammonium chloride are obtained even when salts such as sodium chloride are present. The presence of acids, e.g., hydrochloric acid, is an advantage in the process.

P. E. L. FARINA.

Dissociation of sulphur vapour. I. G. FARBENIND. A.-G. (B.P. 298,599, 17.8.28. Ger., 12.10.27).—Containers, heating elements, etc. for use in the dissociation of the vapour into S_4 and S_2 molecules are constructed of silicon carbide.

L. A. COLES.

Extraction and drying of jellies [gels]. W. J. MÜLLER, H. CARSTENS, and J. DRUCKER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,696,358, 25.12.28. Appl., 17.8.26. Ger., 2.11.25).—See B.P. 279,941; B., 1928, 90.

Catalysts (B.P. 301,577).—See III. **Insecticide** (B.P. 300,439).—See XVI.

VIII.—GLASS; CERAMICS.

The petrographic microscope as an instrument for the glass technologist. H. INSLEY (J. Amer. Ceram. Soc., 1928, 11, 812—828).—The optical properties of crystals are briefly described, and a practical account is given of the use of the petrographic microscope in measuring the optical properties and identifying crystalline compounds in the raw materials and finished products of glass technology. F. SALT.

Physical properties of glass tank block refractories. A. E. MACGEE (J. Amer. Ceram. Soc., 1928, 11, 858—867).—Five glass tank block bodies were tested for crushing strength, porosity, apparent sp. gr., modulus of rupture, modulus of elasticity, impact strength, and thermal shock. Resistance to spalling was predicted from the thermal expansion, elasticity, and transverse strength; bodies highly resistant to spalling had a low and uniform rate of expansion, high tensile and transverse strength, low modulus of rupture, high thermal diffusivity, and were not readily susceptible to thermal fatigue. Thermal fatigue may take place after a time in a body which was strong enough to withstand the initial thermal strains. F. SALT.

Test for adhesiveness of vitreous enamels to metal. W. N. HARRISON and G. T. THALER (J. Amer. Ceram. Soc., 1928, 11, 803—811).—Two strips of enamelled metal are clamped together while they are still in the furnace and then allowed to cool in another furnace from about 500°. The clamped pieces are then tested as a continuous strip of metal under tensile stress, the force necessary to pull the two strips apart being measured. Examination of the area of contact on both strips gives an indication of the nature of the failure, whether due to the enamel-metal bond, or the enamel itself, or both. Enamels containing the oxides of cobalt, manganese, and nickel gave a much stronger bond than enamels without these oxides. F. SALT.

Kallauner and Seger methods of rational analysis [of clays]. W. F. FISHER (J. Amer. Ceram. Soc., 1928, 11, 842—844).—A comparison of the two methods was made on an American kaolin and ball clay. The Kallauner method gave results more closely corresponding with the calculated values than the Seger method. The former is preferred as a method of plant control. F. SALT.

Hydrometer method for determining colloid content [of clays]. P. J. SLEGEL (J. Amer. Ceram. Soc., 1928, 11, 851—853).—The Bouyoucos hydrometer method (cf. B., 1927, 422) of determining the colloid content in suspensions of bentonite and kaolin was investigated. The method was not applicable to viscous suspensions. Hydrometer readings taken on suspensions ground for various periods in the ball mill gave excessively high results. F. SALT.

Determination of iron oxide and titania in clay refractories. W. R. KERR and E. B. READ (J. Amer. Ceram. Soc., 1928, 11, 845—850).—For clays low in silica and unfired clays, direct titration of fusions with potassium bisulphate is sufficiently accurate. An accurate and comparatively rapid method, suitable for all clays,

consists in treating with hydrofluoric and sulphuric acid, dissolving with dilute hydrochloric acid, precipitating the R_2O_3 by ammonia, fusing with potassium bisulphate, and then titrating the iron and titanium oxides.

F. SALT.

X-Ray investigation of the effect of heat on china clays. T. N. McVAY and C. L. THOMPSON (J. Amer. Ceram. Soc., 1928, 11, 829—841).—An English china clay and a washed Georgia kaolin, raw and fired to various temperatures, were examined by the X-ray method to investigate their differences in thermal expansion. The principal constituent of both clays was kaolinite. Between 650° and 950° no diffraction patterns were obtained. At 950° mullite in a finely-divided state was present in both clays, and this increased in quantity and crystal size with a rise in temperature. Another crystalline phase, probably alumina, was also found in the Georgia clay when heated to 950°, 1050°, and 1100°. Cristobalite was present in the Georgia clay fired to 1225° and above, and in small amounts in the English clay between 1250° and 1275°. This cristobalite is the cause of the inflexion occurring in the thermal expansion curves of the fired clays at about 200°. The exothermic reaction at 900° can scarcely be due to crystallisation, since the diffraction patterns at this temperature did not indicate the presence of a large amount of any crystalline phase. F. SALT.

Increase in refractoriness in ceramic bodies in interrupted heat treatment. H. B. HENDERSON and J. H. CALDWELL (J. Amer. Ceram. Soc., 1928, 11, 795—802).—The phenomenon known as "freezing"—an increase in refractoriness due to interruption in the heat treatment, or to a prolonged drop in temperature during the normal firing—was studied on certain raw clays, pottery bodies, and cone mixtures. Cones 9 to 11 inclusive were most susceptible to "freezing." A tendency to "freeze" occurred when the feldspar content of the cone mixture fell below 25% and the clay content exceeded 20%. The difficulty was overcome in a new series of cone mixtures by the use of magnesia in the form of talc. "Freezing" appeared to be a function of the ratio of the feldspathic material to the other aluminous minerals in the mixture, and not due to the constitution of any one mineral. F. SALT.

Effects of coal ash on refractories. T. N. McVAY and K. R. HURSH (J. Amer. Ceram. Soc., 1928, 11, 868—873).—The effects of coal ash were studied on refractories made of diaspore, fireclay with little quartz, fireclay with much quartz, a mixture of diaspore and fireclay, and andalusite. The tests were carried out in a rotary furnace for periods varying from 7½ to 30 hrs. at temperatures from 1500° to 1600°. Petrographic examination of the slags formed showed them to consist of magnetite, mullite, and glass. The formation of a dense, non-porous layer behind the slag tended to prevent the erosion of fireclay bricks. Diaspore bricks were more deeply penetrated by slag, because they remained porous at the furnace temperatures. Bricks slagged by different kinds of coal ash showed the same characteristics. Varying the period and temperature of slagging did not change the reactions taking place. F. SALT.

Variations in pyrometric cone equivalents of silica cements and fireclays. S. S. COLE (J. Amer. Ceram. Soc., 1928, 11, 854—857).—The effect of grinding and calcining on the pyrometric cone equivalent [p.c.e.] test was studied on a number of silica cements and fireclays, which were used "as received," ground to pass 65-mesh, and calcined at 1400° and then ground to the same mesh. The tests were duplicated by two different operators, to discover possible variations due to the "human element." The grinding caused a reduction of the p.c.e. of about $1\frac{1}{2}$ cones (average), whilst calcination reduced it by nearly 2 cones. F. SALT.

See also A., Jan., 26, **Suspensions of kaolin** (DUBRISAY and ASTIER). 44, **Wing top oxygen-gas burner [for glass]** (ROBERTSON).

PATENTS.

Manufacture of [unglazed] ceramic ware. CERAMIC PATENT HOLDINGS, LTD., and J. W. MELLOR (B.P. 302,519, 31.1.28).—2—5% of a flux consisting of magnesium or an alkaline-earth borate and/or an alkali silicate is added to the usual constituents, and the articles are fired at a temperature lower than that normally used for biscuit firing, e.g., at that of the glost kiln. L. A. COLES.

Manufacture of ceramic products. A. J. COULION (F.P. 620,760, 11.8.26. Belg., 3.9.25).—The plastic mass used in making bricks or pottery ware should contain less than 14% of moisture. The moulded shapes are introduced directly without drying into a furnace which contains two adjacent series of chambers which serve successively for drying, preheating, burning, and cooling. The goods are first treated with a current of cold air, which is gradually replaced by hotter air until the articles are thoroughly dried and shrunk. The heating gases are then introduced and, after burning, the goods are cooled in a current of air which is thereby heated for use in drying and preheating fresh charges. A. R. POWELL.

Manufacture of ceramic articles. D. W. ROSS and J. M. LAMBIE (U.S.P. 1,690,208, 6.11.28. Appl., 31.3.25. Renewed, 29.3.28).—Apparatus is described for providing grog in assorted grain sizes, sufficient clay for bonding purposes, a deflocculating agent, mixing water with these materials to form a paste, mixing the paste in a vacuum, and moulding the paste into shapes. F. SALT.

Composition and mould made therefrom, and method of casting pottery in the mould. J. D. WIGGIN and M. M. REMMES, Assrs. to H. B. WIGGIN'S SONS CO. (U.S.P. 1,690,708, 6.11.28. Appl., 29.10.24).—Methods are described for preparing pottery casting slip containing a soluble carbonate, plaster moulds containing a substance which prevents reaction between the carbonate of the slip and the calcium sulphate in the moulds, casting the slip in the mould, removing the cast article, and firing it. F. SALT.

Magnesite, dolomite, or lime bricks. J. CRASSONS and H. BENABENQ (F.P. 620,203, 11.8.26).—The raw material is sintered, ground finely, and mixed with 15—

30% of powdered alkali chloride. The mixture is pressed into shape under high pressure, and the products are burnt for 34 hrs. at 1700°. A. R. POWELL.

Refractory building material (B.P. 301,547).—See IX.

IX.—BUILDING MATERIALS.

Effect of calcium chloride and calcium oxychloride on Portland cement. W. J. PITT (Chem. Eng. Min. Rev., 1928, 21, 65—70).—Contrary to the views of previous workers, calcium chloride acts as an accelerator when used in admixture with Portland cement; e.g., addition of 0.5% of calcium chloride reduced the time of the final set of cement from 4 to 2 hrs., and of 0.5% of calcium oxychloride to 3 hrs. With calcium chloride an increase in tensile strength was noted up to an age of 5 days, with subsequent slight decrease. The use of calcium oxychloride is preferred for reasons of cost and manipulation; it also was the most important chemical agent affecting the decrease of expansion of cement mortars to a marked degree. A 2% admixture of calcium oxychloride gave the greatest increase in early strength together with a decided improvement at later ages, though the cement was less hydraulic. The fracture of such mixtures after 24 hrs. setting showed a porcelain structure without segregation or "laitence." C. A. KING.

Shrinkage effect of celite in mortar or concrete. A. S. LEVENS (Eng. News-Rec., 1928, 101, 507—508).—The addition of celite to mortar or concrete in order to improve its workability has also the effect of increasing the amount of shrinkage when the quantity used exceeds 2% of the weight of cement present. Very little additional shrinkage is caused by adding 2 and 3% of celite to 1:1½:3 and 1:2:4 mixes of concrete, respectively, but a 5% addition to a 1:3:6 mix causes a marked increase. C. JEPSON.

Pyrometric cone equivalents of silica cements. COLE.—See VIII.

PATENTS.

Aluminous cement. VER. DEUTS. EISENPORTLANDZEMENT-WERKE E.V. (F.P. 620,539, 21.8.26).—Ordinary aluminous cement is mixed with a suitable proportion of finely-ground blast-furnace slag. This mixture develops much less heat when mixed with water than does the ordinary cement, and products made from it are stronger and more uniform.

A. R. POWELL.

[Quick-setting] cementing compound. J. MAYES, Assr. to MAYES BROS. TOOL MANUF. CO. (U.S.P. 1,693,488, 27.11.28. Appl., 10.3.27).—The composition, which is mixed with water for use, contains approx. 8 pts. of moulding plaster and 1 pt. of litharge. L. A. COLES.

Composition materials [cellular cement]. E. C. R. MARKS. From INSULEX CORP. (B.P. 301,344 and 301,359, 24.8.27).—Compositions for admixture with water to yield cellular products on setting comprise calcined calcium sulphate, material for the formation of gas bubbles, e.g., calcium carbonate and aluminium sulphate, and a quantity insufficient to exert an appreciable influence on the hydration and setting time of the calcium sulphate

of (A) material having the double function of emulsifying the gas and reinforcing the bubble films, *e.g.*, soap or gum arabic, or (B) bubble-strengthening substances, *e.g.*, talc, silica, lamp-black, etc. L. A. COLES.

Shaped masses. [Slag bricks.] S. YOSHIMOTO, T. OKUMURA, and S. NAKAMURA (F.P. 620,456, 18.8.26. Jap., 2.6.26).—Copper-refinery slags are cast into brick shapes in suitable moulds, and when the bricks are still red-hot they are covered with a mixture of mica, powdered iron, sulphur, and straw, and allowed to cool slowly under a layer of ashes. A. R. POWELL.

Refractory building material. G. KNUDSEN, V. M. GOLDSCHMIDT, and R. KNUDSEN (B.P. 301,547, 3.9.27. Addn. to B.P. 260,298; B., 1928, 232).—Ground olivine rock or olivine sand is subjected to treatment causing recrystallisation and cementing together of the particles; *e.g.*, it is heated at a suitable temperature in the presence of substances such as magnesium, alkali, or alkaline-earth salts, etc., which assist the recrystallisation of magnesium orthosilicate. Binders, *e.g.*, sodium silicate, syrup, etc., iron compounds, *e.g.*, magnetite, aluminium compounds, *e.g.*, clay, and other substances may be added, and the mixtures may be moulded before heating.

L. A. COLES.

Manufacture of heat- and sound-insulating material. P. H. USSING (B.P. 299,266, 13.4.28. Denm., 22.12.27. Addn. to B.P. 289,830; B., 1928, 896).—Metal soaps, other than those of aluminium, such as zinc soaps, magnesium soaps, etc., may be used for the curing of the washed fibres upon which they are precipitated.

H. ROYAL-DAWSON.

Composition to be added to cement mixings for cold glazes or the like. J. LANGBEIN (U.S.P. 1,695,808, 18.12.28. Appl., 20.5.25. Fr., 18.2.25).—See B.P. 247,947; B., 1926, 918.

Coal tar (B.P. 301,645).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Recrystallisation [of mild steel] after hot-rolling. W. TAFEL, H. HANEMANN, and A. SCHNEIDER (Stahl u. Eisen, 1929, 49, 7—12).—The effect of hot-rolling on the grain size of mild steel after recrystallisation is shown by means of a space model of which the axes are the temperature of rolling, the percentage reduction in thickness, and the average grain size. The original grain size appears to have no effect whatever on the final grain size, but a coarse grain structure is obtained if the final rolling is made at 700—900°. To obtain a fine-grained structure the final work should be as heavy as possible and the metal heated above 925°.

A. R. POWELL.

Effect of repeated stresses on the magnetic properties of steel. M. F. FISCHER (U.S. Bur. Stand. J. Res., 1928, 1, 721—732).—Repeated stresses of the reversed-bending type affect the magnetic properties of steel, but are not characteristic of the effect of a fatigue crack transverse to the direction of magnetisation, nor is any indication given of the imminence of failure. The magnitude of the magnetic changes is a function of the applied stresses, the intensity of its

application, and the value of the magnetising force. From similarity of observed changes resulting from the removal of static loads it would appear that the magnetic changes induced by repeated stresses are such as would be caused by partial relief or redistribution of initially existing internal stresses, and are so large as to mask any small effect which might result directly from a fatigue crack. The beneficial result of understressing steel is probably due to the rearrangement of internal stresses.

C. A. KING.

Ternary chromium steels. J. POMEY and P. VOULET (Rev. Mét., 1928, 25, 665—667).—Chromium steels containing nickel and cobalt show a point of inflexion on the dilatometric curve above the A3 point. With a steel containing 10% Ni, 23% Cr, and 4% W, the Ac3 point was between 700° and 800° and the Ac4 point between 975° and 1000°. This point is marked by a falling-off in the magnetic properties as well as by volume changes, and occurs at a slightly lower temperature during cooling.

A. R. POWELL.

Chromium steel with 20% Cr. A. MICHEL and P. BÉNAZET (Rev. Mét., 1928, 25, 668).—Quenching a steel with 19% Cr, 0.8% Ni, and 0.54% C from 1250° in oil preserves its austenitic structure. Subsequent annealing at various temperatures in the Chevenard dilatometer results in negative anomalies in the expansion curve at 150°, 260°, and 600°. The last-named is attributed to precipitation of carbide from the super-saturated austenite phase, and the others to changes in the α -phase produced on annealing. This behaviour is compared with that of a steel containing 6% Cr, 6% W, and 25% Co, which recalesced vividly on annealing it in the austenitic state at 700°, and it is concluded that a high chromium content retards this recalescence and tends to retain a martensitic instead of a pearlitic structure.

A. R. POWELL.

Applications of stainless iron in the nitric acid industry. W. M. MITCHELL (Chem. Met. Eng., 1928, 35, 734—736).—Stainless iron containing 16—19% Cr and under 0.12% C is not resistant to nitric acid made from Chilean nitrate, owing to the presence of hydrochloric acid as an impurity, but is highly resistant to pure synthetic acid. Silicon should not exceed 1% nor manganese 0.4%. Plates of this alloy are used for building absorption towers and tank wagons. Rivets suffer from air-hardening if excessively heated, but the technique of handling them is now understood. Seamless drawn tubing up to 5—6 in. is also available. For heat exchangers chromium-nickel alloy is suitable. Flanges, valves, pipe fittings, etc. are usually cast, and a higher carbon content is necessary, but forgings may also be used.

C. IRWIN.

Determination of vanadium in steel. K. SWOBODA (Chem.-Ztg., 1928, 52, 1014—1015).—2 g. of steel are dissolved in 50 c.c. of sulphuric acid (1:6), and the solution is oxidised with nitric acid and heated with 50 c.c. of 10% ammonium persulphate solution. Excess of ammonia is then added and the whole warmed to dissolve tungsten if present. Addition is then made of another 50 c.c. of ammonium persulphate and of 100 c.c. of acid ammonium molybdate solution, when the greater

part of the separated iron goes into solution, the remainder being taken up by the addition of nitric acid. 10% sodium phosphate solution is then added to the hot solution drop by drop, and the liquid is boiled and filtered, the precipitate being washed with acid ammonium sulphate solution. The filter paper and its contents are heated to fuming with nitric, sulphuric, and phosphoric acids, and the liquid is diluted with water, treated with 5 drops of nitric acid and 50 c.c. of hydrochloric acid, and again evaporated to fuming. After cooling, the whole is diluted to 250 c.c., heated to 80°, and titrated with potassium permanganate.

F. R. ENNOS.

Machinability [of metals]. E. G. HERRERT (Inst. Mech. Eng., 1929. Advance proof. 27 pp.).—No quantitative relationship exists between the machinability of metals and the physical properties existing before and after machining. The hardness of the chips removed by a metal-cutting process indicates the hardness induced in the metal and is a measure of machinability. The original hardness of metals bears no relation to the aptitude for machining; the pendulum work-hardening test gives a general indication in the case of ductile steels, but none in the case of brittle metals, e.g., cast iron. In the actual cutting operation the cutting is done by the built-up edge which forms immediately on the commencement of cutting. The efficiency of cutting and therefore the machinability depends on the natural angles of the built-up edge rather than on that of the tool. Very soft steel has a greater blunting effect on the tool than harder steel; this is attributed to the less efficient cutting angles of the built-up edge. Such edges on acute-angled tools are of a temporary character, being formed and discarded as often as 50 times per sec. Obtuse-angled roughing tools form permanent built-up edges composed of many layers welded together. A power hack-saw with permissive feed and a standard, sharpened saw blade provides a convenient means of measuring machinability in the workshop, using care in applying the results to dissimilar conditions. C. A. KING.

Corrosion of metal pipes by direct and alternating currents. W. BECK (Z. angew. Chem., 41, 1361—1367).—A review of recent work on the corrosion of underground metal pipes by stray currents from electric railways or tramways. A. R. POWELL.

Corrosion of aluminium bronze tubes, its causes and prevention. A. MERZ and E. BRENNKE (Korrosion u. Metallschutz, 1928, 4, 136—140; Chem. Zentr., 1928, ii, 491).—Corrosion by alkali chloride solutions takes place at plastic crystals; suitable cold-working, followed by heat treatment, is necessary. A. A. ELDRIDGE.

Determination of cadmium in cadmium-copper wire. C. BLAZEX (Chem. Eng. Min. Rev., 1928, 21, 74—76).—The loss in weight of a sample when heated at 950° or higher in hydrogen is determined and represents the volatilisation of cadmium and any contained oxygen. The specimen is usually in a form suitable for hammering into strip or foil, in which form complete volatilisation may be effected in 15 min. Cadmium-copper is usually a high-grade alloy, and

serious error due to volatilisation of zinc, lead, sulphur, bismuth, etc. is not likely to occur, or the presence of these elements is previously known. A small deduction (0.05% maximum) should be made for oxygen which may be estimated by microscopical examination of the oxide particles present. Volatilisation of cadmium in a current of neutral gas followed by removal of oxygen with hydrogen is less sure owing to the volatility of cadmium monoxide. C. A. KING.

Reports of investigations: [Canadian] ore-dressing and metallurgical laboratories. C. S. PARSONS, R. K. CARNOCHAN, and J. S. GODARD (Canada Dept. Mines, Ore Invest., 1926, 3—94).—Details of the results obtained in gravity and oil-flotation concentration tests on 8 complex zinc-lead ores, 7 copper ores, and 4 gold ores are given, together with an account of work done to enrich or clean bentonite, graphite, and calcite. A. R. POWELL.

Reports of investigations: [Canadian] hydro-metallurgical laboratory. [Ferric chloride leaching of ilmenite and copper pyrites.] R. J. TRAILL and W. R. McCLELLAND (Canada Dept. Mines, Ore Invest., 1926, 95—109).—Reduction of ilmenite with carbon at 1000° followed by leaching of the product with ferric chloride solution effects removal of 97% of the iron in the ilmenite leaving a residue containing 76% TiO₂ and 3.6% Fe. Electrolytic iron may be recovered from the solution and ferric chloride regenerated by electrolysis in a divided cell using a current density of 35 amp./ft.² at the cathode. A rotating cathode and circulation of the catholyte gives the best results. The electrolyte should have p_H 3.7—3.9 and an average temperature of 80—85°; the voltage varies from 2.5 to 3 according to the conditions. A similar process gives good results on high-grade copper pyrites provided that the ore is first heated in a closed retort at 700—800° to expel sulphur in excess of that required to form cuprous and ferrous sulphides. The product from this treatment is digested with ferric chloride, and the copper removed from the solution by precipitation on scrap iron; the ferrous chloride solution is then electrolysed as described above to remove part of the iron and convert the remainder into ferric chloride for further use in the process. The residue from the leaching operation may be distilled to recover sulphur, and the non-volatile portion smelted to extract its content of gold and silver. A. R. POWELL.

Selective flotation as applied to Canadian ores. C. S. PARSONS (Canada Dept. Mines, Ore Invest., 1926, 112—130).—A general discussion of modern flotation practice with especial reference to the selective flotation of zinc, lead, copper, and iron sulphides from various complex Canadian ores. A. R. POWELL.

Methods proposed and in use for refining aluminous ores. C. L. MANTILL (Chem. Met. Eng., 1928, 35, 746—750).—In addition to the usual Bayer process the following are described. *Hall process.*—Ground bauxite is sintered with coal dust, coke is added, and the whole smelted in an electric furnace. Ferrosilicon and ferrotitanium are tapped off and alumina is blown over from the top. This process is in commercial operation in America and can deal with

high-silica bauxite. *Pedersen process*.—Iron ore, limestone, and bauxite are smelted in an electric furnace and an aluminous slag is obtained. This is leached with sodium carbonate solution containing 10% of caustic soda. This process is in operation in Norway. *Haglund process*.—Bauxite, anthracite, and pyrites are treated in an electric furnace. Alumina and aluminium sulphide result, forming a slag which is liquid at 1100°. It is leached with water and steam and separated from oxides of iron and titanium. A works using this process is projected in Italy. The *Berger, Serpek, and Svendsen processes* (described) have not succeeded in practice. In the *Blanc process* for leucite, iron is removed magnetically, and the rock treated with hydrochloric acid. Aluminium chloride is decomposed by heating at 350° in iron tanks and the hydroxide is calcined. In this process aluminium is a product secondary to potash. The *Halvorsen process* treats labradorite with nitric acid. The acid solution is treated in an autoclave with ammonium salts to render the alumina precipitate powdery. Neither of these two processes is in operation as a method for the production of the metal. The same remark applies to Sherwin's modification of the Bayer process in which excess of silica is fixed with lime. *Laist process*.—Alunite or clay is treated with concentrated sulphuric acid and heated at 300–400°. Potash alum is obtained and decomposed by ignition at 1000°. *Specketer process*.—Clay etc. is treated with a mixture of sulphuric and hydrochloric acids, salt is added, the solution evaporated, and the residue heated to 700°. The leached product is treated as in the Bayer process. This method has not been proved commercially. It is concluded that no method using any material other than bauxite is yet of industrial significance. C. IRWIN.

See also A., Jan., 17, **X-Ray spectrography of copper and some brasses** (LOISEAU). 19, **Titanium in titaniferous slags** (CARSTENS). 24, **Aluminium-calcium alloys** (BOZZA and SONNINO). 36, **Catalytic properties of arsenic** (MOUREU, DUFRAISSE, and BADOCHÉ). 42, **Test for arsenic** (DAUVÉ). **Determination of silver and cadmium potentiometrically** (MÜLLER and HENTSCHEL). 43, **Determination of chromium** (RIPAN). 54, **Chromium powder in organic synthesis** (CHAKRABARTY and DUTT).

Measurement of gas temperatures. SCHMIDT.—See I. **Copper sulphate from brass turnings.** EYUSHENKO.—See VII. **Vitreous enamels on metal.** HARRISON and THALER.—See VIII.

PATENTS.

Apparatus for the manufacture of pig iron. G. DE BETHUNE (U.S.P. 1,693,916, 4.12.28. Appl. 15.7.25. Belg., 5.3.25).—The apparatus consists of a tuyère made up of two cylindrical portions of different diameters connected by an intermediate tapering portion, the innermost cylinder having a greater length and smaller cross-section than the other and being provided with means for cooling its walls; the comminuted material is delivered to the cylindrical portion of greater diameter through a channel below which an injector delivers heated air to the interior of the tuyère,

whilst a second injector, fixed co-axially on the interior of the first, delivers unheated air under pressure.

M. E. NOTTAGE.

Process for obtaining iron from ores. VEREIN. STAHLWERKE A.-G. (B.P. 297,096, 29.5.28. Ger., 16.9.27).—The reaction speed in the chlorination of the ores with chlorine or hydrogen chloride can be considerably increased by carrying out the process under increased pressure, which may also be changed once or repeatedly. The ferric chloride formed is volatilised by heat and subsequently reduced. M. E. NOTTAGE.

Hardening the marginal layers of cast iron alloys. F. KRUPP A.-G. (B.P. 287,156, 20.1.28. Ger., 17.3.27).—A suitable alloyed cast iron (alloyed, e.g., with aluminium), which may or may not contain in addition from 0.3 to 18% of silicon, manganese, chromium, nickel, cobalt, vanadium, molybdenum, tungsten, titanium, or zirconium, is subjected to the action of gases which contain nitrogen in a combined form, e.g., ammonia or cyanogen, at about 500° for more than 24 hrs.

M. E. NOTTAGE.

Manufacture of black-heart, malleable, cast iron from white cast iron. T. KIKUTA, Assr. to TOBATA IMONO KABUSHIKI KAISHA (U.S.P. 1,692,842, 27.11.28. Appl., 29.9.26).—White cast iron is maintained at 850–950° for 15–60 hrs., cooled to 730–650°, maintained at the latter temperature for 20–50 hrs., removed from the furnace, and cooled rapidly; by this means the use of packing material is avoided.

M. E. NOTTAGE.

Treatment for protective purposes of articles composed of iron. W. KLEPSCH (B.P. 302,254, 10.8.27).—Steel or iron is cast in a sand mould prepared with a decarbonising substance so that the casting is decarbonised to a depth of 0.3–40 mm. while cooling. The decarbonising agent may be mixed with the foundry sand, or painted or sprayed on to the surface of the mould before casting. C. A. KING.

Improving the properties of iron-beryllium alloys. SIEMENS & HALSKE A.-G. (B.P. 288,579, 11.4.28. Ger., 12.4.27).—The alloys, which may or may not contain up to about 40% of one or more other elements such as aluminium, nickel, copper, tin, carbon, and silicon, are quickly cooled from above 550° and reheated at 250–500°; they may be mechanically worked before the heat-treatment. M. E. NOTTAGE.

Copper alloy. F. HEUSLER (U.S.P. 1,692,936, 27.11.28. Appl., 17.7.26).—Alloys comprising copper with 0.5–20% Mn and 0.3–8% Si are cast, rolled, forged, annealed above 450°, quenched, and hardened by tempering at 200–350°. A. R. POWELL.

Chemically resistant and machinable copper alloys. F. DOEBLIN (G.P. 451,270, 10.4.25. Addn. to G.P. 447,247; B., 1928, 609).—In addition to the constituents mentioned in the chief patent 1% of arsenic is included in the alloy. The resistance to hydrochloric acid and chlorine solutions is thereby increased.

A. R. POWELL.

Working-up of copper- and zinc-containing ores. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 288,266, 22.3.28. Ger., 7.4.27).—Burnt pyrites con-

taining copper and zinc with a small percentage of sulphur is subjected to a chloridising roast at 580—650° to convert the zinc into chloride and the copper into sulphate. The roasted product is extracted with a small volume of water, which removes nearly all the zinc and only a relatively small amount of the copper, then with much water or dilute acid to dissolve the copper salts. The copper is removed from the zinc solution by treatment with zinc oxide, and from the copper solution by deposition on scrap iron.

A. R. POWELL.

Extraction of tin or arsenic from ores etc. H. L. SULMAN and H. F. K. PICARD (B.P. 301,553, 13.6., 12.7., 13.9., and 16.11.27).—Finely-ground tin ore is heated together with a reducing agent and a sulphurising agent, *e.g.*, sulphur or iron pyrites, at 750—850° until all the arsenic is volatilised as sulphide and the tin converted into stannous sulphide without appreciable loss by volatilisation. The product is leached with ammonium polysulphide solution to dissolve the tin, and the pregnant solution is boiled to expel ammonium sulphide, which is recovered for further use, and to precipitate stannic sulphide, which is roasted to oxide prior to reduction to metal in the usual way.

A. R. POWELL.

Recovery of light metals, especially aluminium, from ores. ALUMINIUM IND. A.-G. (Swiss P. 121,604, 14.12.25).—The ore is reduced under pressure with carbonaceous material in the inner chamber of an electric furnace consisting of two concentric chambers with a connexion at the bottom. The resulting alloy flows into the outer chamber and is there distilled under reduced pressure, the vapours of the light metal being conducted below the surface of a molten bath of the same metal. Heating of the outer chamber is effected with carbon electrodes the temperature of which is maintained above the decomposition temperature of the light-metal carbide.

A. R. POWELL.

Soldering [of aluminium]. H. C. P. WEBER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,689,899, 30.10.28. Appl., 30.7.20).—Ammonium stannous chloride is fused between the two pieces of metal and pressure is applied; the heat of the reaction is sufficient to cause the liberated tin to diffuse into both pieces of aluminium and so make a perfect joint.

A. R. POWELL.

Aluminium alloys. T. GOLDSCHMIDT A.-G. (B.P. 283,927, 10.1.28. Ger., 20.1.27).—An aluminium alloy containing 1.5% of magnesium silicide, 1.2% Cu, 0.5% Ti, and, if desired, 0.8% Mn, after being mechanically worked, is heated from 520° to 550° during $\frac{1}{2}$ hr., rapidly cooled, and then aged for 6—24 hrs. at (a) 110—160°, to give it high elongation values; (b) 130—145°, for increased mechanical strength with high elongation values; or (c) 145—160°, for high mechanical strength. The elongation properties and resistance to corrosion may be enhanced by postponing the ageing above 110° for 6—48 hrs. after the heat treatment and cooling; the mechanical strength may be further improved by mechanically working the material between the heat treatment and cooling on the one hand, and the ageing at above 110° on the other.

M. E. NOTTAGE.

Production of an aluminium alloy. D. R. TULLIS (B.P. 302,440, 12.10.27).—An alloy of aluminium con-

taining, in addition to boron, 3—16% Zn, 1—6% Si, and 0.2—1% Cr or Mn (cf. B.P. 272,326; B., 1927, 606).

C. A. KING.

Electrothermic manufacture of aluminium-silicon alloys practically free from carbide. ELEKTRIZITÄTSWERK LONZA (Swiss P. 121,605, 20.4.26. Ger., 12.5.25).—Mixtures of alumina and silica, or natural aluminium silicates containing such proportions of the constituents that the resulting alloy contains 34—47% Si, are reduced electrothermally with carbon. The product is relatively free from carbide and, after addition of more aluminium, can be used for the production of light alloy castings.

A. R. POWELL.

Alloy. J. A. GANN, Assr. to DOW CHEM. CO. (U.S.P. 1,688,043, 16.10.28. Appl., 27.10.21).—The alloy contains, approx., Mg 90%, Al 9%, and Cu 1.5%.

H. ROYAL-DAWSON.

Working of zinc. W. M. TOWNE, Assr. to E. W. BLISS CO. (U.S.P. 1,686,353, 2.10.28. Appl., 27.7.25).—Zinc is extruded from a suitable press through the annular space between two dies at a temperature between 150° and 300°.

A. R. POWELL.

Condensation of zinc and cadmium vapours. T. GJERSTAD (N.P. 41,476, 24.5.22).—The vapours from the retorts are passed downwards through vertical cylindrical or conical condensers at a high velocity, and at the lower ends of the condensers are subjected to a centrifugal action whereby the condensed metal particles are caused to coalesce without the formation of blue powder.

A. R. POWELL.

Production of zinc dust. F. P. LANNON, JUN., Assr. to AMER. SMELTING & REFINING CO. (U.S.P. 1,688,426, 23.10.28. Appl., 1.2.27).—Zinc fumes from a retort are circulated rapidly to solidify the particles before collection.

C. A. KING.

Manufacture of aluminium from alumina. A./S. MALMINDUSTRI (N.P. 40,864, 27.7.23).—Alumina is reduced with calcium silicide together with a suitable flux. Calcium silicide may also be added to the electrolyte in the manufacture of aluminium by the usual electrolytic process.

A. R. POWELL.

Improving the qualities of nickel-beryllium alloys. G. MASING and O. DAHL, Assrs. to SIEMENS & HALSKE A.-G. (U.S.P. 1,685,570, 25.9.28. Appl., 11.10.27. Ger., 16.9.26).—Nickel alloys with up to 5% Be are heated above 700°, cooled, and worked mechanically.

A. R. POWELL.

Magnetic alloys. H. F. PORTER (B.P. 283,931, 16.1.28. U.S., 20.1.27).—The alloys claimed contain 3—10% Cu, 35—55% Ni, 55—35% Fe, and less than 0.05% C. The copper is melted first, and its oxygen content is utilised in burning out the traces of carbon from the iron; a small quantity of manganese is added to remove sulphur, followed by magnesium to remove oxygen.

A. R. POWELL.

Magnetic alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 302,394, 16.9.27).—Magnetic alloys having a constant permeability of 600 in fields up to 0.1 gauss consist of 50—90% Ni, 5—30% Co, and the balance (not less than 5%) manganese. Up to 6% of the nickel may be replaced by chromium, molybdenum,

silicon, tungsten, aluminium, or vanadium, and the manganese content is preferably 7—12%.

A. R. POWELL.

Welding [electrodes for nickel]. INTERNAT. NICKEL Co., N. B. PILLING, and J. G. SCHOENER (B.P. 301,687, 22.3.28).—For the arc-welding of nickel the welding rod comprises a core of pure nickel malleabilised by the addition of magnesium, and a coating consisting of 19% Ti, 6% Ca, 5% Al, 17% Si, and 53% Fe.

A. R. POWELL.

Production of dense, even deposits of chromium. LANGBEIN-PFANHAUSER-WERKE A.-G. (G.P. 452,595, 5.11.26).—The article to be chromium-plated is first given a thin coating of a noble metal such as silver, gold, or mercury. After this treatment a good deposit of chromium is obtained even in deeply-recessed parts.

A. R. POWELL.

Flotation of ores. A. H. NEY (U.S.P. 1,683,724, 11.9.28. Appl., 27.12.26).—The product of the action of sulphur chloride on *o*-toluidine or other arylamine having one, or two, unsubstituted *o*-positions, or the *o*-aminothiophenol produced by its hydrolysis, is used as a flotation agent.

C. HOLLINS.

Production of a [nickel] catalyst. K. SCHIRMACHER, F. STOLZ, H. SCHLICHENMAIER, and W. KROHS, Assr. to I. G. FARBEIND. A.-G. (U.S.P. 1,695,666, 18.12.28. Appl., 19.7.26. Ger., 22.7.25).—See B.P. 255,884; B., 1927, 528.

Slag bricks (F.P. 620,456).—See IX. **Heat-treating furnace** (U.S.P. 1,694,964). **Electrodeposition** (U.S.P. 1,963,683 and Austr. P. 107,315). See XI.

XI.—ELECTROTECHNICS.

See A., Jan., 25, **Micro-electrodialysis apparatus** (BAER). 36, **Electrolysis of copper sulphate solutions** (CHANOZ). **Preparation of persulphates** (ESSIN). **Electrolytic preparation of potassium permanganate** (RAPIN). 42, **Potentiometric analysis** (MÜLLER and KOGERT). 44, **Automatic apparatus for p_H measurement** (LASSIEUR). 56, **Electrochemical oxidation of α -methyl-naphthalene** (FICHTER and HERSZBEIN). 109, **Use of quinhydrone electrode** (MCKIBBIN and PUGSLEY). 110, **Technique of electro-dialysis** (ETTISCH and EWIG). **Electrolysis in bio-chemistry** (KEIL and SCHIECK).

Insulating oils. BUTKOV.—See V. **Magnetic properties of steel.** FISCHER. **Corrosion of metal pipes by currents.** BECK.—See X. **Measurement of colour.** DE LA BRUÈRE.—See XV.

PATENTS.

Electric furnaces. [A] J. C. WOODSON, and [B] O. A. COLBY, Assrs. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,874, and 1,695,882, 18.12.28. Appl., [A] 28.9.26, [B] 7.10.26).—(A) Resistors are each supported by a pair of removable metallic sections, each having a recessed receptacle containing insulating blocks and being held in assembled relation. (B) Refractory insulating blocks, having a pair of looped resistors positioned in grooves, are each supported by rods removably carried by members secured to the refractory insulating furnace walls.

J. S. G. THOMAS.

Heat-treating [electric] furnace. H. O. BREAKER (U.S.P. 1,694,964, 11.12.28. Appl., 27.2.24. Renewed, 14.6.27).—A removable furnace bottom can be raised into and lowered out of co-operative relationship with the other parts of a furnace having fixed top and side walls, and means, connected with the furnace, are provided for receiving gas displaced from the furnace, and for returning it to the furnace. J. S. G. THOMAS.

Manufacture of electron-emitting bodies. A. LEDERER (B.P. 288,561, 12.9.27. Austr., 12.4.27).—More than 2% of thorium is alloyed with one or more metals of the platinum group, more especially with osmium either alone or with other highly refractory metals, the process being carried out in a reducing atmosphere or a vacuum.

J. S. G. THOMAS.

Electrolytic cell. S. T. WOODHULL, Assr. to AMRAD CORP. (U.S.P. 1,695,989, 18.12.28. Appl., 23.6.26).—A filming electrode, comprising a unit sheet having an extended surface formed of a number of closely spaced folds, is attached to the rigid arms of a filming electrode support, and is arranged with a non-filming electrode in an electrolytic cell.

J. S. G. THOMAS.

Apparatus for electrodeposition. E. M. WANAMAKER, Assr. to ANACONDA SALES Co. (U.S.P. 1,693,683, 4.12.28. Appl., 19.8.26).—An electrolytic cell having a movable frame carries anodes and cathodes, and a pair of shoes making contact, respectively, with a positive and a negative bus-bar arranged near the cell. Contact between the electrodes and the respective shoes is provided.

J. S. G. THOMAS.

Controlling the thickness of electrolytic deposits. L. HERRMANN (Austr. P. 107,315, 26.4.26).—The anode used is of the same shape as the article to be plated except that at places on the anode opposite to those on the cathode where a thin deposit is required the anode is recessed, and at places opposite those where a thick deposit is required the anode is provided with prominences.

A. R. POWELL.

Electrolysis of brine (B.P. 300,603).—See VII. **Magnetic alloys** (B.P. 283,931 and 302,394). **Welding electrodes** (B.P. 301,687). **Chromium plating** (G.P. 452,595).—See X. **Insulating varnish** (B.P. 301,341).—See XIII. **Purification of sugar** (B.P. 302,375).—See XVII. **Pasteurising milk** (U.S.P. 1,692,874).—See XIX. **Blasting fuses** (B.P. 281,239).—See XXII.

XII.—FATS; OILS; WAXES.

Fats. X. **Thiocyanate determination of fats containing linolenic acid.** Analysis of linseed oil. H. P. KAUFMANN and M. KELLER (Z. angew. Chem., 1929, 42, 20—23).—An introduction to the application of the thiocyanogen determination of fats to specimens containing linolenic acid (linseed oil), which is complicated by the impossibility of obtaining a pure sample of natural linolenic acid, and the fact that an equation with four unknowns is involved, of which only three can be determined titrimetrically. To overcome the second difficulty the proportion of saturated acids in the total unsaponifiable acids of linseed oil is determined by methods previously published (cf. Kaufmann, B., 1928, 824). Bertram's method, in which the total saturated

and unsaturated acids are oxidised and the hydroxy-acids so formed separated from the unchanged saturated acids by light petroleum, gives values about 2% higher than the lead salt-alcohol method (Twitchell), the values obtained for a normal linseed oil by the two methods being 9–11% and 7–9%, respectively. A comprehensive summary of the literature relating to linolenic acid is given. J. W. BAKER.

Soaps from organic bases [hydroxyethylamine soaps]. R. B. TRUSLER (Oil & Fat Ind., 1928, 5, 333–347).—Soaps are formed by the combination of alkylamine and arylamine bases with fatty acids, the methyl- and ethyl-amine compounds resembling the corresponding ammonium soaps. Of particular interest are the soaps of the mono-, di-, and tri-hydroxyethylamines. These soaps are soluble in all proportions in most organic solvents, and dilute aqueous solutions resemble those of sodium soaps, but on prolonged keeping hydrolysis of the stearate and palmitate occurs. Aminoethyl stearate is a wax-like solid. The oleates are viscid liquids and act as emulsifiers for fatty and mineral oils (oil-in-water type of emulsion), disinfectants, clays, etc.: they possess considerable detergent properties and are useful as dry-cleaning soaps. 0.3–1% of aminoethyl oleate dissolved in lubricating oils increases their viscosity considerably. The soaps are easily prepared by mixing technical "ethanolamine" with the equivalent amount of fatty acids (solid acids are melted at 60°), the heat of reaction being sufficient to keep the reactants mobile during stirring. E. LEWKOWITSCH.

Deterioration of some silk-scouring soaps on storing. R. TSUNOKAE (J. Soc. Dyers & Col., 1928, 44, 377–379).—Some soaps on storing deteriorate and produce "sweat" or *ase* on their surface, the cause of which is shown to be the presence of unsaturated fatty soaps which suffer oxidation and change in colour, causing discoloration of the soap. There appears to be a definite relationship between the degree of hydrolysis of the soaps and the formation of *ase*; the smaller the degree of hydrolysis the greater is its formation. It has also been found to increase with the quantity of light petroleum extract (i.e., unsaponifiable matter).

L. G. LAWRIE.

Soap gels. P. A. THIESSEN (Kolloid-Z., 1928, 46, 350–355).—A description is given of the properties of soap gels, and experiments are described on the supercooling of solutions of sodium oleate. The number of crystallisation nuclei was counted and found to increase rapidly with the degree of supercooling. The number of nuclei also depends on the previous thermal history of the solution, and is less the higher is the temperature at which the solution has been heated. This indicates that the number of nuclei increases with increasing colloid content of the solution, the colloidal particles thus favouring spontaneous crystallisation.

E. S. HEDGES.

Evaluation of washing agents by surface-tension measurements. B. WALTHER (Z. angew. Chem., 1928, 41, 1083–1089).—The wetting capacity of solutions of a washing agent varies inversely with the surface tension, and measurements of the latter at the concentrations

used in practice give an indication of the cleansing efficiency of the washing agent. For comparison of different washing agents the fatty acid concentration of the solutions must be approximately the same, since solutions containing fatty acids (combined) have a minimum surface tension at a concentration of 0.1–0.2% of fatty acid. This concentration is lower than that used in practice, but the cleansing efficiency of a washing liquor is also determined by other factors, particularly foaming power and absorption capacity, which exert their greatest effect at higher concentrations. From measurements at 40° and 50° on solutions containing about 0.3% of fatty acid the following washing agents are arranged in order of decreasing surface tension: Thompson's soap powder, Persil, sodium oleate, Sunlight soap, and Lux, the difference between the first two mixtures and the pure soaps being very marked. At 20° and 30° the order is inverted, viz., Lux, Sunlight soap, sodium oleate; solutions of Lux have a marked decrease in surface tension from 20° to 40°. The surface tension of solutions of the sodium soaps obtained from rosin, olive oil, coconut oil, palm oil, palm-kernel oil, soya-bean oil, lard, beef tallow, and neatsfoot oil has also been measured over the range 20–50° and at concentrations of about 0.3% of fatty acid. Sodium resinate gives much the highest values at 50° and palm-oil soap the lowest, lard and beef tallow having very similar values at 50°, but much higher at lower temperatures. Coconut oil gives a soap having at 20° a high surface tension, but which falls rapidly to values at 30–50° which correspond with its well-known washing efficiency. All surface-tension measurements were made with a modified form of Traube's stalagmometer, which is described.

R. BRIGHTMAN.

Gel formation in fatty oils. J. SCHEIBER (Kolloid-Z., 1928, 46, 337–345).—A discussion of previously published work, mainly on the formation of gels by the drying of oils and the effects of heat, light, and various addition agents on the process.

E. S. HEDGES.

Extraction of olive oil by the Acapulco process. J. BONNET (Bull. Mat. Grasses, 1928, 219–233).—The results of a trial of the Acapulco extraction process at Marseilles in comparison with normal expression are recorded. A slightly higher yield was obtained of good-quality oil, with an acid value rather lower than that of the expressed oil; the pulpy residue, however, containing 7% of oil and 60% of water, cannot, in the opinion of the author, be utilised economically, whereas the grignons from expressed olives realise more than sufficient to pay labour and press-bag costs. A note in support of the Acapulco process by the inventor stresses the superior quality of the oil so obtained, and claims that a saleable by-product is obtained by sun-drying the extractor pulp. In trials performed by the Inspector of Agriculture at Sfax the yields were (% on total oil in the olives): by Acapulco 97.5, of one quality, acid value 0.27; by expression, 87.75, of three grades, acid values 0.50–0.57, and grignons containing 11.9%. The oil from the Acapulco process possessed an unpleasant metallic taste, probably due to the use of steel blades etc. in the pulping machine.

E. LEWKOWITSCH.

Olive oil analytical method. Use of ultra-violet ray in detection of refined in "virgin" olive oil. S. MUSER (Oil & Fat Ind., 1928, 5, 356—357).—The fluorescence under ultra-violet light of stale and deteriorated virgin oils is the same as that of fresh virgin oils and distinct in colour from that of renovated or refined oils; it is thus improbable that the blue fluorescence of the latter is a consequence of oxidation (cf. Marcille, B., 1928, 530). E. LEWKOWITSCH.

Hydrogenation of soya-bean oil. Application as lard substitutes. A. H. GILL and Y. M. MA (Oil & Fat Ind., 1928, 5, 348—351).—The rate of hydrogenation of soya-bean oil in the presence of a nickel catalyst is approximately proportional to the pressure, the amount of the catalyst (not more than 1% is necessary), and the speed of agitation; the rate also increases with increased temperature, although under high agitation (above 320 r.p.m.) a change of 10° between 150° and 180° had little or no effect. The course of the reactions resembles that of the hydrogenation of cotton-seed oil. E. LEWKOWITSCH.

Determination of the cold test of [animal or vegetable] oils. A. C. ORTHMANN and W. J. ARNER (J. Amer. Leather Chem. Assoc., 1928, 23, 595—599).—In a three-walled Dewar glass flask three fourths full of ether is placed a test tube, 23 cm. long and 1.5 cm. diam., containing the oil sample to a height of 3 cm. and fitted with a thermometer and stopper. The test tube passes through a stopper in the mouth of the flask, which is also provided with an air-inlet tube and an exit tube connected to an air pump. By drawing dry air through the ether the temperature is lowered, and the points are noted when the oil clouds ("cloud point") and when it will no longer flow. The test tube must be removed from the apparatus to determine this latter state. The "pour point" is 1° above the temperature at which movement of the oil ceases. Comparative tests made with this apparatus and that recommended by the American Society of Testing Materials, in which an air-jacket separates the test tube from the freezing mixture of ice and salt, show the former to be simpler, quicker, and more accurate. D. WOODROFFE.

See also A., Jan., 48, Oxidation of fats (PALIT and DHAR). Phenylstearic acid (DE MILT). 61, Colour reaction with cholesterol (NIKOLAEV and KRASTELEVSKAJA). Yeast ergosterol (REINDEL and WASENEGGER). 99, Hydrolysis of beeswax (PRUTTI and DE CONNO). Synthesis and hydrolysis of glycerides (VELLUZ). 103, Separation of vitamin fraction from cod-liver oil (MARCUS). Cod-liver oil (BAILEY, CANNON, and FISHER). Fluorescence of fats containing vitamin-A (MORGAN and MACLENNAN). 105, Oil content of flax seeds (DILLMAN). Composition of oil in relation to characters of plants (FACHINI and DORTA). 110, Determination of fat (ROSENFELD).

Determination of glycerol. RAVENNA. Analysis of glycerin. PRAGER.—See III. Errors in food analysis. REWALD.—See XIX.

PATENTS.

Production of fatty bodies soluble in water. R. VIDAL (B.P. 280,193 and Addn. B.P. 285,473, [A]

26.10.27, [B] 21.12.27. Fr., [A] 5.11.26, [B] 18.2.27).—(A) Non-alkaline water-soluble fatty substances may be obtained by treating fatty acids, animal fats, or oils with a large proportion of alkali hypochlorites without caustic alkalis as in B.P. 223,601 (B., 1925, 929). The resulting products are not soaps, but they have a surprisingly high detergent action, and mineral oils may be freely incorporated with them. (B) In the process by which vegetable fibres are bleached by means of alkali hypochlorites, Turkey-red oil, and castor oil soaps, the hypochlorites may be replaced by hypobromites and hypoiodites, and the simple fatty acids by sulphonated fatty acids and oils, more particularly by sulphoricinic and sulpho-oleic acids. [Stat. ref. to (A).] E. HOLMES.

Extraction and purification of oils in closed circuits. A. ACQUARONE (F.P. 623,474, 19.2.26).—Materials containing oils or fats are extracted with benzene first under increased then under reduced pressure at a suitable temperature in a closed container provided with a mechanical agitator. Finally the whole is heated *in vacuo*. A. R. POWELL.

Plastic masses from linseed and tung oils (G.P. 445,799).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Significance of "white [petroleum] oil" in the varnish industry. E. PYHÄLÄ (Farben-Ztg., 1928, 34, 783—784).—The methods of preparing and refining "white oil" (medicinal paraffin) and its various applications are described. It is suggested that it may be introduced in appreciable quantity into tung oil varnishes without unduly retarding the drying, and into hardened rosin, thus giving a plastic, "tack-free" material. Brief mention is also made of its possible use in the "running" of gums. S. S. WOOLF.

Noxious physiological effects of lacquer solvents. G. SIEBERT (Z. angew. Chem., 1929, 42, 17—19).—A discussion of the injurious physiological effects of various organic solvents employed in the lacquer industries. J. W. BAKER.

Rapid testing of varnish raw materials [resins]. H. BRENDL (Farben-Ztg., 1928, 34, 781—783).—Suggested methods of testing various varnish resins according to rapid laboratory practice are detailed. The materials discussed are rosin, copals, dammar, gum benzoin, acaroid, elemi, mastic, sandarac, shellac, and bitumens. The advisability of conducting comparative tests alongside a satisfactory sample is stressed. S. S. WOOLF.

[Constituents of] euphorbium resin. K. H. BAUER and P. SCHENKEL (Arch. Pharm., 1928, 266, 633—638).—Crude euphorbone may be obtained from the resin in two forms, consisting of needles, m.p. 67—68°, from light petroleum, and of amorphous, warty masses, m.p. 115°, from methyl alcohol, respectively. The crystalline form was converted into the amorphous variety on warming, with the loss of occluded light petroleum. Repeated recrystallisation alternately from methyl alcohol and acetone, afforded a *product*, m.p. 129°. The following constants of euphorbone were determined: α +15.56° in 4% chloroform solution; iodine value 91.34 (Hanus), 66.1—105.3 by Kaufmann's thio-

cyanogen method according to mode and time of reaction; hydrogenation value (Grün's method) 66.01—96.9. No isolable derivative was obtained on oxidation with nitric, chromic, or perbenzoic acid, potassium permanganate, or ozone. Fractional crystallisation from methyl alcohol furnished a small quantity of crystalline, optically inactive *euphorbol*, $C_{26}H_{46}O$, m.p. 122°, an unsaturated compound, which afforded *hydroeuphorbol*, m.p. 114° (*acetyl* derivative, m.p. 129°), when treated with hydrogen in the presence of palladised charcoal or barium sulphate. *Euphorbol* contains an alcoholic hydroxyl group. It did not give a positive cholesterol reaction, and it could not be converted into a digitonin. It is not identical with either *A*-, *C*-, or *R*-*euphorbone* described by Huppert, Swiatkowski, and Zellner (*A.*, 1928, 93).

S. COFFEY.

See also *A.*, Jan., 26, *Alumina gels* (BONNELL).

Plastic masses. SCHMIDT.—See V.

PATENTS.

[**Manufacture of**] **lithopone.** F. G. BREYER and C. W. FARBER, Assrs. to NEW JERSEY ZINC CO. (U.S.P. 1,690,099, 6.11.28. Appl., 29.12.26).—Zinc sulphate and chloride solutions are purified separately and barium sulphide solution is added at 50—60° until an excess equivalent to 0.5—1 c.c. of 0.1*N*-iodine solution is present in 25 c.c. of a filtrate. The crude lithopone pulp is at once diluted with water, washed free from chloride, and the hydroxide content adjusted so that 250 c.c. of filtrate liquor are equivalent to 2.5—7.0 c.c. of 0.25*N*-hydrochloric acid. The filtered pulp is dried (moisture content 5—8%) and calcined at 650—725°, the alkalinity being adjusted, if desired, as in U.S.P. 1,446,637 (*B.*, 1923, 366 *A*).

R. BRIGHTMAN.

[**Production of**] **nitrated carbohydrate solutions [cellulose nitrate lacquers].** HERCULES POWDER CO., and G. H. PETERS (B.P. 293,434—5, 20.10.27. U.S., 8.7.27).—(A) Non-blushing lacquers contain cellulose nitrate etc. together with other ingredients immiscible with water, *e.g.*, butyl acetate as solvent, and toluene as the volatile diluent. (B) Cellulose nitrate from which excess but not all the water has been removed is mixed with suitable ingredients immiscible with water to yield emulsions which are applied direct or after treatment in centrifugal apparatus and/or with hygroscopic salts to remove the water.

L. A. COLES.

Production of insulating varnish, which consists principally of rosin and tung oil. H. OHTA, and ASAHI GARASU KABUSHIKI KAISHA (B.P. 301,341, 17.8.27).—A mixture of rosin, tung oil, glycerin, and an alkali or alkaline-earth sulphide or selenide is heated at 240—300° in the presence of a catalyst, *e.g.*, metallic aluminium, and the product diluted with a solvent, such as turpentine oil.

S. S. WOOLF.

Manufacture of lustrous, plastic masses from linseed and tung oils. A. ROGLER (G.P. 445,799, 8.3.25).—A mixture of zinc oxide with linseed and tung oils is treated with sufficient benzoic or cinnamic acid to combine with all the zinc.

A. R. POWELL.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBENIND. A.-G. (B.P.

301,696, 6.11.28. Addn. to B.P. 261,029; *B.*, 1928, 532).—The products of higher mol. wt. obtainable from dimethylolurea by eliminating water by cautious treatment with an alkaline agent, dissolved or suspended in an organic solvent (preferably phenolic, in order to absorb disengaged formaldehyde), substantially in the absence of water, with the aid of acid condensing agents at raised temperatures, are employed in place of the dimethylolurea used in the process previously described.

S. S. WOOLF.

Obtaining condensation products of formaldehyde and urea. POLLOPAS, LTD., E. C. C. BALY, and E. J. BALY (B.P. 301,626, 11.11.27).—Vitreous masses are prepared by the condensation of pure dimethylolurea (m.p. at least 134°) by boiling for $\frac{1}{2}$ hr. in an aqueous weakly acid solution. Small quantities of formaldehyde may be added to prevent the formation of monomethylolurea. The condensation product may be obtained as a viscous syrup for moulding purposes or may be run into sulphuric acid and precipitated as a white flocculent mass which is subsequently washed, dried, and hot-pressed.

S. S. WOOLF.

Manufacture of linoleum covering material. A. EISENSTEIN, Assr. to G. SCHICHT A.-G. (U.S.P. 1,693,917, 4.12.28. Appl., 13.3.25. Czechoslov., 16.1.25).—The liquid and solid constituents are mixed in a proportion in which they are pulverulent, the mixture is oxidised at above 70° but below the temperature of carbonisation, and the product is maintained at this temperature and in the pulverulent condition.

M. E. NOTTAGE.

Grinding mills (B.P. 301,348 and 301,781).—See I. **Synthetic resin** (B.P. 280,246).—See XVII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Evaluation of the quality of fillers in rubber manufacture. M. PAVLENKO and P. NAZAROV (*J. Chem. Ind. Moscow*, 1927, 4, 642—650).—A consideration of the relation between the viscosity of a colloidal system consisting of rubber, solvent, and filler, the plasticity of the system when devoid of solvent, and its elasticity and strength after vulcanisation. Variation of the filler alone imparts to the system a characteristic viscosity. By measurement of the time required for the fall of a glass ball in the viscous mass, relative viscosities of the system rubber-xylene-carbon black were measured for different carbon fillers. The viscosity of the rubber solution without filler did not appreciably change with time; characteristic curves were obtained showing the relative viscosity as a function of time for each carbon black filler. An initial rise (followed by a fall due to disintegration of the rubber) is due to the fact that a uniform mixture is not attained at once. Fillers producing the highest relative (maximum) viscosity gave the strongest vulcanisates. The better was the sample of filler, the longer the rubber mixture remained flocculent. Similar results were obtained with kaolin and chalk fillers.

CHEMICAL ABSTRACTS.

See also *A.*, Jan., 71, **Osmotic experiments with caoutchouc solutions** (KROEFELIN and BRUMSHAGEN). 72, **Effect of silent discharge on caoutchouc** (FROMANDI).

PATENTS.

Utilisation of [rubber] latex and product thereof.

W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,690,150, 6.11.28. Appl., 25.11.27).—Latex is thickened by adding about 5% of hæmoglobin in 30% solution and 2% of zinc oxide, and stirring until the latter react. Formaldehyde may be added to increase the water-resistivity and tensile strength.

R. BRIGHTMAN.

Drying of mixings of latex and filling materials.

K.D.P., LTD. (B.P. 277,373, 12.9.27. Ger., 10.9.26).—By continuously exposing new surfaces during the heating operation, mixtures of latex and filling materials are dried, without coagulation, to a plastic mass capable of being worked and shaped in the usual manner.

D. F. TWISS.

Production of differently coloured marbled, veined, or streaky sponge rubber.

GUMMI U. BALATAWERKE MATADOR A.-G., and F. GIRG (B.P. 302,102, 2.4.28).—Differently coloured primary materials containing the necessary colours, gas-forming agents, and softeners, and of different consistency, are drawn into thin sheets, then superposed, formed into a compact roll, which is then twisted, and again rolled into strips. These strips are placed side by side to form a plate, and are then vulcanised in a mould. The sharpness of definition between the various colours may be increased by subjecting the sheets before superposition to superficial vulcanisation.

D. F. TWISS.

Production of shaped rubber objects. K.D.P., LTD., Assees. of E. A. HAUSER (B.P. 277,376, 12.9.27. Ger., 10.9.26).—Latex, particularly concentrated or containing stabilising agents, and compounded if desired, is sprayed on to a heated surface, *e.g.*, on to a hot, rotating core, for the production of a tube. As an alternative to heating the surface, spraying may be effected with hot air. Filling materials which are difficultly miscible with latex, or are liable to cause premature coagulation, *e.g.*, lamp black, can be sprayed independently, the jets being so directed that the materials mix prior to impinging on the surface.

D. F. TWISS.

Deposition of organic materials [rubber etc.] from aqueous dispersions containing these materials and articles manufactured therefrom.

DUNLOP RUBBER CO., LTD., R. F. MCKAY, and W. H. CHAPMAN (B.P. 301,367, 25.7.27).—An anodic, creamy or pasty deposit is produced on a partially-immersed rotatable drum by electrophoresis of an aqueous dispersion of rubber or similar material, compounded or otherwise, and is continuously transferred at the exposed portion of the drum to a continuous surface, such as of fabric. In this way rubbered fabric may be produced or, if the continuous surface is smooth, the rubber sheet may be subsequently separated by stripping.

D. F. TWISS.

Vulcanisation of rubber articles. GOODYEAR TIRE & RUBBER CO., Assees. of R. W. SNYDER (B.P. 288,248, 21.2.28. U.S., 7.4.27).—Vulcanisation is effected by carrying the articles, hanging on a conveyor, down a column of heated liquid until a desired hydrostatic pressure is attained; they then pass along an intermediate horizontal layer of liquid under the same pressure,

and finally are removed by way of a second vertical column of the liquid. The rate of movement is such that the desired degree of vulcanisation is attained during immersion.

D. F. TWISS.

XV.—LEATHER; GLUE.

Chrome tanning compounds. E. STIASNY (Collegium, 1928, 554—567).—The following factors should be studied: basicity value, degree of basicity of the chromium salt, precipitation value, p_H value, degree of "verolung," astringency, and the proportion of sulphate groups in the chromium complex. The p_H value of freshly-prepared basic chrome liquors gradually diminishes, owing to the formation of "ol" compounds, *e.g.*, $\text{Cr}-\text{OH}-\text{Cr}$, whereby the molecule contains two or more chromium atoms. "Verolung" leads to an increased size of the colloidal particles. Highly-colloidal compounds do not tan, neither do crystalloid chromium salts, hence the best tanning action is possessed by semi-colloidal basic chromium salts in which there are partially "verolte" chromium complexes containing several chromium atoms in the complex, and in which the hydroxy-groups possess some free residual valencies. Reversal of the "verolung" process is affected by the acid radicals present in the following order of diminishing effect: $\text{C}_2\text{O}_4 > \text{HCO}_2 > \text{SO}_4 > \text{Cl} > \text{NO}_3$. The acid radicals in basic chromium chloride liquors are all ionisable. Sulphate groups migrate to the chromium complex during the ageing, heating, addition of sodium sulphate to a solution of hexa-aquochromium sulphate, or if it is rendered basic. Chlorine is expelled from the chromium complex when a solution of chromium chloride is rendered basic. The migration of sulphate groups into the chromium complex diminishes the hydrolysis, and hence the acidity of chrome liquors, and facilitates "verolung," thus causing molecular condensation and increase in the semi-colloidal properties; this explains the better tanning properties of chromium sulphate liquors. No oxalate groups are ionisable amongst the chromium oxalates, because of their strong affinity for the chromium atom, which is only equalled or exceeded by hydroxo-groups. Mono-oxalato-salts are rendered valuable tanning agents by adding alkali. The anionic chromium complexes have a precipitation value ∞ ; addition of alkali increases their basicity, diminishes their astringency, increases the negative charge, and removes the complex from chromium hydroxide. The composition of the chromium complexes in chrome leather differs from that of those used in tanning. Different products are obtained by rendering chrome liquors basic with sodium hydroxide and carbonate, respectively, due to the formation of carbonato-chromium complexes in the latter case. The following method is used in determining the basicity of liquors containing carbonato-complexes. The liquor is oxidised with sodium hydroxide and hydrogen peroxide, treated with barium chloride, filtered, and excess alkali is titrated in the filtrate.

D. WOODROFFE.

Extraction of solid tanning materials by the Koch and Procter apparatus under identical condition. J. G. PARKER (J. Amer. Leather Chem. Assoc., 1928, 23, 590—595, and J. Soc. Leather Trades'

Chem., 1928, 12, 564—568).—Various tanning materials were extracted in the Koch and Procter forms of apparatus, collecting 2 litres in 5 hrs. and 1 litre in 3 hrs., respectively. The total extracted matter was slightly higher with the Procter extractor and when 2 litres were collected. The Koch apparatus is easier to manipulate but soon chokes, thus necessitating a fresh extraction. The percolate was colourless, and practically all tanning materials were extracted after 4 hrs. No evidence of hydrolysis of the tannin was obtained by extractions of sumac, algarobilla, and myrobalans with 2 litres of water during 5 hrs. as compared with the old official method using 1 litre of water for 3 hrs. For complete extraction of solid tanning materials, therefore, use of the Procter apparatus with 2 litres of water during 4 hrs. is recommended.

D. WOODROFFE.

Darmstadt apparatus for tannin analysis. J. G. PARKER (J. Soc. Leather Trades' Chem., 1928, 12, 520—524).—The lower end of a glass cylinder, 15 cm. long and 5.5 cm. diam., is fitted with a Jena sintered-glass filter plate and screwed into a metal cap in which it rests on a thin sheet of rubber. The upper end can be similarly fitted or may be closed with a rubber bung. The cylinder, without bung and cap, is weighed roughly, the cap screwed on the base, the necessary dry hide powder and distilled water are added, and the bung is fitted. The whole is shaken several times and allowed to remain for 1 hr. Chrome alum solution is then added and, after shaking in a machine, the mixture is left overnight. The bung and cap are now removed, the cylinder is placed in a special glass funnel with a rubber ring, and the whole inserted in a filter flask connected to a vacuum pump. The powder is washed with distilled water until free from sulphates, then partially dried. The cylinder and contents are again weighed and a few drops of distilled water added to produce the correct amount of moisture. The hide powder is broken up with a glass rod, the metal cap replaced, 100 c.c. of the tannin solution are added, the top is closed with the bung, and the apparatus shaken for 10 min. The cap is removed, the cylinder placed in the special funnel, and the detannised liquor is drawn off into a clean dry filter flask.

D. WOODROFFE.

Measurement of the colour of tanning extracts. A. DE LA BRUÈRE (J. Soc. Leather Trades' Chem., 1928, 12, 485—489).—Measurements of the colour of tanning extracts by the Lovibond tintometer were shown to vary with different operators and the concentration of the solution used, and concordant results were not obtained by calculating to a basis of 0.5% tannin. Spectrophotometric methods of measuring the colour are too delicate. An apparatus comprising a photo-electric cell is suggested to obviate the personal factor. Light is passed through a colour filter, then through the tanning extract solution on to the cell. The deflection of a galvanometer in the circuit is noted and expressed as a percentage of that registered when the tannin solution is replaced by distilled water. Similar results are obtained for the other colours of the spectrum, and they are plotted as a curve. Differences in the violet as well as in the red and yellow are made apparent by this method.

D. WOODROFFE.

Analysis of artificial bating materials [for leather]. V. KUBELKA (Collegium, 1928, 604—611).—The Schneider-Ulcek method (B., 1927, 662, 758) of determining the enzyme activity of an artificial bate depends on the activity of the erepsin, which hydrolyses the tryptic hydrolysis products into free amino-acids. The Kubelka-Wagner method (B., 1927, 260) is free from such objection.

D. WOODROFFE.

Salt stains. I. Hide damage. E. STATHER (Collegium, 1928, 567—599).—Five kinds of "salt stains" have been identified. (a) So-called "Salzstippen"—small pimples on the grain caused by the crystallisation of the salt, which may or may not be accompanied by bacterial damage. (b) Blue, lilac, or reddish stains on the flesh side hitherto attributed to the action of chromogenic bacteria or to bacterial decomposition of adhering fat. (c) Brick-red to carmine-red stains on the flesh side due to heating, and often accompanied by hair slipping and grain damage. (d) Lemon-yellow to brownish-orange stains (4—8 mm. diam.), more prevalent on the flesh side and characterised by attack on the hide tissue. These are true salt stains. (e) A second form of true salt stain (bright yellow to orange, 3—5 mm. diam.) is distinguished by the association of a number of stains to form a band.

D. WOODROFFE.

Action of common salt on [raw] hide. M. BERGMANN (Collegium, 1928, 599—604).—The porosity from the grain side of pieces of raw calf-skin was determined using water, then a salt solution, and finally water again. It was less with the salt solution than with water. The diminution was the greater, the greater the concentration and amount of salt solution used. The porosity with water after the salt treatment was less than the initial water-porosity for treatment with salt solutions containing more than 0.06%. A 0.03% salt solution caused a 57% increase in the porosity to water, which indicated an irreversible effect on the hide substance. A 2.3% salt solution showed practically no penetration of raw pelt.

D. WOODROFFE.

Hydrolysis of leather. G. ARBUSOV (Vestnik, 1927, 337—342).—Leather is a system not in equilibrium. During hydrolysis re-arrangement of uncombined or loosely combined tannin takes place, resulting in equilibrium between the solid and liquid phases and the formation of a chemical compound. On treatment of leather with methyl alcohol, replacement of tannin by alcohol may occur. Previous treatment of leather with alcohol does not change the weight relationships between hide substance and tannin existing in the hydrolysis products. Upper leathers gave hydrolytic products of constant composition independent of the duration and method of hydrolysis, but different from those of sole leather.

CHEMICAL ABSTRACTS.

Water-absorption and penetration tests for sole leather. L. M. WHITMORE and G. V. DOWNING (J. Amer. Leather Chem. Assoc., 1928, 23, 603—606).—A piece of sole leather of standard size is cut, weighed, immersed in water at 20° for 2 hrs., drained, weighed, again immersed in the water for 22 hrs., drained, and again weighed. The first gain in weight is expressed as a percentage of the total gain in weight for the

24 hrs.' period of immersion, which is assumed to give complete saturation of the leather with water. The percentage obtained is the "rate of saturation" for 2 hrs. Concordant results varying from 55% to 100% have been obtained, and the method gives a good indication of the quality of the leather.

D. WOODROFFE.

Wear-resistance of sole leather. U. J. THUAA (J. Soc. Leather Trades' Chem., 1928, 12, 505—520).—In the tests reported by the U.S. Bureau of Standards (J., 1923, 1142A) and Woodroffe (B., 1926, 989) the effect of humidity was ignored. In Sweden tests are made on both dry and damp leather. A standard method of determining the wear-resistance should be evolved and employed by the three Associations of Leather Trades' Chemists. Various forms of machine for determining this factor are reviewed, and the following is suggested for use. It consists of a vertical disc, 30 cm. diam., and revolving at 30 r.p.m., on which are mounted six samples of the leather. The circumference of the disc makes contact with a carborundum paper mounted on a horizontal revolving disc. The vertical disc is mounted on a weighted lever so that at the point of contact of the leather samples with the abrasive there is a force equal to the weight of 70 kg. An automatic brush and blower remove the powdered leather from the abrasive. The relative coefficient of wear-resistance is the surface of contact necessary to reduce the thickness by 1 mm./cm.² The test can be performed dry with "silex" or emery as abrasive, or damp with carborundum kept moist.

D. WOODROFFE.

See also A., Jan., 86, **Detection of gallic acid and tannin (CELSI)**. 95, **Nutritive properties of gelatin (JACKSON, SOMMER, and ROSE)**.

Condensation of badan extract. GODNEV.—See IV. **Plastic masses.** SCHMIDT.—See V. **Effluents from leather works.** SNOEK.—See XXIII.

PATENTS.

Providing a finish to leather, hides, skins, etc. J. R. C. JORGENSEN (B.P. 301,554, 6.7.27).—The leather etc., the surface of which may have been degreased and/or abraded, is stretched, coated with a highly viscous, highly concentrated, or easily solidifying solution of an organic or inorganic cellulose ester or ether, the surplus solution is scraped off, and the film dried. Two or more coats of cellulose derivative of different composition may be applied to produce different effects, or filling materials, pigments, dyes, colours, or metal powders may be added to the coating solution. Suitable apparatus is described.

D. WOODROFFE.

Vegetable glue and its production. I. F. LAUCKS and G. DAVIDSON, Assrs. to I. F. LAUCKS, INC. (U.S.P. 1,689,732, 30.10.28. Appl., 29.10.23).—A seed flour having a high protein content, e.g., soya-bean or linseed flour, is mixed with aqueous caustic alkali, which may also contain an alkaline-earth hydroxide. Copper salts may be added to increase the resistance to water, and tanning agents, alkali silicates, and rosin may be present.

F. G. CLARKE.

Manufacture of vegetable glue. I. F. LAUCKS

and G. DAVIDSON, Assrs. to I. F. LAUCKS, INC. (U.S.P. 1,691,661, 13.11.28. Appl., 9.3.27).—See B.P. 298,511; B., 1928, 905.

XVI.—AGRICULTURE.

Reclamation of the Fresno-type black alkali soil.

W. P. KELLY and E. E. THOMAS (Calif. Agr. Exp. Sta. Bull., 1928, No. 455, 1—37).—The crop-producing power of Fresno black alkali soil, the unproductivity of which is due to an excess of soluble salts, especially sodium carbonate, and the abnormal composition of the clay-like constituents, is improved, although at different rates, by the use of gypsum, sulphur, ferric sulphate, or alum, which act on the soluble carbonate and clay constituents simultaneously. Gypsum acts by virtue of its soluble calcium, whilst the other substances, being acidic, decompose soluble carbonate, whereby soil calcium, especially carbonate, is dissolved and thereupon reacts with the clay constituents. Sulphur is the most economical, but is slow in action since its effect follows its oxidation by bacterial agency. Special bacterial inoculation is not advantageous. For permanent reclamation the soil must be well drained.

CHEMICAL ABSTRACTS.

Hydrogen peroxide-hydrochloric acid treatment of soils as a method of dispersion in mechanical analysis.

G. B. BODMAN (Soil Sci., 1928, 26, 459—470).—A comparison of the international method of soil treatment for mechanical analysis with the older process of rubbing up with very dilute ammonia is recorded. In highly organic and in calcareous soils higher values for the finer fractions were obtained by the acid-peroxide method. In a hard-pan soil the ammonia treatment produced higher proportions of clay. In other cases differences were small. The treatment of calcareous soils with acid drastically alters the nature of the ions associated with the clay complex and therefore with its physical properties. The practical value of such mechanical analyses is questioned. The colloidal contents of soils as determined by pipette-sedimentation methods were greater than when Robinson's water-absorption process was adopted. In the latter instance very fine grinding of the soil had little effect on the experimental values obtained.

A. G. POLLARD.

Graphical representation of the buffering power of soils.

W. U. BEHRENS (Forts. Landw., 1928, 3, 299; Bied. Zentr., 1928, 57, 534—535).—Polemical. The author holds that buffering power is best characterised by a series of titration measurements plotted with p_H values for abscissæ and "buffering," dS/dp_H , for ordinates. Limitations of single- and double-value determinations are pointed out.

H. L. RICHARDSON.

Effect of high concentration of organic or ammoniacal nitrogen on nitrification in soil.

N. V. JOSHI (Agric. J. India, 1928, 23, 473—481).—Nitrate production from oil cake and ammonium sulphate in soil increased with the amounts added, up to a maximum beyond which further addition of nitrogen resulted in the accumulation of ammonia and sometimes of nitrites which slowly disappeared after some weeks. Leaching of soil after the completion of a period of nitrification increased its capacity for

nitrification. The nitrifying power of a soil is considered to be mainly influenced by the actual number of organisms present. Nitrite or ammoniacal nitrogen added to soil in excess of the maximum amount which the soil can nitrify disappears, the decomposition of the nitrite being the more rapid. A. G. POLLARD.

Exchangeable cations in soils as determined by means of normal ammonium chloride and electro dialysis. B. D. WILSON (Soil Sci., 1928, 26, 407—422).—There is considerable variation in the total amounts of exchangeable bases in different soils, but there is a general parallelism between this value and soil fertility. The ammonium chloride method of Kelley and Brown indicates larger amounts of exchangeable bases than Bradfield's electro dialysis process, the differences being slight in the case of calcium but very considerable for potassium and magnesium. With heavier currents the electro dialysis method gives values approaching those of the ammonium chloride process. Leaching of dialysed and washed soils with ammonium chloride extracted relatively large amounts of magnesium, potassium, iron, and aluminium, but little calcium. The total content of cations in diffusates from electro dialysed soils is conveniently determined by direct titration, this giving more satisfactory results than determinations of the adsorbed ammonium ions. After electro dialysis and washing, the p_H values of the soils investigated approximated to 4.0. It is suggested that normal ammonium chloride solution may extract from soil cations other than those of the exchangeable bases, and that this is attributable to the partial decomposition of the clay complex by hydrochloric acid produced by the adsorption of ammonium ions by the soil.

A. G. POLLARD.

Retention of phosphate by hydrated alumina, and its bearing on phosphate in the soil solution. L. B. MILLER (Soil Sci., 1928, 26, 435—439).—Electrometric studies of the precipitation of aluminium phosphates from mixed solutions of aluminium chloride and potassium phosphate with soda are recorded. The capacity of the precipitate to retain phosphate is greatest over the range p_H 3.0—4.5 where the precipitate is composed of $AlPO_4$. Addition of alkali beyond the point equivalent to p_H 4.5 results in a gradual decomposition of the precipitate and the dissolution of the phosphate. At p_H 7.5 the formation of soluble aluminates begins and proceeds simultaneously with the dissolution of phosphate, the latter action being complete at p_H 8.5. The possibility that similar reactions occur in the soil is discussed.

A. G. POLLARD.

Ratio of sulphur to phosphorus in Western Oregon soils and losses of sulphur through drainage and cropping. J. S. JONES (Soil Sci., 1928, 26, 447—453).—The importance of the maintenance of adequate supplies of sulphur in soils is emphasised. Losses of sulphur from soil in drainage water are much heavier than is the case with phosphate. The average amount of sulphur removed by crops is one third that of phosphorus, and in many cases fertilising with sulphur may prove more beneficial than the use of phosphates.

A. G. POLLARD.

Relationship of sulphur content of soils and

plants. F. HENGL and P. RECKENDORFER (Forts. Landw., 1928, 3, 598; Bied. Zentr., 1928, 57, 536—537).—In considering smoke injury it is essential to know how far the sulphur content of a plant may be affected by that of the soil, especially when sulphate has been added in fertilisers. Crop plants were manured with fertilisers containing chloride and sulphate; at the time of flowering, the respective chlorine or sulphate content of plants so treated was considerably above that of the controls, but when the plants were ripe the differences were less, sulphate content in particular being little affected. In field observations no relationship was found, except in extreme cases, between sulphate content of soils and plants; further, natural variations in the sulphur content of plants were greater than increases due to smoke gases. H. L. RICHARDSON.

Effect of sulphur on soils and on crop yields. R. R. McKIBBIN (Md. Agric. Exp. Sta. Bull., 1928, No. 296, 65—114).—The amount of water-soluble sulphur in a soil is not a measure of its total sulphur content. Soils low in total sulphur readily oxidise sulphur to sulphate. Sulphur behaves as an amendment rather than as a fertiliser, by bringing the reaction of the soil solution to neutrality. The amount of water-soluble phosphorus in a soil is governed by the reaction, and not by the total phosphorus content. Sulphur oxidation generally tends to make the phosphorus less soluble. The effect of sulphur mixtures on crop yields of Maryland soils is discussed. Sulphur should not be used in acid phosphate mixtures, but it generally increases the value of raw rock phosphate.

CHEMICAL ABSTRACTS.

Hydrometer method for studying soils. B. A. KEEN (Soil Sci., 1928, 26, 261—263).—Recent work of Bouyoucos (B., 1927, 422, 454, 498) is criticised. It is maintained that the hydrometer method as described is unsuitable for the mechanical analysis of soils, giving results which are essentially qualitative.

A. G. POLLARD.

Microscopic method of studying bacteria in soil. H. J. CONN (Soil Sci., 1928, 26, 257—259).—The author's method for examining soil organisms by direct staining of a soil film is modified. Improved staining results by limiting the solubility of the dye in water. In the case of rose-bengal, the calcium salt is less soluble than the sodium compound and the dye solution is prepared with the addition of 0.01—0.03% of calcium chloride. Higher concentrations of calcium chloride should be avoided or the staining is too intensive and portions of soil organic matter become stained and obscure the organisms. The preliminary acid treatment of the soil film is now omitted.

A. G. POLLARD.

Precipitation of calcium oxalate in the presence of iron, aluminium, titanium, manganese, magnesium, and phosphates, with special reference to the determination of total soil calcium. H. D. CHAPMAN (Soil Sci., 1928, 26, 479—486).—Calcium oxalate is precipitated in solutions at p_H 4.0. To the soil solution or extract is added ammonium chloride to ensure the presence of at least 6 g. before precipitation, together with 1 g. of oxalic acid in solution, 10 c.c. of 1.76*N*-acetic acid, and 10 drops of 0.04% bromocresol-green solution. The total volume of solution is

adjusted to 150–200 c.c., brought nearly to boiling, and dilute ammonia added until the colour changes from yellow through yellowish-green to pure green. After boiling for 5–10 min. the calcium oxalate is precipitated and allowed to settle on the steam-bath. After 3 hrs. the calcium may be determined by titration with permanganate. A. G. POLLARD.

Crop response to lime on acid soils. R. E. STEPHENSON (Soil Sci., 1928, 26, 423–434).—The general problem of soil fertility, as influenced by liming, is discussed. It is considered that the supply of the ions of the essential nutrients is a greater factor in the fertility of acid soils than the degree of acidity or the concentration of toxic ions. A. G. POLLARD.

Relation of lime to the absorption of iron by plants. W. P. ALLYN (Proc. Indiana Acad. Sci., 1927, 37, 405–409).—The excessive use of calcium carbonate on soils does not render iron non-available for absorption by the maize plant. Deposition of iron at the nodes following heavy liming does not necessarily indicate greater total absorption of iron. The amount deposited is decreased by the application of potash or manure. Chlorosis induced by lime results from a disturbance in the metabolism of iron after absorption by the plant.

CHEMICAL ABSTRACTS.

Reactions of seedlings to weak concentrations of hydrochloric acid and calcium. J. R. SKEEN (Soil Sci., 1928, 26, 471–478).—Sand-culture experiments with various seedlings indicated that the toxicity of acid soils may not be entirely the result of the toxicity of the hydrogen ion, but may in part be the outcome of a deficiency of other necessary ions, notably calcium. The need for calcium varies very considerably with different species of plants. A. G. POLLARD.

Potash and nitrogen manuring of potatoes. H. WIESSMANN and E. SCHRAMM (Forts. Landw., 1928, 3, 625; Bied. Zentr., 1928, 57, 539–541).—From eleven field experiments in different districts it appeared that (except on one soil which did not respond to nitrogen) the total yield of starch was little increased by potash manuring unless nitrogen was also added. Further nitrogen caused larger increases in starch yield than did further potash. Starch content, as distinct from yield, was highest without potash or nitrogen, and lowest with the heaviest applications. Potash (40% salts) depressed starch content, whether or not nitrogen was applied, and the depressing effect of potash was greater than that of nitrogen. H. L. RICHARDSON.

Determination of sugar and dry matter in root crops. A. ZADE (Forts. Landw., 1928, 3, 207; Bied. Zentr., 1928, 57, 550–551).—Roots are taken at random from each plot to make 100 or 200 per treatment, and sampled in the field by a special contrivance. The mixed sample is preserved with toluene in a flask that may be sealed with paraffin, and at any convenient time up to 14 days this material may be pulverised and analysed. According to the values given, the greatest change in dry matter is 0.4%, and in sugar 0.6%, after a fortnight. The method has been used for three years, and is recommended for groups of experiments. H. L. RICHARDSON.

Effect of different proportions of calcium nitrate and potassium dihydrogen phosphate on the growth of wheat in sand cultures. D. D. BROWN (Soil Sci., 1928, 26, 441–446).—In sand cultures of wheat high proportions of calcium nitrate in the nutrient solution induced heavy tillering and succulent growth, the number of tillers increasing with the amount of nitrogen used. Relatively large proportions of potassium phosphate produced large root development, smaller aerial growth, poor tillering, early maturing, and low transpiration of water. Intermediate but unbalanced proportions of nitrate and phosphate in the nutrient caused in many cases chlorosis and malformation of the plants. Analysis of the plants showed that calcium, potassium, phosphate, and sulphate are absorbed in amounts proportional to their concentrations in the nutrient solution. The iron and aluminium contents of the plant ash were practically constant in all cases. High concentrations of potassium in the nutrient tended to reduce the absorption of magnesium by the plant, and in some cases high concentrations of calcium appeared to limit the absorption of nitrate. Total ash content was highest in the chlorotic plants grown in unbalanced solutions and lowest in the high-nitrate cultures. A. G. POLLARD.

Evaluation of the root-soluble soil nutrients phosphoric acid and potassium. GERLACH (Z. Pflanz. Düng., 1928, 7B, 579–584).—Differences in the limiting values of root-soluble nutrients as expressed by Neubauer and by Roemer are discussed. In field trials far fewer soils exhibited deficiencies of potassium or phosphate than when examined by the Neubauer and Roemer methods. The different experimental conditions of the three methods of soil examination are largely responsible for the divergent results obtained. A. G. POLLARD.

Comparison of Rhenania phosphate with superphosphate and basic slag [as fertilisers]. DENSCH (Forts. Landw., 1928, 3, 356; Bied. Zentr., 1928, 57, 544–546).—In a year's pot and field experiments with several acid soils, superphosphate tended to give the largest increases in yield; there was nothing to choose between Rhenania phosphate and basic slag. The former is accordingly classed as a "high-value" fertiliser. H. L. RICHARDSON.

Nitrogen manuring. F. MÜNTER (Z. Pflanz. Düng., 1928, 7B, 553–579).—Results of numerous field trials extending over a number of years are utilised to elucidate the most advantageous use of nitrogenous fertilisers for various soils, crops, and climatic conditions. The importance of the soil reaction in this connexion is emphasised. A. G. POLLARD.

Petroleum oil as a carrier for nicotine [in insecticides]. E. R. DE ONG (J. Econ. Entomol., 1928, 21, 502–504).—Oil-nicotine mixtures are more effective than is the oil alone as an insecticide for brown apricot scale; kerosene-nicotine mixtures are nearly as effective as heavier oil-nicotine mixtures, are cheaper, and less detrimental to plant tissue.

CHEMICAL ABSTRACTS.

See also A., Jan., 49, **Plant colouring matters: crocetin and lycopin** (KARRER, HELFENSTEIN, and

WIDMER). 105, Daily growth and oil content of flax seeds (DILLMAN). Evolution of carbon dioxide and absorption of oxygen in germinating seeds (FRIETINGER). Decomposition of hexoses in plants (ZALESKI and SCHATALOWA-ZALESKAJA). 106, Chemistry of latex-bearing plants (ZELLNER). 107, Influence of oxygen and carbon dioxide on growth of *Ophiobolus graminis* (FELLOWS).

Sewage sludge as fertiliser. RUDOLFS.—See XXIII.

PATENTS.

Manufacture of insecticides. H. W. AMBRUSTER (U.S.P. 1,691,454, 13.11.28. Appl., 4.12.23).—An ore containing an arsenic, copper, or lead compound, e.g., scorodite, is finely ground so that it is absorbed by insects, particularly the boll weevil. The ore may be ground wet with tannin or other dispersing agent, and the suspension sprayed upon the plants. When soluble ingredients which would harm the plants are present, they are removed by washing, or converted into insoluble compounds. F. G. CLARKE.

Insecticide and fungicide. M. LACROIX and H. BREYER (B.P. 300,439, 13.2.28).—A mixture of powdered naphthalene (4 kg.), flowers of sulphur (4 kg.), and barium sulphate (5 kg.) has added to it successively with stirring tar oil (2.5 kg.) and copper nitrate (2 kg.), sodium silicate (1.5 kg.), formic acid (1.5 kg.), and potassium silicate (2.5 kg.), and the product obtained by boiling 2 kg. of pine sawdust with 15 litres of water until the volume has been reduced to 10 litres, sieving, and adding 67 litres of milk of lime, or, if a solid product is required, up to 75 kg. of slaked lime. L. A. COLES.

Waste from agave plants (B.P. 301,284).—See XVIII.

XVII.—SUGARS; STARCHES; GUMS.

Determination of sucrose and of starch syrup. J. D. FILIPPO (Chem. Weekblad, 1928, 25, 676—679).—The method is based on the destruction of practically the whole of the laevulose without loss of any considerable proportion of the dextrose by boiling with hydrochloric acid; the solution is decolorised by means of charcoal and the dextrose determined polarimetrically. This result, in combination with a separate iodometric determination of the laevulose, gives the sugars from both sources. S. I. LEVY.

See also A., Jan., 49, Sugars (MIŠIĆ). 50, Oxidation of carbohydrates (KÜCHLIN and BÖESEKEN). 85, Volumetric determination of reducing sugars (BLAZZO). 91, Polarimetric determination of small amounts of dextrose (LUNDGAARD and others). 99, Enzymic condensation of formaldehyde to sugar (BODNAR). Hydrolysis of carbohydrates (PIUTTI and DE CONNO). 101, Fermentation of maltose by *Bacterium pullorum* (EDWARDS). Aërobic oxidation of dextrose (COOK and STEPHENSON).

Products from sorbitol (B.P. 301,655).—See III. Acetone-butyl alcohol fermentation of molasses. MEZZADROLI and MAGNO.—See XVIII.

PATENTS.

Purification of sugar. E. C. R. MARKS. From CORN PRODUCTS REFINING Co. (B.P. 302,375, 15.8.27).—Colloidal and metallic compounds present as impurities in starch-converted dextrose solutions are removed by subjecting the solution, in the cathode compartment of a two-compartment electrolytic cell, to the action of an electric current whereby the colloids and metallic hydroxides are precipitated and subsequently removed by filtration. Two types of cell are described in which the sugar solution is added (a) to the cathode compartment and water to the anode compartment, precipitation occurring only in the former, and (b) to both compartments, the diaphragm being perforated to allow the passage of colloidal particles. In this, precipitation occurs chiefly in the cathode compartment, but also to some extent in the anode compartment. The anode solution may be used for acidifying starch for conversion. W. J. BOYD.

Syrup or molasses from beet. E. GIRON (F.P. 623,595 and 624,219, [A] 22.10.26, [B] 8.11.26).—(A) Beet slices are treated with dilute acid and subsequently heated under pressure in an autoclave. (B) The beet is first treated under pressure to extract the sugar juices, and the residue is boiled with a dilute mineral acid in a copper autoclave. The whole process is repeated as often as necessary, and the united extracts are neutralised, filtered, and concentrated to $d\ 1.4$. A. R. POWELL.

Treatment of corn syrup and corn-sugar liquors. C. H. CHRISTMAN, Assr. to NAT. ALUMINATE CORP. (U.S.P. 1,692,817, 27.11.28. Appl., 21.2.27).—Solutions of hydrolysed carbohydrates are purified by neutralisation with sodium aluminate. W. J. BOYD.

Synthetic gum or resin. CANADIAN ELECTRO PRODUCTS Co., LTD., H. W. MATHESON, and F. W. SKIRROW (B.P. 280,246, 8.11.27. U.S., 8.11.26).—Aliphatic vinyl esters are treated with above 1% of saturated aliphatic aldehydes, preferably with heat and/or under pressure, with or without the addition of catalysts (hydrogen chloride). B. FULLMAN.

Butyric acid by fermentation (F.P. 620,363).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Darkening of worts during the preparation of pale beers. H. STADLER (Woch. Brau., 1928, 45, 579—582).—The composition of the brewing liquor is of prime importance, and with this is associated the quality of the malt, especially as regards husk, and the colour of the hops. The colour of worts taken at various stages in the brewing process was compared by evaporating or diluting to a definite concentration, boiling with hops, and fermenting. The colours of the resulting beers can be determined more accurately than those of the worts. During sparging the colour and p_H increase as the amount of solids decreases; thus in one case the colours of the wort containing 3.1, 0.8, and 0.4% of solids were 7, 10, and 15, respectively, on Brand's scale when brought to a concentration of 12% of solids.

The total wort collected showed an increase of colour from about 2.5 when first drawn off to 8 when all was collected. The increase of colour in transferring from mash tun to copper barely exceeds the limit of experimental error, and that during boiling is slight. Open and closed, copper and iron, steam- or fire-heated vessels give similar results. Some increase of colour occurs during cooling, but is associated with insoluble, finely-divided protein, and is removed during fermentation. Old and discoloured hops occasion an undesirable increase and reddening of the colour, and by extracting hops with waters of different temporary hardness it was found that this extraction of colour also increases with decreasing hydrogen-ion concentration. This would take place during the hop sparge. The virtue of gypsum in brewing liquor is explained by its action in raising the hydrogen-ion concentration, thereby inhibiting the extraction of reddish colouring matter from the hops. Sulphured hops may decrease colour in the copper, but their use is not advisable (cf. Wanderscheck, B., 1928, 871).

F. E. DAY.

Silicic acid in beer. H. NETSCHER (Woch. Brau., 1928, 45, 582—585).—Brewing materials all contain appreciable quantities of silicic acid. The author finds quantities similar to those reported by other investigators, his figures for brewery and laboratory worts being 0.0174 and 0.0046 g./100 c.c. (as SiO_2), respectively. For the latter he finds 0.0074 g./100 c.c. after hopping and 0.0070 after fermenting. The silicic acid is partly precipitated with the insoluble matter at various stages during the brewing process, as much as 1.18% being found in the deposit on the coolers. To test its possible association with haze in pasteurised beers, five beers were examined. They contained 0.0122—0.0205 g. of silica per 100 c.c., and in general it was found that those with a high proportion gave more deposit and developed haze more rapidly than those with less silicic acid. Mashings were made under various conditions with distilled water and with supply water containing about 0.001 g. of silica per 100 c.c. Analytical data of the beers from these are given, and on pasteurisation it was noted that the deposit and turbidity were greatest in those containing most silicic acid and less broken-down protein. A high proportion of silicic acid improved the brightness of the filtered beer. It is concluded that silicic acid is an appreciable factor in the colloid systems concerned in the production of haze and deposits in beer.

F. E. DAY.

Rapid refractometric analysis of still wines and fortified still wines. R. SAAR (Z. Unters. Lebensm., 1928, 56, 144—158).—Using the formulæ $A = 0.3595 \times (R + N) - 4.21$, and $E = 0.1247(R - N) - 3.41$, the values of A , the alcohol content in vol.-%, and E , the extract content in g./100 c.c., can be calculated from the refraction R determined by immersion refractometer at 17.5° and the value $N = 1000 - 1000s$, s , the sp. gr., being found by hydrometer. In this way alcohol contents of 25—45 vol.-% can be determined, and the extract content can be found when the alcohol content lies between 30 and 41 vol.-%. The method is not applicable to liquors rich in sugar and certain still wines with high content of essential oil. Adulteration with

methyl alcohol is detectable by the negative value found for E .

W. J. BOYD.

Determination of glycerol in wine. C. DE COQUET (Bull. Soc. Pharm. Bordeaux, 1928, 66, 69—78; Chem. Zentr., 1928, ii, 607).—The usual methods have been examined and certain recommendations made.

A. A. ELDRIDGE.

Occurrence, detection, and determination of lauric acid in alcoholic liquors. J. GROSSFELD and A. MIERMEISTER (Z. Unters. Lebensm., 1928, 56, 167—187).—The chief fatty acid constituent of fusel oil from wine is not decanoic acid (cf. Windisch, Arb. Kaiserl. Gesundheitsamte, 1893, 8, 139—228, 257—293), but lauric acid present as the ethyl ester. The lauric acid content of fusel oils depends on their previous treatment, and can be removed by rectification. In the detection of lauric acid in alcoholic liquors, the ether extract from 50 c.c. of the liquor is saponified with alcoholic potash, the alcohol removed, the residue dissolved in 3 c.c. of water and mixed with 0.2 c.c. of glycerin, and 0.25 c.c. of magnesium sulphate solution (150 g./litre) added to the hot solution. The solution is filtered hot and allowed to cool, whereupon magnesium laurate gradually separates as a flocculent precipitate; 0.2 mg. of lauric acid can be detected in this way. A method of determining lauric acid by precipitation as magnesium laurate is described.

W. J. BOYD.

Acetone-butyl [alcohol] fermentation and its application to molasses. G. MEZZADROLI and G. MAGNO (Giorn. Chim. Ind. Appl., 1928, 10, 551—554).—Experiments are described which show that the butyl alcohol fermentation of sucrose requires as nearly as possible anaerobic conditions. The most suitable concentration of sugar in the wort is 5—6%, but 10% may be employed if the wort is subjected to prolonged boiling prior to the fermentation. The inversion of the sucrose proceeds, *pari passu*, with its conversion into acetone and butyl alcohol. Cane molasses wort, being poorer in nutrient substances than that obtained from beet molasses, requires addition of ammonium phosphate, or peptone, or both. The total yields of solvent products vary from 27 to 31% by wt. of the invert sugar, the best results being obtained with worts seeded with spores of the micro-organism. The p_H value of the wort is about 6 before and 5.4 after the fermentation. Cereal starch yields 33% of the fermentation products.

T. H. POPE.

Viscous fermentation of lemonade. R. GUYOT (Compt. rend. Soc. Biol., 1928, 97, 857—859; Chem. Zentr., 1928, ii, 822).—A *Torula* was isolated which aerobically fermented dextrose, levulose, sucrose, starch, and gum, but not mannitol or inulin. In anaerobic conditions, inversion took place; lactose and dextrose were attacked, but not mannitol or inulin. The product is not cellulose or gum.

A. A. ELDRIDGE.

See also A., Jan., 48, **Determination of lactic acid** (LEHNARTZ). 61, **Yeast ergosterol** (REINDEL and WASENEGGER). 99, **Enzymic condensation of formaldehyde to sugar** (BODNÁR). **Lipases** (GYOROKU and TERASHIMA). **Ricinus lipase: hydrolysis of esters and carbohydrates** (PIETTI and DE CONNO). **Hydroly.**

sis of glycerides (VELLIZ). Action of phosphatases (NEUBERG and JACOBSON). Pyrophosphatase (KAY). 100, Metaphosphatase (KITASATO). Proteinase and polypeptidase of yeast (GRASSMANN and DYCKERHOFF). 101, Inactivation of invertase and raffinase by heat (NELSON and PAPADAKIS). Aërobic and anaërobic metabolism of cultivated yeasts (WINDISCH). Decomposition of cellulose by aerobic bacteria (DUBOS). Fermentation of maltose by *Bacterium pullorum* (EDWARDS). Aërobic oxidation of dextrose (COOK and STEPHENSON). Production of gelatinase in *B. proteus* (MERRILL and CLARK). 108, Alcoholic fermentation by *Aspergillus flavus* (YUILL). Action of asparaginase from *Aspergillus niger* (BACH). 110, Colorimetric determination of lactic acid (MENDEL).

Fluorescence of wine in ultra-violet rays. MIGLIACCI.—See XIX.

PATENTS.

Manufacture of butyric acid by fermentation. SOC. LEFRANC & CIE. (F.P. 620,363, 3.4.26).—Molasses or other by-product containing sucrose is diluted to obtain a 7% sucrose solution, which is then fermented for 70–80 hrs. at 44–45° with frequent additions of milk of lime to remove resinous products and proteins. The clarified solution is evaporated to recover calcium butyrate, which is then decomposed with sulphuric acid and the butyric acid purified by distillation. From the mother-liquor calcium acetate may be recovered after further evaporation.

A. R. POWELL.

Treatment of waste accruing from removal of fibres from the agave and like plants. H. R. FOUQUE (B.P. 301,284, 30.5.28).—The material is repeatedly disintegrated, washed, and pressed, and the resulting juice is sterilised and at the same time freed from chlorophyll and albuminous matter by heating it for, e.g., 20 min. at 120° in a tinned copper or aluminium vessel, and passing the cooled liquor through a sterilised filter press. The clear wort is then fermented at 30–32° in closed iron tanks by Moreau yeast (from Algerian agave plants). A fermentable wort may also be obtained from the finely-disintegrated stalks if these are first heated under pressure with dilute ($\frac{1}{2}$ %) sulphuric acid. Antiseptics, such as fluorides, which do not affect Moreau yeast, may be added to the worts if desired. The solid residue from these operations is used in suitable gas producers, the ash from which is used as a fertiliser.

D. J. NORMAN.

XIX.—FOODS.

Chemical composition of certain food pastes and the modifications they undergo when boiled in water. L. SETTIMI (Atti R. Accad. Lincei, 1928, [vi], 8, 314–317).—Results of analyses of various qualities of materials of the macaroni type are given. After being boiled in water, these substances exhibit physical modifications resulting from the imbibition of water, and contain about 20% of soluble starch, 6% of reducing sugars calculated as dextrose, and appreciably less soluble nitrogen compounds than the original material. The fat present is not modified by the boiling.

T. H. POPE.

Detection of rice flour in pepper powder. M. WAGENAAR (Z. Unters. Lebensm., 1928, 56, 205–208).—The process for determination of rice flour in other flours by means of fuchsin solution (B., 1928, 384) is not applicable to the examination of pepper powder, but a solution of carmine in ammonia and glycerin can be used satisfactorily.

W. J. BOYD.

Composition of Californian creams. N. C. SMITH (Calif. Dep. Agric. Mo. Bull., 1927, 16, 728–736).—The percentage of calcium oxide in the ash best detects neutralisation by lime; acidity, iodine, Polenske, Reichert-Meißl, and Valenta values were of little value.

CHEMICAL ABSTRACTS.

Cause of the fluorescence of milk and of wine in ultra-violet rays. D. MIGLIACCI (Boll. Chim. farm., 1928, 67, 673–674).—A summary of work on this subject.

T. H. POPE.

Detection of cacao embryo in cocoa products. W. SCHMANDT (Z. Unters. Lebensm., 1928, 56, 198–199).—Technique for the microscopical detection of fragments of embryo in cocoa is described.

W. J. BOYD.

Effect of sugar, acid, and "set" on the keeping properties of jams. F. HIRST (Food Manuf., 1928, 3, 447–450).—All three factors are concerned. In a well-set jam, containing, e.g., only 60% of sugar, yeasts cannot penetrate. To prevent slight fermentation if wild yeasts gain access, or to prevent growth of *Penicillium glaucum*, 65% of sugar is necessary; such jams, however, are too sweet, and tend to crystallise when stored. Acid has a slight effect on the growth of yeast, but not (at 0.5–1%) on that of *Penicillium*.

CHEMICAL ABSTRACTS.

Volumetric method of determining tin in conserves and other foodstuffs. B. GLASSMANN and S. BARSUTZKAJA (Z. Unters. Lebensm., 1928, 56, 208–212).—The sample (50 g.) is incinerated, and the ash extracted with nitric acid to remove iron, copper, and lead. The residue is reduced to metallic tin by fusion with potassium cyanide for 20 min. at a dull red heat. The tin is washed with water to remove potassium cyanide and cyanate, and then dissolved in hydrochloric acid in a flask fitted with a Bunsen valve; 1 g. of pure zinc is added whereby metallic tin is re-formed and redissolved after the zinc has disappeared. The solution is cooled in a stream of carbon dioxide, and the stannous chloride titrated with 0.02N-potassium dichromate solution in the presence of potassium iodide and starch as indicator. The potassium dichromate solution is standardised against pure tin. The total time required for the determination is 5½ hrs.

W. J. BOYD.

Determination of hydroxymethylfurfuraldehyde in honey. J. FIEBE (Z. Unters. Lebensm., 1928, 56, 200–203).—The volumetric method of Troje (B., 1925, 1004) by which he indicated the presence of hydroxymethylfurfuraldehyde in genuine honey gives erroneous results. Owing to the presence in honey of other substances which are soluble in ethyl acetate and react with alkaline iodine solution, or which influence the solubility of sugars in ethyl acetate, the iodine equivalent of the extract does not represent the true hydroxy-

methylfurfuraldehyde content. Genuine honey which does not give a positive Fische test and therefore is free from hydroxymethylfurfuraldehyde may show a considerable quantity by Troje's method. W. J. BOYD.

A double source of error [in food analysis]. B. REWALD (Chem.-Ztg., 1928, 52, 1013).—In the determination of fat in seeds, feeding-stuffs, etc. by the ordinary methods of extraction with ether or benzene, a small proportion of "residual fat" remains unextracted, which consists principally of lipoids; these constitute less than 1% of vegetable substances, but may amount to 3—4% in those of animal origin. Since the lipoids contain nitrogen their presence also leads to an error in the proportion of protein if calculated from the total nitrogen. For exact determinations of fat and protein the lipoids are determined or removed by treating the extracted material several times with 96% alcohol, and then with a mixture of alcohol and benzene, evaporating, and weighing; if sugars are present the dry extract is again extracted with absolute ether or benzene. F. R. ENNOS.

See also A., Jan., 86, **Detection of colouring matter** (HOFMAN). 91, **Composition of human milk** (BELL). **Effect of heat on milk** (DAUM). 95, **Nutritive properties of gelatin** (JACKSON, SOMMER, and ROSE). 103, **Vitamin-A in maize** (HAUGE and TROST). 105, **Sodium and potassium content of lentils and peas** (KÜSTER and UMBRECHT). 106, **Nutritional chemistry of the banana and water-melon** (KONDO and others). 107, **Proteins of buckwheat flour** (HARA). **Proteins of Italian millet** (KONDO). **Purine bases of seed and meal of soya bean** (DUCCESCHI).

Lard substitutes. GILL and MA.—See XII. **Fermentation of lemonade.** GUYOT.—See XVIII.

PATENTS.

[Dried] milk products. J. GOLDING, and DECO, LTD. (B.P. 302,439, 10.10.27).—Milk and whey are fed simultaneously but separately on to a heated drying or evaporating surface of the drying-roll type, so that they are mixed thereon and obtained as a yellowish-white powder, or as an unbroken sheet, which is readily ground. The product may be used with advantage in the making of bread or as infants' food etc.

W. J. BOYD.

Preservation of cheese. **Preservation of dairy products.** E. E. ELDERIDGE, ASST. to KRAFT-PHENIX CHEESE CO. (U.S.P. 1,693,025—6, 27.11.28. Appl., [A] 10.2.27, [B] 13.6.27. Renewed [B] 18.5.28).—The cheese is comminuted, mixed with a small amount of (A) water-soluble pectin, or (B) gum tragacanth, at not above 52°, subdivided, and introduced into closed shipping receptacles and heated in these at rest to 66°.

W. J. BOYD.

Apparatus for pasteurising milk. J. O. TEMPLETON, ASST. to ELECTROPURE CORP. (U.S.P. 1,692,874, 27.11.28. Appl., 6.1.26).—The milk is purified by passage between electrodes in an apparatus which automatically ceases to operate when the temperature of the milk falls below a certain point. The fluid entering the apparatus passes through a coil over which the treated fluid is discharged.

W. J. BOYD.

Manufacture of a flour improver and products treated therewith. N. V. NOURY & VAN DER LANDE'S HANDELSMAATSCHAPPIJ, and J. A. L. VAN DER LANDE (B.P. 300,568, 12.5.27. Cf. B.P. 300,515; B., 1929, 71).—A wheaten cereal is heated at 35—90° in the presence of 0.05—0.005% of acid until only 95—30% of the gluten is retainable in a washing test. Non-volatile acids may be used, but it is preferable to use a volatile acid (e.g., hydrochloric acid). As the presence of 15—20% of moisture is advantageous, and the exclusion of oxygen often of importance, a closed vessel may be used. The improver is added to flour in the proportion of 0.5—2.0%, whereby the strength of the flour is increased, but the improver does not yield a satisfactory bread by itself. W. J. BOYD.

Production of fodder and nutritive products. J. E. NYROP (B.P. 301,651, 14.12.27).—Soya beans, maize, or earth nuts are ground with water, skimmed milk, etc., and an oil containing vitamin-A is emulsified in the resulting aqueous suspension. This emulsion, after thorough mixing, is atomised into a hot, inactive gas, e.g., carbon dioxide or nitrogen, at a sufficiently low temperature that a dry product is obtained which may reversibly form cream- or milk-like products with water, skimmed milk, etc. W. J. BOYD.

Production of artificial coffee aroma. H. STAUDINGER and T. REICHSTEIN, ASSTS. to INTERNAT. NAHRUNGS- & GENUSSMITTEL A.-G. (U.S.P. 1,696,419, 25.12.28. Appl., 15.10.26. Ger., 4.11.25).—See B.P. 260,960; B., 1928, 347.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Assay of mercury oxycyanide and mercury cyanide pastilles. E. SCHULEK and A. STASIAK (Arch. Pharm., 1928, 266, 638—641).—Hydrocyanic acid may be determined by the bromine-iodometric method, previously described (A., 1925, ii, 327, 606), provided that sodium chloride or potassium bromide is first of all added to the solution. The solution containing mercuric cyanide is treated with phosphoric acid and bromine water, followed by sodium chloride or potassium bromide. Excess of bromine is subsequently removed with phenol and the cyanogen bromide is finally determined by titrating with 0.001N-sodium thiosulphate solution the iodine set free from potassium iodide solution. The method described by Rupp and Lewy (A., 1928, 860) gives low results.

S. COFFEY.

Stability of morphine in aqueous solution especially during sterilisation. R. DIETZEL and W. HUSS (Arch. Pharm., 1928, 266, 641—667).—Aqueous solutions of morphine hydrochloride decompose on heating, and the change may be followed spectroscopically since morphine shows distinct absorption bands in the wave-length region 3000—4400 Å. As the time of heating is increased the absorption band is gradually shifted towards the visible region and becomes less well defined. The stability of aqueous solutions of morphine depends to a marked extent on the hydrogen-ion concentration. Alkaline solutions ($p_H > 7$) undergo a change at room temperatures; approximately neutral

solutions are relatively unstable at higher temperatures, whilst acid solutions ($p_H < 7$) are more stable. No decomposition is observed when solutions ($p_H \leq 5.5$) are heated at 100° for 1 hr. or longer. The change is due to the oxidation of morphine to ψ -morphine, and the two components may be determined in a solution by comparing the absorption curve with those obtained with known mixtures.

S. COFFEY.

Rapid detection of extract of *Atractylis gummifera* in liquorice extract. P. CONDORELLI (Atti II Cong. Naz. Chim. pura appl., 1926, 1353—1355; Chem. Zentr., 1928, ii, 592).—The extract (5 g.) is treated with boiling water (50 c.c.) containing a little ammonia, cooled to 20° , and acidified with dilute sulphuric or hydrochloric acid, whereby it is considered that a glucoside, atractylic acid, is precipitated. The precipitate, when heated with 2—3 drops of concentrated sulphuric acid, gives an odour of valeric acid (Angelico's reaction); it may also be oxidised by Bertolo's method.

A. A. ELDRIDGE.

Detection of industrial spirit in pharmaceutical tinctures. W. MEYER (Pharm. Ztg., 1928, 73, 1600—1602).—Tinctures suspected to contain inferior spirit may be prepared in part from alcohol distilled from industrial spirit originally rendered non-potable by addition of pyridine to fully rectified alcohol. Distillation by means of hot water at 90° of spirit rendered non-potable in this way yields a product containing no methyl alcohol or acetone, and only traces of pyridine. A microchemical method of testing for such traces of pyridine by means of its double compound with cadmium chloride is described.

S. I. LEVY.

Commercial evaluation of cloves. W. A. N. MARKWELL and L. J. WALKER (Perf. Ess. Oil Rec., 1928, 19, 496—497).—Five existing methods for the assay of cloves to be used for oil distillation are reviewed, and the following new method is described. About 0.5—1 g. of the cloves reduced to a No. 20 powder is weighed into a tared Petri dish with lid. It is heated at 110° until of constant weight, and from the difference in weights is subtracted the amount of moisture determined by Dean and Stark's method. The percentage of volatile substances other than water is thus obtained. Results agreeing well with yields given by commercial distillations are obtained.

E. H. SHARPLES.

Essential oil of *Trilobum Sup. Siler*. B. RUTOVSKI and K. GUSSEVA (Riechstoffind., 1927, 230; Chem. Zentr., 1928, i, 267).—The oil, obtained in a yield of 3.27%, had d_{20}^{20} 0.8886, n_D^{20} +131.65°, n_D^{20} 1.4862, acid value 1.05, ester value 41.58, ester value after acetylation 59.3, and contained α -limonene (about 55%; tetrabromide, m.p. 103°) and α -perillaldehyde (about 40%; semicarbazone, m.p. 197° ; oxime, m.p. 102°). The presence of α -pinene and azulene was conjectured.

E. H. SHARPLES.

Determination of ionone. R. D. HENDRIKSZ and A. RECLAIRE (Perf. Ess. Oil Rec., 1928, 19, 493).—The following modification of the method of Reclaire and Spoelstra for the determination of citronellal (B., 1928, 426) is applied to ionone determination. 5 c.c. of ionone

are refluxed for 2 hrs. with a solution obtained by dissolving 15 g. of hydroxylamine hydrochloride in 37.5 g. of water, and adding to this solution 18 g. of potash in 37.5 g. of water. The mixture, still hot, is poured into a separating funnel, the aqueous layer separated, and the oximated oil washed three times with hot brine and filtered as hot as possible. In about 0.5—1 g. of the dry oil the nitrogen is determined by the Kjeldahl-Gunning method. The ionone content is calculated from the formula $x = 53.82a/(14 - 0.042a)$, in which a = c.c. 0.2*N*-sulphuric acid required for 1 g. of oximated oil.

E. H. SHARPLES.

Sesamin and sesamolin. W. ADRIANI (Z. Unters. Lebensm., 1928, 56, 187—194).—Sesamin, found in sesamé oil to the extent of 1%, was crystallised repeatedly from alcohol to a constant m.p., 122.5° . The pure material did not give the Baudouin reaction with furfuraldehyde and hydrochloric acid. It had $[\alpha] +68.23^\circ$ in chloroform. Elementary analysis and mol. wt. determinations indicated the formula $C_{20}H_{18}O_6$. Sesamolin found in sesamé oil to the extent of 0.3% had m.p. 93.6° and $[\alpha] +218.4^\circ$ in chloroform. The formula $C_{20}H_{18}O_7$ found by Malagnini and Armanni (Chem. Ztg., 1907, 31, 884) was confirmed. It is hydrolysed by concentrated hydrochloric acid according to the equation $C_{20}H_{18}O_7 + H_2O = C_7H_6O_3$ (sesamol) + $C_{13}H_{14}O_5$. Sesamol, the phenolic substance to which the Baudouin reaction of sesamolin is due, was found to have m.p. 65.5° instead of 57° as found by Malagnini and Armanni (*loc. cit.*). The name *samin* is suggested for the substance $C_{13}H_{14}O_5$ now isolated for the first time. It formed colourless crystals, m.p. 103° $[\alpha] +103^\circ$ in chloroform, and did not give the Baudouin reaction.

W. J. BOYD.

See also A., Jan., 49, Sodium and potassium tetrabismuth tartrates (KÖBER). 72, Nopinene ozonide (BRUS and PEYRESBLAUQUES). 74, Pyridine and quinoline derivatives (RÄTH and PRANGE). 76, Histidine (CHEMNITZ). 79, Microchemical reactions of homatropine (WAGENAAR). Absorption spectra of ergot alkaloids (HARMSMA). 81, Morphine alkaloids: thebaizone and products of ozonolysis (WIELAND and SMALL). 82, *Strychnos* alkaloids (LEUCHS, BENDER, and WEGENER). *Helleborus* group: new alkaloids from *H. viridis* (KELLER and SCHÜBEL). Unsymmetrical arseno-compounds (PALMER and KESTER). 83, Pyridine-3-arsinic acid (BINZ, RÄTH, and GANTE). Derivatives of 1:4-benzisooxazine (NEWBERRY, PHILLIPS, and STICKINGS). 84, Derivatives of 4-amino-3-hydroxyphenylarsinic acid (BALABAN). Organo-selenium compounds (TAKAMATSU). 86, Determination of colouring matter (HOFMAN). 86, Reactions of colchicine (EKKERT). 96, Cicatrising agents (JUSTIN-MUELLER). 97, Assays of Chinese ephedrine (FENG and READ). 101, Purification of diphtheria toxin. (ABT). 102, Extraction of hormone of corpora lutea (GLEY). Testing of ovarian preparations (KOCHMANN). Preparation of oestrus-producing hormone (ALLAN and others). Regulation of production of insulin (GRAFE and MEYTHALER). 103, Separation of

vitamin fraction from cod-liver oil (MARCUS). Determination of vitamin-A (NELSON and JONES). 104, Conversion of vitamin-B into automatin by radiation (ZWAARDEMAKER). Purified ergosterol and its esters (BILLS and HONEYWELL). Vitasterol-D (JENDRASSIK and KEMÉNYFFI). 106, Glucosides of digitalis leaves (WINDAUS). Glucosides of *Adonis vernalis* (FROMHERZ). *Oenanthe sarmentosa* (GOODRICH and LYNN). Extract from *Sphacele parviflora* (HASENFRATZ). 110, Extraction of cholesterol and its esters from tissues and body fluids (MÜLLER). Micro-determination of cholesterol (HORIYE).

Euphorbium resin. BAUER and SCHENKEL.—See XIII.

PATENTS.

Extraction of theobromine. A. BOEHRINGER (B.P. 302,207, 12.9.27).—Natural products containing theobromine (cacao, cacao waste, cacao husks) are treated with dilute alkaline solutions (e.g., excess of alkaline-earth hydroxides), at not above 50–60°, in a countercurrent diffusion battery (or, especially in the case of finely-ground materials, a countercurrent decantation or classification battery). E.g., using milk of lime, 90–95% of the theobromine is recovered on treating the extract with hydrochloric acid; or the material is treated with a limited quantity of alkaline-earth hydroxide, and the residue extracted with 2–5% soda lye, the extract in the latter solvent being then treated with alkaline-earth hydroxide, the impurities separated, and the liquid acidified. The materials may receive an initial treatment with water. Materials which have been soaked before extraction are lixiviated with water at varied temperature and pressure. B. FULLMAN.

Extraction of theobromine from natural products. N. V. SOC. CHEM. IND. "KATWIJK" (B.P. 287,507, 21.3.28. Holl., 22.3.27).—The ground material is intimately mixed with a large excess of magnesia or alkaline-earth hydroxide and water is added in such amount that the mixture remains solid. Either immediately or after 24 hrs., further water is stirred in at, e.g., 70° to give an extract of the desired concentration, which is filtered and decomposed in known manner.

R. BRIGHTMAN.

Removal of nicotine from tobacco. H. FEDERMANN (B.P. 302,560, 17.7.28).—Tobacco, in a slowly-rotating perforated metal drum, is treated in closed cycle for some hours with the same quantity of gaseous ammonia heated at a low temperature (42°) and at a pressure only slightly above atmospheric. After passing through the tobacco the ammonia is freed from nicotine by passage through trichloroethylene or carbon tetrachloride and recirculated. Water vapour is supplied to maintain the natural moisture of the tobacco. After the ammonia treatment the tobacco is treated with carbon dioxide to remove the ammonia. B. FULLMAN.

Manufacture of aminoalkyl ethers of oximes and their salts. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,956, 9.9.27).—Water-soluble products, suitable for subcutaneous injection, are obtained by heating oximes with β -diethylaminoethyl chloride and alcoholic

sodium ethoxide. The β -diethylaminoethyl ethers of oximes of cyclohexanone, b.p. 135°/15 mm. (hydrochloride, m.p. 95°), camphor, b.p. 150–160°/14 mm. (hydrochloride, m.p. 172°), and anisaldehyde (hydrochloride, m.p. 145°) are described. C. HOLLINS.

Manufacture of basic ethers of resorcinol. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,695, 18.8.27).—Basic ethers of resorcinol which have a strong contracting effect on blood-vessels and on the muscles of the uterus are obtained by treating a saturated monoalkyl resorcinol ether with an aminoalkyl halide in an alkaline medium. Monomethyl resorcinol ether and β -diethylaminoethyl chloride in presence of sodium ethoxide afford *m*-methoxyphenyl diethylaminoethyl ether, b.p. 160–166°/13 mm. (hydrochloride, m.p. 138–140°). *m*-Methoxyphenyl β -dimethylaminomethylbutyl ether, b.p. 170–172°/12 mm., and *m*-ethoxyphenyl β -diethylaminoethyl ether, b.p. 171–179°/12 mm., are similarly obtained. R. BRIGHTMAN.

Manufacture of arylaminoalkylcarbinols [β -amino- α -arylethyl alcohols]. W., K., L., W., and F. MERCK (E. MERCK) (B.P. 280,574, 10.11.27. Ger., 10.11.26).—Phenacylamine salts are hydrogenated in presence of a nickel catalyst. Phenacylmethylamine hydrobromide, when shaken with hydrogen gas in presence of 10% of nickel catalyst precipitated on pumice or asbestos, affords β -methylamino- α -phenylethyl alcohol, m.p. 77°. β -Amino- α -phenylethyl alcohol (hydrochloride, m.p. 40°) and β -methylamino- α -phenylpropyl alcohol (hydrochloride, m.p. 189–5°) are similarly obtained. R. BRIGHTMAN.

Manufacture of 2-hydroxypyridine-5-carboxylic acid. C. RÄTH (G.P. 447,303, 12.11.24).—2-Chloro-5-cyanopyridine is heated at 100° under pressure with 20% alcoholic sodium hydroxide solution, or at 150° with hydrochloric acid (*d* 1.19). The reaction product is diluted and, in the first case, acidified. 2-Hydroxypyridine-5-carboxylic acid is precipitated, and on recrystallisation from methyl alcohol has m.p. 302–303°.

A. R. POWELL.

Manufacture of 8-hydroxyquinoline and derivatives thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,545, 2.9.27).—8-Hydroxyquinolines are obtained in practically quantitative yield by heating 8-aminoquinolines with mineral acid or zinc chloride in aqueous or aqueous alcoholic solution at 180–190° at 5–6 atm. 8-Hydroxyquinoline and 6:8-dihydroxyquinoline, m.p. 153°, b.p. 207°/16 mm. (from 8-amino-6-hydroxyquinoline, m.p. 177°, or 8-amino-6-methoxyquinoline), are described. C. HOLLINS.

Manufacture of ethers of 6:8-dihydroxyquinoline. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,947, 1.9.27).—6:8-Dihydroxyquinoline is converted by alkylating agents into mono-ethers, the 6-alkoxyl derivative predominating, or, with excess of the reagent, into di-ethers. The same products are obtained by Skraup's reaction from 4-aminoresorcinol ethers. A second alkyl group may be introduced into the mono-ethers. The following quinolines are described: 6:8-dimethoxy- (m.p. 56°, b.p. 132–134°/1 mm.; anti-

epilepsin), 8-hydroxy-6-methoxy- (m.p. 125°; anti-pyretic), 6:8-diethoxy- (m.p. 60°, b.p. 153°/2 mm.; intestinal remedy), 8-hydroxy-6-ethoxy- (m.p. 125°), 6-methoxy-8-β-diethylaminoethoxy- (b.p. 193°/4 mm.), 6-ethoxy-8-β-diethylaminoethoxy- (b.p. 190°/1 mm.; from 4-β-diethylaminoethoxy-o-phenetidine, b.p. 166–168°/1 mm.). C. HOLLINS.

Preparation of cinchophen [2-phenylcinchoninic acid]. R. PASTERNAK, Assr. to C. PFIZER & Co. (U.S.P. 1,690,128, 6.11.28. Appl., 21.4.27).—Equimolecular quantities of 30% aqueous pyruvic acid, aniline, and benzaldehyde are charged into boiling 95% alcohol at about 100°. Side reactions yielding diketopyrrolidine anil are reduced. R. BRIGHTMAN.

Manufacture of arsenobenzenes. A. ALBERT (B.P. 300,716, 26.8.27).—Aromatic arsenic compounds containing ter- or quinque-valent arsenic, or mixtures of the two, are reduced to arsenobenzenes with hypophosphorous acid or a hypophosphite in presence of less than 0.33 mol. of sulphurous acid or a sulphite and of hydriodic acid or an iodide if required. With more than 1 mol. of sulphurous acid colourless arsenobenzenes which contain sulphur probably attached to arsenic are obtained. Thus, in presence of 0.025 mol. of sulphurous acid, the semicarbazone of 2-carboxymethoxy-4-aldehydophenylarsinic acid affords the corresponding arsenobenzene. With 1 mol. of the same semicarbazone and 1 mol. of the semicarbazone of 4-aldehyde-2-hydroxyphenylarsinic acid, the disemicarbazone of 2-hydroxy-2'-carboxymethoxy-4:4'-dialdehydoarsenobenzene, decomp. above 200°, is obtained. The disemicarbazones, decomp. 240° and 230°, respectively, of 2:2'-dihydroxy-4:4'-dialdehydoarsenobenzene and of 2:2'-dihydroxy-4:4'-diacetoxyarsenobenzene are similarly obtained. In presence of 1–2 mols. of sulphur dioxide in acetic acid at 80–85° the semicarbazone of 4-aldehydo-2-hydroxyphenylarsinic acid yields a substance, decomp. at 225°; similar products, decomp. at 220° and at 230°, are obtained in alcoholic hydrochloric acid in presence of hydriodic acid and by boiling under a reflux in presence of sulphurous acid. With 1 mol. of sulphurous acid in alcoholic hydrochloric acid in presence of hydriodic acid, 3-amino-4-hydroxyphenylarsinic acid affords a product, decomp. at 230°. R. BRIGHTMAN.

Synthesis of aromatic arsenic compounds containing iodine. A. D. MACALLUM (B.P. 300,286 and 300,538, 4.5.27).—(A) 5-Iodo-3-nitro-4-hydroxyphenylarsinic acid on reduction with titanous or ferrous oxide in cold alkaline solution affords 63–66% of the zinc or lead salt of 5-iodo-3-amino-4-hydroxyphenylarsinic acid. darkens at 95°, decomp. in a vacuum at 65°, the N-acetyl derivative of which, m.p. 158–159° or 190–191° (from 50% acetic acid), is converted by reduction with sodium hyposulphite at 55–60° into 5:5'-diacetamido-4:4'-dihydroxyarsenobenzene, m.p. 194°. Treatment of the latter in ether suspension with iodine and hydrolysis of the di-iodide with alkali hydrogen carbonate gives 5-iodo-3-acetamido-4-hydroxyphenylarsenoxide, m.p. 182–183°. (B) 5-Iodo-3-nitro-4-hydroxyphenylarsinic acid is converted (yield 78–79%) by hypophosphorous

acid in methyl alcohol at 55–60° into 5:5'-di-iodo-3:3'-dinitro-4:4'-dihydroxyarsenobenzene, which when suspended in ether and treated successively with iodine and alkaline hydrogen carbonate solution affords 5-iodo-3-nitro-4-hydroxyphenylarsenoxide, m.p. 170–210°.

R. BRIGHTMAN.

Extracts of the internal secretory organs of females. SOC. CHEM. IND. IN BASLE (B.P. 285,856, 16.2.28. Switz., 23.2.27).—The fresh organs are mixed with an indifferent freezing agent (solid carbon dioxide), if necessary with addition of a dehydrating agent (sodium sulphate), and the product is powdered and extracted with solvents at ordinary or raised temperature.

B. FULLMAN.

Separation of the cardio-active glucoside of *Bulbus scillæ* into two components. R. E. ELLIS. From CHEM. FABR. VORM. SANDOZ (B.P. 300,726, 1.9.27).—The cardio-active glucoside of *Bulbus scillæ* obtained, e.g., by the processes of B.P. 199,400 and 217,247 (B., 1924, 692, 997) is separated in aqueous methyl alcohol by fractional dissolution or precipitation including salting out, or by fractional extraction with an organic solvent, e.g., ethyl acetate, into two components A and B. The less soluble component (A) has α_D^{20} –78°, crystallises from methyl alcohol with 6.5% of solvent of crystallisation (lost *in vacuo* at 76°), has a physiological activity per mg. of 1050 according to the frog standard of Houghton and Straub, and is readily hydrolysed to aglucone-A, which sublimes *in vacuo* at 180–200°, losing 1H₂O. Component B is non-crystalline, dextrorotatory, slowly hydrolysed to crystalline aglucone-B, m.p. 228–229° (decomp.), and has a physiological activity per mg. of about 1500–1600 frog doses. The separation may equally be effected on the glucoside-tannin compounds with relatively crude material; in such cases a treatment with a tannin-precipitating agent is given prior to isolation.

R. BRIGHTMAN.

Manufacture of anti-serum for prevention or treatment of scarlet fever. I. G. FARBERIND. A.-G. (B.P. 276,024, 15.8.27. Ger., 14.8.26).—Scarlet fever bacilli or varieties thereof are cultivated either on solid nutrient media, e.g., solidified Löffler's serum, or on liquid media, e.g., beef broth containing 2% of peptone, and the bacilli are separated from the medium and made into a suspension for injection. The suspension and culture are alternatively injected subcutaneously or intravenously, e.g., into horses, either with or without previous inoculation of the culture with streptococci, and when immunisation of the animal is complete the serum is recovered in the usual manner.

R. BRIGHTMAN.

Manufacture of physiologically-active substances from [secretory] organs [of females]. W. MERKI. Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,692,509, 20.11.28. Appl., 26.1.28. Switz., 23.2.27).—See B.P. 285,856; preceding.

Production of remedies from glandular organs. G. SCHRÖDER (U.S.P. 1,695,612, 18.12.28. Appl., 29.11.26. Ger., 16.12.25).—See B.P. 263,155; B., 1927, 925.

Manufacture of pharmaceutical preparations. W. SCHOELLER and K. SCHMIDT, Assrs. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,693,055, 27.11.28. Appl., 23.7.27. Ger., 2.8.26).—See B.P. 275,213; B., 1928, 83.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effect of environment on photographic sensitivity. I. S. E. SHEPPARD and E. P. WIGHTMAN (Phot. J., 1929, 69, 22—35).—The effect of p_H on the sensitivity of photographic emulsions has been studied, using both aged and freshly coated plates. The adjustment of p_H before or during exposure by bathing aged plates in buffer mixtures gave only small changes of speed with p_H , and no greater changes were observed on remelting the emulsion and adjusting the p_H in the melt. Bathed plates prepared from fresh emulsion showed greater changes, which became more marked when the p_H adjustment was made in the emulsion before coating. An emulsion prepared with inert gelatin and a similar emulsion prepared with thiourea-activated gelatin both showed dependence of speed on p_H , but the results were not definitely reversible. In all cases the speed increased as the p_H was raised and *vice versa*, Rawling's results (B., 1927, 125) having been confirmed for freshly prepared emulsions. Theories of the cause of the effect and its variation are discussed. J. W. GLASSETT.

Anti-fogging and anti-sensitising effects. S. E. SHEPPARD (Phot. J., 1929, 69, 37—40).—A definite chemical mechanism of nucleus inactivation is suggested for the action of thioanilides, which, reacting chiefly in the tautomeric form $SH \cdot CR : NPh$ ($R = \text{alkyl}$), are assumed to form complex silver compounds preferentially at the interface of sensitising specks of silver sulphide with silver bromide or of silver with silver bromide, owing to the greater reactivity of the silver bromide at these points. This mechanism is extended to the photographic activity of the iminazoles and dye sensitizers. J. W. GLASSETT.

Development after fixation of inverted or solarised photographic plates. H. BELLIOU (Compt. rend., 1928, 187, 1289—1292).—Plates developed after fixation for 10 min. in *N*-solutions of sodium bisulphite or thiosulphate showed images (direct or inverted) produced by red or infra-red radiations, the region previously solarised being the densest, though the solarised portion of the spectrum was less dense than the solarised background. The author's hypothesis (B., 1927, 174) is accordingly modified, the sodium thiosulphate being considered to be capable of behaving as a slow developer as well as a fixing agent. The experiments of other workers have led to the same results. J. GRANT.

PATENTS.

Photographic sensitive materials. A. HAMBURGER (B.P. 301,962, 10.9.27).—The sensitivity of photographic emulsions is increased by treating them, either before or after coating, with the double silver salt formed by adding 75 c.c. of a 5% solution of silver

nitrate gradually with stirring to 21.5 litres of a 0.4% solution of sodium diborate. Sensitisation may also be effected by bathing the coated plates, before exposure, in a solution of a sensitising dye to which an alkaline borate has been added. J. W. GLASSETT.

Manufacture of dichromate-glue printing plates for hand-printing or press deep printing. M. WIELAND (U.S.P. 1,692,528, 20.11.28. Appl., 7.11.24. Ger., 13.11.23. Renewed 22.9.28).—The quantity of liquid binder which is used is such that the ability of the glue to swell is preserved. F. G. CLARKE.

Production of monochrome images on photographic films. SOC. TECHN. D'OPTIQUE ET DE PHOTOGRAPHIE (S.T.O.P.) (B.P. 283,954, 6.1.28. Fr., 22.1.27).—The rear side of the support for the sensitive layer is moulded into spherical caps, which act as lenses with their focal planes in coincidence with the sensitive layer. The incident light is thereby concentrated into a large number of bright points which appear as black dots on the negative. For reproduction, the negative is replaced in the camera and the position and diameter of the diaphragm of the objective are adjusted until the white field which surrounds each dot disappears. J. W. GLASSETT.

Retarding the [photographic] developing action of *p*-aminophenol. SILESIA VER. CHEM. FABR. (G.P. 450,959, 1.5.25).—The developer solution is treated with a soluble hydrogen carbonate. A. R. POWELL.

XXII.—EXPLOSIVES; MATCHES.

Accuracy of the methyl-violet, zinc iodide-starch, and Abel heat test as compared with other methods of testing the stability of smokeless powders. F. LENZE and L. METZ (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 340—343, 381—384, 428—431).—Samples of S powder manufactured at various times from 1910 to 1928 were tested by the methyl-violet and zinc iodide-starch tests, Abel heat test, German test at 132°, storage at 100° and 75°, Bergmann-Junk test, and Hansen's hydrogen-ion test. Of these the Bergmann-Junk was the most suitable. The methyl-violet test merely showed when decomposition began, and gave no information about the subsequent behaviour of the powder. The quality of the test paper employed influenced the results. The zinc iodide-starch test suffered from the same disadvantages, and in addition the results varied with the weather. Storage at 100° gave useful information, whilst for checking other tests of short duration storage at 75° was valuable. S. BINNING.

Nature of nitrocellulose. H. BRUNSWIG (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 337—340, 384—387).—The theory that nitrocellulose is a mixture of unaltered cellulose and cellulose trinitrate, which was deduced by Herzog and von Náray-Szabó by X-ray methods (A., 1928, 48), is criticised on several grounds. The theory fails to explain why nitrocelluloses with the same physical and chemical properties can be obtained either by nitrating cellulose or by denitrating nitrocellulose. According to the theory, the decomposition velocities

of various nitrocelluloses would be directly proportional to their content of cellulose trinitrate, but the relation between nitrogen content and decomposition velocity is actually exponential and not linear. Various phenomena attending purification and stabilisation of nitrocellulose are also against the theory. These facts and also the relation between the solubility of nitrocellulose in ether-alcohol and the nitrogen content and the dependence of both on the composition of the nitrating acid are better explained by the theory that nitrocellulose is a mixture of cellulose nitrates of varying degrees of nitration.
S. BINNING.

Mixtures of pentaerythritol tetranitrate and nitroglycerin as shell-filler and initiating explosive of high brisance. A. STETTBACHER (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 345—348).—Pentaerythritol tetranitrate when mixed with 20% of nitroglycerin can be compressed at 600 atm. to d 1.69—1.70 without exudation of nitroglycerin. The mixture is suitable for filling shells, as an initiating explosive, and in detonating fuses. The detonation velocity is 7000—8000 m./sec. Firing tests on iron plates show that a mixture of the tetranitrate with nitroglycerin (80 : 20) is more brisant than pure nitroglycerin, a mixture of picric acid and nitroglycerin (80 : 20), guhr-dynamite 70%, or trinitrotoluene.
S. BINNING.

PATENTS.

Explosive. F. H. BERGEIM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,691,955, 20.11.28. Appl., 15.4.27).—The dinitrate of a nitroglycerol containing a branched chain, e.g., of β -methyl- or β -ethyl- β -nitropropylene glycol, is used.
F. G. CLARKE.

Cyano-nitrate explosive. F. H. BERGEIM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,685,771, 2.10.28. Appl., 15.9.27).—Cyanohydrins derived from aliphatic alcohols are nitrated at 0—10° with a mixture of 40% of nitric acid and 60% of sulphuric acid, and the reaction mixture is poured into a large volume of water. The oily precipitate is washed with 5% sodium carbonate solution, then with water. Cyanoethyl nitrate is useful as an addition to dynamite explosives.
A. R. POWELL.

Production of erythritol tetranitrate. F. H. BERGEIM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,691,954, 20.11.28. Appl., 15.4.27).—A sulphuric acid solution of erythritol is added to nitric acid.
F. G. CLARKE.

Ignition pellets for electric blasting fuses. W. ESCHBACH and W. FRIEDERICH (B.P. 281,239, 31.10.27. Ger., 23.11.26).—The safety of blasting operations is increased by using lead salts of di- or tri-nitrophenol in ignition pellets.
B. FULLMAN.

Explosive. A. C. SCOTT, Assr. to MEXICO, LTD. (U.S.P. 1,695,932, 18.12.28. Appl., 28.7.26. U.K., 12.8.25).—See B.P. 262,491; B., 1927, 126.

XXIII.—SANITATION; WATER PURIFICATION.

Purification of the sewage of Elmsborn (Holstein) with special reference to effluents from leather works. A. SNOEK (Collegium, 1928, 612—

621).—1 pt. of tannery sewage and 2 pts. of other sewage is mixed and treated by the Dywidag patent process (Dyckerhoff and Widmann), which yields a perfectly clear, odourless liquor incapable of putrefaction owing to its high oxygen content, and is practically free from spores after sand-filtration. D. WOODROFFE.

Sewage sludge as fertiliser. W. RUDOLFS (Soil Sci., 1928, 26, 455—458).—Analysis of many sewage sludges shows that aerobically treated sludge has an average nitrogen content of 5% and anaerobic sludge of 2.25%. The large annual losses of nitrogen incurred through the failure to utilise sewage sludge are emphasised.
A. G. POLLARD.

Progress in chlorination of water, 1927—1928. L. H. ENSLOW (J. Amer. Water Works' Assoc., 1928, 20, 819—846).—The tendency in the chlorination of water supplies is to apply the chlorine in two doses, before and after the filters. By this means the bacterial load on the filters is reduced, slime, mud spots, and cracks in the sand bed are minimised with a consequent increase in length of filter runs, and a better effluent is obtained, rendering the post-filter dose largely precautionary when the *B. coli* content of the raw water does not exceed 60 per 100 c.c. In addition, pre-chlorination reduces algal growths in the settlement basins, retards septic action in the deposited sludge, and, when iron is present in the raw water, may reduce the quantity of coagulant required. The increase in chlorine consumption should not exceed 50%.
C. JEPSON.

Algæ control [in water purification] by creating turbidity at Louisville, Ky. W. H. LOVEJOY (Eng. News-Rec., 1928, 101, 505—507).—Excessive algal growths coincident with periods of low turbidity in the raw water caused rapid choking of the filters at Louisville, Ky. An artificial turbidity, created by means of mud from the reservoirs, more than doubled the average length of the filter runs, probably owing to many of the organisms being carried down by the mud as it settled.
C. JEPSON.

Effluent water of cellulose manufacture. HEIDUSCHKA and MUNDS.—See V.

PATENTS.

System and apparatus for sewage [disposal]. G. G. SMITH (U.S.P. 1,695,669, 18.12.28. Appl., 24.3.25).—A sewage-disposal system consisting of a settlement tank, with an additional tank for storage of sludge and a filter on which the tank effluent is distributed by means of fixed sprays.
C. JEPSON.

Neutralising the alkalinity of water. A. H. WHITE (U.S.P. 1,693,065, 27.11.28. Appl., 7.12.25).—Sufficient phosphoric acid is added to bring the acidity of the water, after removal of its carbon dioxide content, to not more than 0.5%.
W. G. CAREY.

Separators [for town's refuse]. HEENAN & FROUDE, LTD., and R. C. WAREHAM (B.P. 302,021, 4.11.27).

[Automatic] apparatus for [base-exchange] treatment of water. AKTIEBOLAGET FILTRUM (B.P. 293,033, 22.6.28. Swed., 1.7.27).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 1, 1929.

I.—GENERAL; PLANT; MACHINERY.

Rapid method for approximate determination of sorption isothermals of vapours on charcoal. I. Principle of the method. A. J. ALLMAND and J. E. MANNING. II. Simple retentivity test. A. J. ALLMAND and L. J. BURRAGE (J.S.C.I., 1928, 47, 369—372 T, 372—376 T).—I. In order to evaluate the efficiency of a charcoal for sorbing vapours, a complete knowledge of its sorption isothermal is necessary. In practice, owing to experimental difficulties, this isotherm is never determined, but is substituted by a constant-pressure "breakdown" test, by tests made on adsorption from solution, by measurements of heat of sorption, etc. The authors have worked out a simple method of obtaining the sorption isotherm or, rather, a close approximation to it. The method depends on charging a column of charcoal with the vapour concerned at a certain pressure, and then following the rate at which this sorbed vapour is removed by passage of a stream of dry air. The tangents to these "rate" or "retentivity" curves give a measure of the vapour pressure above the charcoal at any instant in the desorption process, and from them the form of the isothermal can readily be deduced.

II. The apparatus and method used for obtaining the retentivity curves are fully described. Examples of the derived isothermals are given, together with a comparison between derived and directly measured true isothermals. The agreement in the latter case, though not perfect, is good.

Method for simultaneous measurement of plasticity and elasticity. E. C. BINGHAM and J. W. ROBERTSON (Kolloid-Z., 1929, 47, 1—5).—A method for measuring plasticity is described in which the substance is contained in a capillary tube, the ends of which are subject to a definite alternating pressure. The method has been applied to a 43% ammonium oxalate solution, and it is shown that the elastic deformation is directly proportional to the shearing power. E. S. HEDGES.

Drying of solids. I. T. K. SHERWOOD (Ind. Eng. Chem., 1929, 21, 12—16).—The drying of a solid may proceed either by evaporation at the surface or within the solid, and the resistance to internal liquid diffusion may be great or small. Very wet solids dry at a constant rate, the mechanism being similar to the evaporation of a liquid, but as drying proceeds conditions change. The case where evaporation is at the surface and internal diffusion-resistance is the limiting factor is considered in detail. The equations are similar to those for heat conduction in solids, and are plotted as a series of curves for different ratios of free liquid content to critical free liquid content. A graphical method is

described for obtaining the diffusion constant of Newton's equation from experimental results. Wood and clay are examples of this mechanism in drying if the slabs used are not too thin and if air is circulated mechanically over the surface. The drying of soap, on the other hand, shows a decrease in the diffusion constant as drying proceeds, connected with the shrinkage which occurs. This result is confirmed by determinations of moisture gradient in a slab. C. IRWIN.

Theory of fine grinding. IX. Connexion between the statistical diameter and statistical volume of irregularly-shaped particles of crushed sand. X. Connexion between the statistical diameter of crushed sand particles and their statistical surface. G. MARTIN [with E. A. BOWES]. XI. Calculations relating to diameters, surfaces, and weights of homogeneous grades of crushed quartz sand. G. MARTIN (Trans. Ceram. Soc., 1928, 27, 247—258, 259—284, 285—289; cf. B., 1927, 623).—IX. Experimental evidence is presented to show that, with irregularly-shaped particles of crushed sand, a statistical volume constant is given by V/d^3 , d being the statistical diameter of the particles, and V the corresponding statistical volume. The mean value for this constant, obtained with five samples of carefully graded, air-elutriated sand, was 0.277. The value was sensibly the same for particles varying in size from 116,520 to 2188 per g. Hence the average shape of sand particles is the same, whether they be large or small.

X. Similarly, the statistical surface constant is given by S/d^2 , where S is the statistical surface of the particles and d the diameter. For sand particles ranging from 0.00333 to 0.01089 cm. in diam., this constant is about 2.1. It is sensibly the same for large and small particles.

XI. The chief numerical data relating to 20 homogeneous grades of quartz particles ranging in size from 31,000,000 to 1240 per g. are given, and methods are presented for calculating the volumes, surfaces, and weights of powders of different size. F. SALT.

Optical method for measuring size of particles in suspensions. G. I. POKROVSKI (Kolloid-Z., 1929, 47, 55—58).—Sources of error in the gravimetric method of measuring the size of particles in suspensions are indicated, and an apparatus is described by means of which the determination can be made by a purely optical method. This is based on the measurement of the polarisation of the light scattered by the suspension. E. S. HEDGES.

Crystal growth in aqueous solutions. I. Theory. W. L. McCABE (Ind. Eng. Chem., 1929, 21, 30—33).—If a known weight of seed crystals of known screen

analysis is suspended in a saturated solution and the solution slowly cooled without formation of fresh nuclei, a theoretical method can be developed for the calculation of the final weight and screen analysis of the crystals produced. It is assumed that no considerable degree of supersaturation occurs and that each crystal as it grows retains its geometrical form. The weight is calculated from solubility data and the result interpolated in an equation relating final and initial weights and particle sizes.

C. IRWIN.

Chemistry of mists and dusts. H. REMY (Chem.-Ztg., 1928, 52, 677—679, 698—699).—The difference in behaviour of sulphur trioxide made in a contact plant, which is absorbed only to a slight extent in water, but completely in sulphuric acid of 98.5% concentration, and of that obtained by passing air through fuming acid, which is less completely absorbed by sulphuric acid than by water, being merely rendered invisible by passing through the acid, is attributed to the fact that the trioxide obtained by the latter method is moist, whereas that made by the contact plant is dry. The former is a mist, of particle size about 10^{-4} cm.; the latter a colloidal dust, of particle size 10^{-5} cm. The extent to which a dry colloidal dust is absorbed by aqueous fluids is proportional to the vapour pressures of the fluids, and is influenced by their viscosities. In no case were the particles found to be electrically charged. The influence of particle size is discussed.

S. I. LEVY.

Simplified methods of potentiometric and conductometric analysis and their industrial application. T. CALLAN and S. HORROBIN (J.S.C.I., 1928, 47, 329—343 r).—The general principles of potentiometric and conductometric analysis are briefly explained. Simplified methods of potentiometric analysis involving the use of end-point cells are discussed and the preparation and use of such cells without the use of expensive electrical apparatus is described. A number of applications of such end-point cells are described, including their use in the determination of acidity, alkalinity, and halides in dyestuffs and coloured solutions, analysis by means of titanous chloride, and the determination of amines and phenolic compounds such as aniline, phenol, etc. by means of potassium bromate.

The various forms of apparatus for conductometric analysis are reviewed and an investigation into the use of the thermionic valve, as a rectifier to enable direct reading to be obtained on a *D.C.* instrument, is described, in the course of which the most suitable type of valve, the best conditions of anode voltage, grid bias, etc. were determined. A new simple apparatus in which alternating current from the lighting mains is transformed down, applied to the titration cell, rectified by a crystal "detector," and measured on a *D.C.* millivoltmeter is described. The conductometric method is particularly applied to titrations of sulphates with barium salts, of weak acids and bases, etc., for which the potentiometric method is unsuitable.

PATENTS.

Mechanical furnace. O. H. BUSE, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,693,366, 27.11.28. Appl., 13.10.25).—A two-stage mechanical furnace comprises

a furnace chamber having a charging pot above it provided with a stirrer and with an opening for gravity-discharge into the furnace, the opening being closed by a removable plug provided with a sealing groove situated between the plug and the wall of the opening when in the closed position.

L. A. COLES.

Rotary-hearth furnace. O. A. COLBY, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,883, 18.12.28. Appl., 14.7.27).—A rotary-hearth furnace comprises material-carrying trays pivotally mounted upon a hearth, and means for normally maintaining material being heated on the trays and for tilting the trays to discharge the material. J. S. G. THOMAS.

Tiltable furnace. G. SCHMITT, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,863, 18.12.28. Appl., 16.2.27. Ger., 20.4.26).—A cylindrical furnace casing is arranged between spaced, vertical members forming a base and having each a horizontal bearing surface and a vertical guiding surface. The casing is supported by means extending through its centre of gravity, and means are provided for engagement of the furnace with the guiding surfaces and for tilting the furnace.

J. S. G. THOMAS.

Pulverulent-fuel furnace. C. DUQUENNE (U.S.P. 1,693,597, 4.12.28. Appl., 10.10.25. Fr., 27.10.24).—An air-grate is placed between the combustion chamber and ash pit of the furnace; the grate is formed of a number of interfitting iron sections forming conduits for passage of air to a hollow wall and thence to the combustion chamber. The iron sections are covered with refractory material and supported by beams within the air conduits, the beams thus being exposed everywhere except on the top to the air currents and kept cool thereby.

B. M. VENABLES.

Furnace regulation. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. R. SMITH (B.P. 283,903, 19.1.28. U.S., 19.1.27).—A complicated system of electrical relays is provided by which the ratio of fuel to air is kept constant under varying load. In a boiler the fuel supply is regulated by the steam pressure, and if a number of boilers are working together the steam pressure in the common main regulates, through a master relay, the fuel supply to all the boilers, while individual relays keep the fuel-air ratio constant.

B. M. VENABLES.

Grinding, crushing, or like mills. A. STEINBRÜCKNER (B.P. 301,667, 10.1.28).—In a mill of the type where a grinding ring runs round rollers or where a beater member is rotated inside a stationary grinding ring, an annular space is left between the outside of the ring and the casing in which is a ring-shaped bucket conveyor driven either separately from or by the ring for the purpose of lifting coarse material which has settled in the lower part of the casing up to the top, whence it falls through curved side passages back into the grinding zone. A stream of air is also injected tangentially into the lower part of the annular space and exhausted from the upper part, carrying with it the finely-ground material. A classification device is inserted in the air-exhaust pipe.

B. M. VENABLES.

Crushing, grinding, and refining machine. S. BRAMLEY-MOORE (U.S.P. 1,692,884, 27.11.28. Appl.,

6.4.28).—A cylindrical casing is provided with an interior, fixed grinding surface, and rotating grinding elements are carried by levers which are driven by a wheel within the apparatus. A disc on another co-axial shaft rotates with, and has angular adjustment relative to, the wheel. The disc operates on the levers to vary the pressure between the grinding elements and surface. B. M. VENABLES.

Rotary dryer. F. A. JONES (U.S.P. 1,694,708, 11.12.28. Appl., 24.3.28).—An inclined rotary kiln is surrounded by longitudinal pockets, the material under treatment being charged into the upper ends, and discharged from the lower ends of these. L. A. COLES.

Liquid filter. A. WUEST (U.S.P. 1,693,741, 4.12.28. Appl., 23.5.27).—A cylindrical filtering unit of which the wall comprises, in order outwards, the filter medium, the support for the medium or inner screen, and an outer screen formed from two troughs, of semi-circular section, interlocked at their edges. B. M. VENABLES.

Refrigerating apparatus. P. W. TAYLOR (B.P. 303,569, 8.10.27).

Refrigerating plants. C. H. COOK, and SERVEL, LTD. (B.P. 303,425, 3.9.27).

Refrigeration apparatus. SILICA GEL CORP., Assees. of E. B. MILLER (B.P. 292,938, 20.12.27. U.S., 27.6.27).

Freezing mixture (B.P. 298,623).—See VII. **Centrifugal apparatus for recovery of vapours** (B.P. 298,226).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Constitution and classification of coal. A. C. FIELDNER (Fuel, 1929, 8, 36–45).—Systems of coal classification are discussed in the light of our present knowledge of its constitution, and the programme of the American Engineering Standards Sectional Committee is outlined. It is concluded that coal should be classed primarily on the basis of its intrinsic chemical and physical properties, and that use classification should be secondary to and as far as possible correlated with scientific classification. Scientific classification depends on two primary factors, the composition and type of the original coal-forming vegetation and the degree of metamorphism or coalification of the vegetable residue. The criteria to be considered for classifying under these two general factors are proximate and ultimate analyses, calorific values, microscopical examination, extraction with solvents, reaction with reagents, and destructive distillation. A. B. MANNING.

Vitrain. M. M. EVANS, L. SLATER, and R. V. WHEELER (Fuel, 1929, 8, 30–36).—Various types of plant structure, in particular parenchyma, periderm, and xylem, have been observed in British vitrains by microscopical examination of polished and etched surfaces in incident light. From a study of its micro-structure, vitrain appears to be a simple substance (as distinct from the aggregates, clarain and durain) consisting of ulmin compounds which have infilled plant cells and partly, if not entirely, replaced the materials of the cell walls. Although the evidence regarding the

species of plants to which the structures relate is indirect, it appears probable that certain vitrains at least represent the residues of *Sigillaria* and of *Lepidophlois*. A close similarity between the structures of vitrain and fusain has sometimes been observed when the two are in close association. Some details are given of the method used in preparing specimens for microscopical examination. A. B. MANNING.

Relationship between the sp. gr. and ash contents of the coals of Korea and Bokaro : coals as colloid systems. L. L. FERMOR (Rec. Geol. Survey of India, June, 1928; Fuel, 1929, 8, 16–29).—A study of coals from Korea and from Bokaro has established the empirical relationship, $a = 100(g - k)$, between a , the ash content, g , the sp. gr., and k , the sp. gr. of the pure coal substance, which is constant for each field (1.28 and 1.26, respectively). The relationship holds up to coals containing 50% of ash. The higher sp. gr. of a few coals which form exceptions to the rule is due to the high iron content of the ash. The sp. gr. of the coals which conform to the linear law, however, are higher than values calculated on the assumption of simple admixture of pure coal and pure shale. This is explained on the supposition that the coals are colloidal systems, in which the ash forms the disperse phase, and the vitrain the dispersion medium. Evidence is adduced also of the colloidal dispersion of the moisture in the vitrain. The bearing of this point of view on the classification and the treatment of coal is discussed. A. B. MANNING.

Reactions and gas-flow in [gas] generators as a basis for their constructional design. E. TERRES (Gas- u. Wasserfach, 1928, 71, 1205–1213).—The percentage of hydrogen in the gas forms a better criterion of the efficiency of the generator operation than the percentage of carbon dioxide. Determinations of the gas composition at points within the fuel bed show that the various reactions occurring therein take place in zones, not disposed horizontally, but extending to the surface of the fuel bed. The gas of the central zone is of good composition, but that in the outer zones contains successively greater percentages of carbon dioxide. The position and breadth of the zones contributing carbon dioxide to the gas depend on the cooling of the fuel bed near the outer surface of the generator. For each size of fuel there is an optimum depth of fuel bed, further improvement of the gas being then possible only by increasing the diameter of the generator, assuming, at the same time, that it is possible to maintain a uniform distribution of air over the cross-section. The reactivity and the sieve analysis of the fuel are of importance in judging its suitability for use in the generator. The amount of dust in the gas and the resistance of the fuel bed to the air are determined principally by the proportion of fuel less than 5 mm. size. A short description of the generator used in the experiments is appended to the paper. A. B. MANNING.

A source of error in the carbon balance [of gas-producer tests]. WEHRMANN (Gas- u. Wasserfach, 1928, 71, 1253).—Errors may arise in drawing up a carbon balance for a gas producer from the absorption

of carbon dioxide in the gas scrubber, the aspirator, and in the gas burette. Analyses of the gas taken directly from the producer outlet have been compared with those of corresponding samples taken after the gas has passed the scrubber, the former determinations being made gravimetrically and the latter volumetrically. The decrease in the percentage of carbon dioxide due to absorption varied from 0.4 to 0.9, the average being 0.65.

A. B. MANNING.

Chemical processes in the gasification of brown coal. H. KNOPF (Gas- u. Wasserfach, 1928, 71, 1218—1221, 1242—1245, 1259—1263).—Measurements of temperature and of gas composition have been made at points in the axis of the fuel bed of a Heller gas generator charged with brown coal. Although the proportions of hydrogen, carbon monoxide and dioxide, and steam in the gas correspond in general with the water-gas equilibrium ($\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$) a detailed study of the results leads to the conclusion that the water-gas reaction itself plays little part in the generator reactions. The measurements of temperature correspond with that of the solid fuel; that of the gas must always be higher except at the base of the generator. The gas temperatures have been calculated by drawing up heat balances for different zones of the fuel bed. The differences between the gas and fuel temperatures are greater the drier is the fuel. The relation between these temperature differences, the gas composition, the depth of the fuel bed, and other factors is discussed. Except when wet fuel is used, an increase in the depth of the fuel bed beyond a certain limit has no influence on the gas composition.

A. B. MANNING.

Water-gas from bituminous coal. J. GWOSDZ (Gas- u. Wasserfach, 1928, 71, 1233—1239, 1253—1259).—Recent practice in the operation of water-gas producers with bituminous coal, particularly in America, and its probable influence on the development of the German gas industry are critically discussed.

A. B. MANNING.

Cyclic process for ammonia recovery from coke-oven gases. V. I. DENISOV (J. Chem. Ind. Moscow, 1928, 5, 13—18).—The cyclic process, and losses of ammonia occasioned therein, are discussed. Treatment of the stock water with lime is unremunerative. Corrosion of iron apparatus by the ammonia solution does not take place.

CHEMICAL ABSTRACTS.

Cyclic ammonia process. M. D. LIEPIATSKIK (J. Chem. Ind. Moscow, 1928, 5, 480—483).—In the lime water process the ammonia recovered represents 0.22% of the coal used; in the cyclic process the yield is 0.13—0.22%. The losses of ammonia are discussed, and desirable modifications (e.g., distillation of crude ammonia water by external heat) are suggested. The ammonia obtained by the cyclic process is contaminated with carbon dioxide and hydrogen sulphide.

CHEMICAL ABSTRACTS.

Equilibrium combustion of a mixture of carbon monoxide and hydrogen. C. C. MINTER (J.S.C.I., 1929, 48, 35—38 r).—Examination of a set of analytical data obtained by U.S. Bureau of Mines shows that when carbon monoxide and hydrogen are burnt simultaneously in the same mixture at high temperatures (1200—1500°),

the two gases are not oxidised independently. Graphs connecting the concentration of carbon monoxide and hydrogen with time show that the reactions are both unimolecular, with the same velocity coefficient. The ratio of the fractional changes in concentration of the two gases in the same interval is found to be equal to unity. The two gases thus disappear at the same rate, the rate being proportional to the first power of the concentration of the reacting gases. This means that the rate of oxidation is in both cases independent of the oxygen concentration. This anomalous result is explained by assuming that the water-gas reaction is operating, and, under such conditions, the rate of oxidation would be dependent on the rate at which the water-gas equilibrium is adjusted to changes in concentration of the carbon monoxide and hydrogen.

Production of gaseous, liquid, and solid hydrocarbons from methane. I. Thermal decomposition of methane. H. M. STANLEY and A. W. NASH (J.S.C.I., 1929, 48, 1—8 r).—The thermal decomposition of methane has been examined from the point of view of the production of higher hydrocarbons. By passing a stream of methane through silica tubes heated to 1000—1200°, liquid, solid, and higher gaseous hydrocarbons were produced. The liquid and solid products were chiefly of aromatic nature. A special study was made of the gaseous products using low-temperature condensation methods of analysis, and among these were identified, besides hydrogen, acetylene, ethylene, and small quantities of ethane, higher olefines, and aromatic hydrocarbons. For each temperature there is a heating period which gives optimum yields of higher products the duration of which is decreased by rise in temperature. The best yields of higher hydrocarbons were obtained at a temperature of 1150° and a heating duration of 0.6 sec. Under these conditions the yield of a light oil and tar was 11.0% calculated on the methane used, while the quantities of acetylene and ethylene in the reaction gas corresponded to a total conversion of methane into these gases of 8.8%. Thus the total conversion of methane into higher hydrocarbons was nearly 20% of the theoretical, while only about 6% of methane had been converted into its elements. The yield of light oil was 4.8%, or 0.2 gal. per 1000 cub. ft. of methane. The use of comparatively long heating periods tended to cause methane to decompose into its elements almost exclusively, this tendency being greatly increased by the use of large heating surfaces and of catalytically active materials such as iron and nickel. The mechanism of the process is discussed in the light of the theory of nascent residues.

Chemical study of low-temperature tar. G. T. MORGAN, D. D. PRATT, and J. ROSS (J.S.C.I., 1929, 48, 29—34 r).—A sample of low-temperature tar from coal of the 6 ft. seam of Kinneil (Linlithgow) has been submitted to a solvent treatment at temperatures not exceeding 120°. After successive extractions with aqueous alkali and dilute acid, the neutral oils were distilled up to this temperature and the residual oil was treated successively with acetone and petroleum (b.p. 40—60°) in order to remove respectively waxes

and solid aromatic hydrocarbons. During this fractional separation neutral materials were isolated having the properties of resins. When rendered slightly acid the alkaline extract of the tar yielded crystallisable phenols, a resinous variety of phenols termed resinols, and, in addition, a further quantity of the neutral resins. Acidification with mineral acid brought about a separation of carboxylic acids mixed with acidic materials of a resinous character, to which the term resinic acids is applied. The true phenols (crystallisable phenols and resinols) were separated from phenolate-soluble materials by one or other of two alternative extractions, aqueous alkalis with organic solvents or caustic soda-brine reagent. The true phenols were then divided into crystallisable phenols, tar acids, and resinols by systematic employment of ether and light petroleum. When rendered alkaline the acidic extract of the tar furnished crystallisable and resinous bases, the latter being termed resinamines. Comparative experiments were then made on distilled and undistilled Kinneil tar. Distillation which decreased the amounts of alkali-extractable material, neutral oils, waxes, and higher aromatic hydrocarbons, led, moreover, to the formation of 31% of pitch as contrasted with about 5–6% of insoluble residue left after treatment by solvents. By the foregoing method of extraction four groups of resinous materials have been separated from low-temperature tar: neutral resins, phenolic resins or resinols, acidic resins or resinic acids, and basic resins or resinamines. In suitable organic media these resinous materials can be employed as stains and varnishes. By avoidance of high temperature, other decomposable constituents of the tar such as waxes and higher aromatics have been obtained in appreciably higher yields.

Causes of and means of reducing the water content of tar. K. BUNTE (Gas- u. Wasserfach, 1929, 72, 1–3).—The separation of water from tar depends primarily on the sp. gr. and viscosity of the latter. With viscous tars a better separation is usually obtained by warming the mixture and allowing it to settle. Increase in temperature, however, diminishes the difference in sp. gr. between water and tar, and may therefore be disadvantageous with a light tar. The sp. gr. of the tar determines the relative positions of the tar and liquor overflows from the settling tanks. For good separation these tanks should be deep and roomy, and the tar condensing at the various points of the cooling plant should be well mixed before arriving at them. A high water content and emulsion formation may be further due to surface tension effects, the presence of free carbon, the partial miscibility of water and phenols in the tar, the presence of ammonium phenoxides, and colloidal phenomena.

W. T. K. BRAUNHOLTZ.

Carboxylic acids in peat tar. G. STADNIKOV and W. SABAWIN (Brennstoff-Chem., 1929, 10, 1).—The oil which passes over when peat tar is distilled with steam contains saturated and unsaturated fatty acids (about 0.2–0.3% of the tar) having six to nine carbon atoms. It appears probable that these acids are present in the original peat.

W. T. K. BRAUNHOLTZ.

Biochemical studies on pityrol. I. Introduction. S. KOMATSU. II. Distillation of rice bran. S. FUJITA. III. Neutral constituents of pityrol. S. SAKAMI. IV. Acidic constituents. B. MASUMOTO. V. Basic constituents. S. SUZUKI (Mem. Coll. Sci. Kyoto, 1928, 11, 481–495, 497–503, 505–515, 517–519, 521–532).—I. Bran from "Shinrinki" rice, when dry-distilled from a Fischer aluminium retort at 200–450°, yields 28% of coke, 36% of aqueous liquor, 12% of gas, and 24% of the tar known as pityrol. The volatile (13%) and non-volatile (87%) portions of pityrol may be separated into neutral, acidic, and basic constituents. Neutral substances form the bulk of the tar, those from the volatile portion consisting mainly of the unsaturated hydrocarbons C_nH_{2n} (C_9 – C_{14}) and C_nH_{2n-2} (C_8 – C_{14}). The non-volatile neutral portion contains polymethylene hydrocarbons of high mol. wt. and some aromatic hydrocarbons. A fraction, b.p. above 250°/10 mm., produced cancer in a rabbit's ear by repeated application. As has been previously noted with other tars, pityrol itself has a pathological effect differing from that of its constituents. The basic portion of pityrol, though small in quantity, is the cause of the offensive odour of the tar; it contains methyl derivatives of pyridine, also quinoline and aniline. The volatile acidic portion of the tar probably contains $C_{10}H_{15} \cdot OMe$, and has a highly irritating effect on the rabbit ear. Rice bran yields more tar than peat or wood under similar conditions, owing to a greater content of protein and fats. De-fatted bran yields less tar. The unsaturated and saturated hydrocarbons in pityrol are derived from the fats in the bran (chiefly palmitic and oleic acids) by thermal decomposition and polymerisation. The saturated hydrocarbons found are those most thermally stable: C_9H_{20} , $C_{10}H_{22}$, $C_{15}H_{32}$, $C_{30}H_{62}$. The acidic compounds in pityrol, mainly furan derivatives, are formed from the pentosans, cellulose, and soluble carbohydrates of the bran, and the proteins in the bran yield the basic constituents of the tar. Pyrrole, though absent from the tar, is probably an intermediate compound in the formation of the pyridine bases.

II. Two samples of rice bran, A and B, the rice oil having been removed from the latter, had the following percentage compositions, respectively: water 14.7, 14.1; ash 9.3, 12.0; crude fat 22.4, 0.7; protein 12.9, 16.5; cellulose 11.4, 14.6; pentosans 8.7, 11.1; reducing sugars 1.3, 1.5; sucrose 10.6, 13.6. Dry samples of A and B were distilled from a Fischer retort. The composition of the coke and aqueous liquor was similar in both cases, but the gas from A contained more olefines and less carbon monoxide than that from B, whilst the preponderance of neutral constituents in the tar from A was less marked in the tar from B.

III. Successive treatment of the volatile neutral portion of pityrol with concentrated and fuming sulphuric acid separated the saturated hydrocarbons (each member of the series from octane to pentadecane being later isolated) from the unsaturated hydrocarbons forming the bulk of the sample. The treatment with sulphuric acid caused polymerisation of these latter, and also formation of acid esters and alcohols. By

identification of these compounds it was deduced that C_9H_{16} , $C_{10}H_{18}$, $C_{12}H_{20}$, and $C_{14}H_{24}$ are the chief constituents of this part of pityrol. The non-volatile neutral portion of pityrol contains polymethylene hydrocarbons similar to those found in Russian and Japanese petroleum and low-temperature coal tars.

IV. The acidic portion of pityrol, a dark brown oil having a phenolic odour, was fractionated, and the middle fraction methylated and separated into 18 fractions under reduced pressure. The presence of at least one methoxy-compound, $C_{10}H_{15} \cdot OMe$, b.p. $70-74.5^\circ/7$ mm., d_4^{25} 0.6922, n_D^{25} 1.4698, was deduced from the analytical and physical data.

V. Both the soluble and insoluble portions of the basic constituents of pityrol were found to contain primary, secondary, and tertiary bases, but no trace of pyrrole. By fractional distillation, and crystallisation of their picrates, hydrochlorides, or double salts with mercuric chloride, the following bases were identified: 2-methyl-, 3-methyl-, 2:6-dimethyl-, 2:4-dimethyl-, 2:4:6-trimethyl-pyridine, aniline, and quinoline; also 3-dimethylpyridines (?), the double salts of which, m.p. $163-164^\circ$, 110° , and 112° , respectively, gave analyses agreeing with $C_7H_5N \cdot HCl \cdot 2HgCl_2$; a pyridine derivative (?) (double salt, $C_5H_5N \cdot HCl \cdot 2HgCl_2$, m.p. 116°); and a tetramethylpyridine (?) (picrate, m.p. 106°). From the mother-liquor after the separation of quinoline picrate, crystals, m.p. 180° , were obtained, which await further investigation. B. W. ANDERSON.

ψ -Cumene derivatives in crude wood spirit. B. HOLMBERG (Svensk Kem. Tidskr., 1928, 40, 304-315).—The substance of unknown composition previously described (A., 1923, i, 226) as obtained during the purification of wood spirit distilled without previous neutralisation from pyroligneous acid derived from a mixture of pine wood (80%) and spruce wood (20%) has now been identified as 5- ψ -cumylacetone. On reduction with zinc and hydrochloric acid it yielded 5-propyl- ψ -cumene, b.p. $226-228^\circ$, d_4^{20} 0.887, n_D^{20} 1.5095. With sodium and alcohol 5- ψ -cumyl-sec.-propyl alcohol, m.p. $74-75^\circ$, was formed. Bromination in acetic acid gave 3:6-dibromo-5- ψ -cumyl- α -dibromoacetone, m.p. $116-117^\circ$. An ethereal solution yielded with magnesium methyl iodide 5- ψ -cumyl-tert.-butyl alcohol, m.p. $45-47^\circ$. On oxidation with sodium hypobromite durylic acid (ψ -cumene-5-carboxylic acid) resulted. Alkalis decomposed it slowly giving resinous products, together with small amounts of durylic acid. In addition to the first-named compound a small amount of 6- ψ -cumenol has now been identified among the products of the above-mentioned distillation; this substance has not previously been reported as occurring in wood spirit. 5- ψ -Cumylacetic acid, m.p. $117-120^\circ$ (ethyl ester, b.p. $141-142^\circ/11$ mm., d_4^{20} 1.006, n_D^{20} 1.507), is also described. H. F. HARWOOD.

Transformation of the fatty acids during geological epochs. I. G. L. STADNIKOV and A. E. WEIZMANN (J. Russ. Phys. Chem. Soc., 1928, 60, 1123-1131). II. G. L. STADNIKOV and Z. S. VOZSCHINSKAJA (Ibid., 1133-1136).—I. In order to elucidate the mechanism of formation of hydrocarbon oils in the earth's crust from organic matter, the deeper layers of

peat-bog deposits, which represent transition stages between peat and young bituminous coals, were investigated. The primary tars from these deposits were found to contain large quantities of highly polymerised and dehydrated acids of the fatty series. The solids themselves (from the Sucho-Kujatsk district, Siberia) were powdered, and extracted successively with (a) benzene, (b) chloroform, (c) ethyl-alcoholic potassium hydroxide, (d) amyl-alcoholic potassium hydroxide, (e) ethyl alcohol, (f) water. Analysis of the extracts showed them to contain the following: (a) (1.6% of total) a brown solid, similar in appearance to peat bitumen, which on esterification and fractionation *in vacuo* was found to be a mixture of fatty and unsaturated acids of the $C_{12}-C_{22}$ series; (b) (0.46%) a gummy tar (not analysed); (c) only salts of acids, both fatty and unsaturated, ranging from octoic to C_{16} acids; (d) mostly fatty acids, including octoic, boiling range $100-215^\circ$, and also some unsaturated acids; (e) a mixture of acids, boiling range $120-280^\circ$, including the butyric, valeric, and hexoic, and higher fatty acids up to C_{11} ; (f) mostly valeric acid, together with an unsaturated acid of high mol. wt. The results thus obtained confirmed the analysis of the primary tars.

II. The samples from peat-bogs of the Matagausk and Sucho-Kujatsk districts were similarly examined. Extract (b) was not analysed, as it contained only 0.22% of black, viscous tar. Extract (a) (1.8%) was a brown solid, which, on hydrolysis, yielded a mixture of acids m.p. 30° (approx.), containing 72.79% C and 10.38% H, and having saponif. value 186.3, iodine value 11.6, approx. formula $C_{15}H_{26}O_2$. Extract (c) gave also a mixture of acids, both fatty and unsaturated, of the series $C_{10}-C_{18}$. Extract (d) on careful fractionation *in vacuo* gave butyric, valeric, and hexoic acids. Extract (e) yielded a solid which approximated to an unsaturated C_8 acid, whilst (f) contained acids, both fatty and unsaturated, of the C_6-C_8 series. The acids, apparently, were all monobasic, and included, in all probability, hydroxy-acids, which formed lactones.

M. ZVEGINTZOV.

Oxidation of motor fuels. E. BERL, K. HEISE, and K. WINNACKER (Z. physikal. Chem., 1928, 139, 453-481).—The self-ignition temperatures of mixtures of the inflammable vapours of motor fuels with air at normal pressures and at pressures up to 20 atm. have been determined together with the influence of the addition of anti-knocking substances on these temperatures. In the case of mixtures of air with pentane, *n*-hexane, and cyclohexane increased pressure has only a slight depressing effect on the temperature of self-ignition for a mixture of constant composition. With ether vapour and air the effect is somewhat greater, but with benzene-air and alcohol vapour-air mixtures ignition does not occur at 800° under the pressures employed. cycloHexene at 60° and 20 atm. is polymerised by the oxygen in the admixed air. In general, substances which cause knocking most readily are those which are the most easily oxidised. The compression strength increases according to the series, hexane, cyclohexane, cyclohexene, and benzene. The addition of lead tetraethyl or iron carbonyl raises the ignition temperatures to approximately the same

extent. The oxidation of the hydrocarbons in the presence of insufficient oxygen has also been investigated together with the effect of the addition of lead tetraethyl, iron pentacarbonyl, and finely-divided lead and iron. In contradistinction to the far-reaching changes in hexane brought about by oxidation, the addition of lead tetraethyl or iron carbonyl is accompanied by only a small alteration in the b.p. curve of pure hexane; the anti-knocks prove to be powerful negative catalysts for the conveyance of oxygen, and act by decomposition into the corresponding finely-divided metal. The effect is simulated to a smaller extent by lead and iron pulverised in a luminous arc. Hydrocarbons rich in hydrogen decompose into hydrogen and an unsaturated residue both of which are oxidised forming water, hydrogen peroxide, and organic peroxides. The finely-divided metal inhibits this formation of peroxides before the succeeding intramolecular oxidation can take place.

L. S. THEOBALD.

Oils for turbines. M. VAN RYSELBERGE (Bull. Fed. Ind. Chim. Belg., 1928, 7, 445—470).—The characteristics of lubricating oils for steam turbines, their requirements, manufacture, method of use, and value of laboratory tests as a means of selection of the most suitable are reviewed. A consideration of the usual physical tests—density, flash points, and viscosity—shows that these are no aid to the selection, and three further specific characteristics are investigated. Twelve oils have been examined with respect to their lubricating power on a Dittmar machine, in which a shaft is rotated in a bearing by means of an electric motor. The bearing is lubricated with each oil in turn. The motor is started, and stopped when the shaft reaches 1400 r.p.m., and the time is recorded for the shaft to come to rest. The determination is carried out at four temperatures. Curves drawn, however, are almost superposable, and from this and other considerations the test, as a selective one, is rejected as unsatisfactory. The rise in temperature of the oil or of the bearing on the same machine after running for 60 min. seemed to be promising, but was discarded owing to the impossibility of duplicating results. A final study was made of the emulsive properties of lubricating oils. The steam-emulsifying method of the I.P.T. is considered as accurate, but insufficiently selective for turbine oils. The method adopted consisted in agitating for 2 min. 40 c.c. of oil and 20 c.c. of distilled water in a 100 c.c. Pyrex cylinder by means of a current of hydrogen, and recording each minute the volume of water separated. This is done at 20°, 50°, and 80°, and the oil with the best demulsibility at all temperatures is considered to be the one likely to give best results for steam-turbine lubrication. With the oils examined, origin, viscosity, and density (with the exception of two oils, $d > 0.900$) had no relation to the classification of oils by demulsibility. Certain anomalies were noted in which the rates of separation were greater at the lower temperatures than at the higher, and this was attributed to influences of the method and degree of refining.

H. S. GARLICK.

Water-miscible mineral oil preparations. R. HART (Ind. Eng. Chem., 1929, 21, 85—90).—The manufacture of clear water-miscible or "soluble" oils is

primarily a problem of miscibility, and free oleic acid is essential to a uniform product. Miscibility curves for several emulsifiers, mineral oil, and oleic acid are given, by means of which uniform and non-uniform mixtures can be traced. Free oleic acid decreases the stability of the emulsion and may even prevent it. A number of terms are defined which simplify investigation of soluble oils, and make for greater convenience in comparing them. The kind and quantity of mineral oil have practically no effect on the quantity of oleic acid that the mixture will tolerate and still give good emulsions. This factor, however, varies with the kind of emulsifier, and even with the same emulsifier provided it is subject to adjustments. Alcohol has the following effects: (a) acts as liquefier for the soap; (b) at first decreases, then increases, the quantity of free oleic acid required for a homogeneous mixture; and (c) exerts no direct effect on the emulsion. The addition of alkali to a soluble oil containing acid sulphonated oil as the emulsifier yields the following results: (a) in the absence of alcohol, it at first decreases then increases the quantity of free oleic acid required for a homogeneous product; (b) in the presence of sufficient alcohol, the more alkali is present the less oleic acid is required to clear; and (c) the more neutralised is the sulphonated oil the better it functions as emulsifier, the completely neutralised oil being the best in this respect. Methods of testing raw materials and the stability of soluble oils based on the above results are developed.

H. S. GARLICK.

Metallic colloids and knock suppression. H. L. OLIN and W. J. JEBENS (Ind. Eng. Chem., 1929, 21, 43; cf. B., 1927, 66).—Further experiments showed that nickel sols prepared by heating nickel carbonyl in boiling hexane increased the highest useful compression ratio (H.U.C.R.) of gasoline to nearly the same extent as undecomposed nickel carbonyl, but that by increasing the temperature of decomposition to 85° the improvement becomes negligible. Lead tetraethyl behaves similarly, the lead sol produced at 250° giving an increase in H.U.C.R. of 7.5%, but that prepared at 300° shows no increase.

H. S. GARLICK.

The double linkings in vaseline, especially in that used for smokeless powders. R. POGGI (Giorn. Chim. Ind. Appl., 1928, 10, 601—605).—Application to a number of unsaturated compounds, such as amylene, allyl alcohol, oleic acid, cinnamic acid, anethole, dihydrocarvone, and terpineol, of the methods of Hanus (B., 1901, 1246) and Rosenmund and Kuhnnehn (B., 1924, 23; 1925, 214) for determining the iodine or bromine values of fats and oils shows that the latter method gives results in the better accordance with the calculated values. Tests made with vaselines from various sources show, in general, moderately good agreement between the two methods, but for Hellfrisch's vaseline and Kahlbaum's white vaseline the Hanus method gives, respectively, 82.2 and 85.2 mg. of bromine per 1 g., whereas the Rosenmund method gives 0.7 and 8.1. Certain qualities of commercial vaseline are found to contain very small proportions of compounds with double linkings.

T. H. POPE.

Determination of sulphur in benzol. W. B. DAVIDSON (Gas J., 1929, 185, 95—96).—The benzol is diluted with four times its volume of sulphur-free methylated spirit, and 10 c.c. of this mixture are burned in a special burner, during $1\frac{1}{2}$ —2 hrs. Air for combustion is purified by passing through a scrubber moistened with caustic soda solution, and the combustion products are drawn up a chimney of Pyrex glass, from which they pass to a perforated bulb which dips below the surface of a solution of neutral hydrogen peroxide. The neck of the flask, by which the gases escape, is filled with broken glass moistened with the same liquid. The contents of flask and scrubber are titrated with 0.1N-sodium carbonate to determine sulphuric acid. The method can be applied to paraffin or lubricating oils, or to petrol by suitable modification.

R. H. GRIFFITH.

Synthetic ammonia plant. PALLEMAERTS.—See VII. **Protection of underground pipes.** SLATER.—See X. **Colour of lamp blacks.** HOCK.—See XIII.

PATENTS.

Coke ovens. KOPPERS CO., Assees. of J. BECKER and J. VAN ACKEREN (B.P. 278,012, 26.9.27. U.S., 27.9.26).—Steam jets are provided in the oftakes from high-chambered regenerative coke ovens so that a forced draught can be applied therein while charging the ovens. By this means all volatile matter evolved during the charging may be collected, and at the same time a regulated pressure above atmospheric may be maintained in the whole gas-collecting main system. Any of the ovens may then be charged without subjecting the remainder to the danger of damage caused by drawing air through the floors of the coking chambers. In the arrangement preferred, each oven is provided with an oftake on either side of a central charging opening.

A. B. MANNING.

Carbonising and gasifying pulverised coal and treating ores. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 277,660, 12.9.27. U.S., 14.9.26).—Finely-pulverised coal is heated to a temperature just below the fusion point, maintained at that temperature until practically no more volatile matter is evolved, and then heated to a higher temperature in order to complete carbonisation. Under these conditions no agglomeration or fusion of the material occurs. The coal, reduced to a fineness of at least 100-mesh, passes down narrow vertical tubes within a retort, up through the space surrounding the tubes, and down through an annular chamber to the outlet. The volatile products of carbonisation travel with the material until it passes into the annular chamber; the vapours and gases then pass into an upper compartment and thence to suitable outlets. The retort is heated by a fire box at the base, the hot gases from which pass up through tubes arranged concentrically within those down which the coal travels. By controlling the rate of passage of the coal and the temperature of the fire box the rate of heating is arranged to fulfil the desired conditions. The process may be used for heating ores or other materials.

A. B. MANNING.

Gas producers. W. M. CROSS (B.P. 289,491, 28.4.28. U.S., 28.4.27).—Pulverised fuel, together with a mixture

of air and steam in such proportions and at a sufficiently high initial temperature to give on interaction with the fuel producer gas of any desired calorific value, is injected tangentially into the upper part of a vertical producer. The air and steam are preheated by means of hot waste gases or by the producer gas itself, and are then further heated in a superheater, which is preferably of the oil-fired type. The temperature of the air-steam mixture is automatically controlled. A baffle above the gas outlet in the lower part of the producer separates impurities from the gas, which then passes through a dust separator to the holder.

A. B. MANNING.

Gas purifiers, scrubbers, and the like. R. W. BROADHEAD, and BROADHEAD CONSTRUCTIONS, LTD. (B.P. 301,440, 30.8.27).—Apparatus for handling gases which have a corrosive action on steel is constructed of steel which is faced internally with concrete, the latter being preferably reinforced where exposed to corrosive action.

A. B. MANNING.

Separation of benzene and similar hydrocarbons from coke-oven and like gas by compression and cooling. GES. F. LINDE'S EISMASCHINEN A.-G., and SOC. MÉTALL. DE SAMBRE & MOSELLE (B.P. 275,633, 3.8.27. Ger., 4.8.26).—The compressed gas is cooled in a first stage to 40° only, eliminating naphthalene, lubricant oils, and water, which are led away through conduits and valves heated above this temperature, without condensing the benzene. It is then expanded under such conditions that the ratio of the pressure before and after expansion lies between 3 and 6, e.g., 4 atm. before and 1.1 atm. after expansion, so that the main portions of benzene and water are separated out from the gas before it enters the expansion machine. Those parts of the apparatus in which separation of solid benzene takes place are duplicated for use while the other is heated to melt the benzene. Alternatively, the separated benzene may be held in solution in toluene, and the mixture separated utilising the heat of compression.

H. S. GARLICK.

[Treatment of gases from] destructive hydrogenation of coals, tars, mineral oils, etc. I. G. FARBERIND. A.-G. (B.P. 279,072, 6.10.27. Addn. to B.P. 254,713; B., 1927, 644).—The process described in the main patent is modified by effecting the decomposition of the gas with carbon dioxide, either alone or in association with steam or other gases not richer in oxygen than air. The mixed gases are passed over heated refractory masses impregnated, if desired, with catalysts.

A. B. MANNING.

Treatment of the gaseous products liberated in the carbonisation of marine algæ. G. J. B. CHAMAGNE (B.P. 284,583, 11.8.27. Fr., 11.8.26).—The products evolved in the carbonisation of marine algæ in the furnace described in B.P. 275,998 (B., 1928, 325) are subjected to electrical precipitation, e.g., by arranging a high-tension electrical conductor axially in the smoke stack. The condensate is collected and treated for the recovery of its constituents, or is used directly, e.g., as an insecticide.

A. B. MANNING.

Distillation of tar, oil, and other materials. INTERNAT. COMBUSTION ENGINEERING CORP., Assees. of W. RUNGE (B.P. 295,945, 6.9.27. U.S., 21.9.26).—The

tar or oil is vaporised in a still of the pattern described in B.P. 277,952 (B., 1929, 116), and is then fractionally condensed. Tubular condensers are used, their temperatures being controlled by passing the hot waste gases from the still, cooled by the addition of the requisite amount of air, through the spaces about the tubes. The waste gases traverse the condensers in succession, the amount of air to be added thereto before entering each condenser being automatically regulated by means of a suitable thermostatic device. The last condenser, however, is water-cooled. A. B. MANNING.

Cracking of hydrocarbon oil. C. ARNOLD. From STANDARD DEVELOPMENT CO. (B.P. 302,211, 12.9.27).—Using a plant similar to that of B.P. 274,763 (B., 1927, 741), the oil to be cracked is first passed into the heated still, whence the vapours pass into the fractionating zone where light vapours are separated. The cracking coil and digester are used for cracking either a condensate from the fractionating tower or residual feed oil from the still. The contents of the digester may be discharged either into the still or into the fractionating tower, and a fraction from the tower may be returned to the still. H. S. GARLICK.

Conversion of hydrocarbon oils into lighter oils. C. ARNOLD. From STANDARD DEVELOPMENT CO. (B.P. 302,415, 22.9.27).—In the process of cracking oils under pressure by passing them rapidly through a heater coil and then into a large thermally insulated chamber where the oil remains for a substantial time at cracking temperature, the coil is operated at a temperature considerably higher than that desirable in the chamber, but excessive rise of temperature therein is prevented by adding a relatively cool oil to the hot oil at a point immediately before the hot oil enters the digestion chamber, whereby the temperature is controlled and the added oil brought up to cracking temperature. The cooling oil may be a diverted portion of the oil stream passing to the coil, or an oil adapted for cracking in the chamber, but not in the coil, and/or a condensate. H. S. GARLICK.

Manufacture of hydrocarbons. J. A. A. ZACON (B.P. 301,949, 7.9.27).—An aqueous emulsion of a mixture of a powdered and purified carboniferous product is continuously injected into a reaction apparatus in which it is exposed at 150 atm. and 300° to the action of an electric spark or arc, with or without the presence of hydrogenation catalysts. The products are expanded, cooled, and condensed, and the crude hydrocarbon mixture is fractionated. H. S. GARLICK.

Manufacture of valuable hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,253, 9.6.27).—Carbonaceous materials which contain substantial amounts of non-aromatic hydrocarbons are treated with an excess of hydrogen at temperatures above 550° and at pressures of at least 20 atm., preferably in the presence of catalysts, so that hydrocarbon mixtures are formed which boil below 200° and consist mainly of aromatic, unsaturated aliphatic, and hydro-aromatic hydrocarbons. The catalysts used, *e.g.*, chromium oxide, alone or admixed with zinc, manganese, or aluminium oxides, etc., are so chosen that the cracking action predominates over the hydrogenating action. A. B. MANNING.

Purification of petroleum hydrocarbons. A. G. BLOXAM. From ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 301,955, 9.9.27).—A diminished sulphur content and an improved odour of petroleum distillates are obtained by subjecting them to extraction with liquid sulphur dioxide and treating the remaining insoluble portion (after removal of sulphurous acid) with a dilute alkaline solution of lead oxide, with or without the addition of small quantities of elemental sulphur.

H. S. GARLICK.

Separation of gaseous or low-boiling hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,203, 8.9.27).—Cracked gases, especially olefines, liquefied if necessary, are separated into their constituents by fractional distillation under pressure preferably through several columns maintained at different temperatures and successively lower pressures. The separation of ethylene, propylene, and butadiene is described. C. HOLLINS.

Separation of paraffinous constituents etc. from [mineral] oils. AKTIEBOLAGET SEPARATOR-NOBEL, Assecs. of AKTIEBOLAGET SEPARATOR (B.P. 276,658, 13.8.27. Swed., 24.8.26).—To a mixture of oil and paraffinous constituents in which the oil is the lighter constituent a heavier component is added which renders the material to be separated lighter than the oil mixture. The oil is separated in a centrifuge in which the chamber in which the lighter component collects is provided with a mechanical transporting device. By this means the separated material is positively and mechanically transported to the outlet continuously during the running of the centrifuge. H. S. GARLICK.

Decolorising or bleaching montan wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,626, 15.9.27).—Finely-divided crude or deresinified montan wax is suspended in a warm solution of a chromic salt. The requisite amount of chromic acid to effect bleaching is then run in and the temperature raised to above 100°, simultaneously evaporating water and separating the bleached wax from the spent solution. The wax is allowed to remain in a warm state until complete separation from emulsified solution has taken place.

H. S. GARLICK.

Lubricating or like oils. H. L. CALLENDAR, R. O. KING, and E. W. J. MARDLES (B.P. 295,230, 4.5.27).—Partial decomposition and oxidation of mineral and vegetable lubricating oils may be prevented or hindered by the addition of a completely oil-soluble lead aryl or alkyl, with, if necessary, a small proportion of an organic halogen compound. Suitable proportions are 0.25–2% by vol. of lead tetraethyl. When the halogen compound is used it may be added, *e.g.*, as 2 pts. by vol. of the lead tetraethyl to 3 pts. of ethylene dibromide. E. HOLMES.

Method and apparatus for burning finely-divided fuel. PEABODY ENGINEERING CORP., Assecs. of J. P. LEASK and H. T. DYER (B.P. 286,293, 18.2.28. U.S., 4.3.27).

Pulverised fuel burners. INTERNAT. COMBUSTION, LTD., and F. II. ROSENCRANTS (B.P. 302,195, 6.9.27).

Gas burners. SOUTH METROPOLITAN GAS CO., D. CHANDLER, and J. J. COOKE (B.P. 302,532, 11.4.28).

Incandescence gas burners. A. J. FALKNER (B.P. 294,827, 6.12.27).

Process and device for burning heavy oils. W. OSTHOFF (B.P. 303,300, 10.3.28).

Oxidation of organic compounds (B.P. 303,268).—See III. **Nitrogenised metallic compounds** (B.P. 277,714—5). **Hydrogen** (U.S.P. 1,689,858 and B.P. 276,687).—See VII. **Pickling of iron etc.** (B.P. 293,701).—See X. **Mineral oil derivatives** (U.S.P. 1,694,461—2).—See XII. **Medicinal paraffin** (B.P. 302,954).—See XX.

III.—ORGANIC INTERMEDIATES.

Synthesis of methyl alcohol. E. AUDIBERT (Chim. et Ind., 1928, 20, 1015—1022).—A résumé of the work carried out by the author on the synthesis of methyl alcohol from carbon monoxide and hydrogen under high pressure and presented at the Second Conference on Bituminous Coal, Pittsburg, 1928. The conditions of experiment throughout were: pressure 150 atm., rate of circulation of gases over catalysts 5000 m.³ (N.T.P.) per 1 m.³ of catalyst volume, and gas mixtures containing hydrogen and carbon monoxide in the ratio 5 : 1 (with catalysts having copper as the chief constituent), and 2 : 1 (with those of which zinc oxide is the basis), yields of methyl alcohol are expressed in g. per litre of catalyst volume per hour. It was found that in addition to the physical changes which occur in a catalyst as a result of prolonged use even with carefully purified gases, in the case of copper catalysts a "fouling" of the solid took place owing to the deposition of carbon compounds of high mol. wt. The rapidity of fouling is a function of the partial pressure of carbon monoxide in the gas, and it is reduced to an unimportant value when the proportion does not exceed one sixth. Zinc oxide catalysts do not foul with gases containing 33% of carbon monoxide; such a proportion, in fact, gives the optimum yields of methyl alcohol. When zinc oxide and copper catalysts are heated in hydrogen at, say, 550° for a number of hours, each suffers a loss of activity, that of the zinc oxide catalyst being the greater. The activity of a copper catalyst is completely removed by the addition of small amounts of the oxides of tin and bismuth; those of boron, titanium, and vanadium have no immediate effect, although they accelerate the rate at which prolonged heating reduces the activity. Aluminium and cerium oxides, on the other hand, increase the activity. The addition of beryllium and chromium oxides to a zinc oxide catalyst increases the activity, the presence of tungsten oxide affects it adversely, whilst uranium oxide is without effect. The yields of methyl alcohol obtained with each catalyst under the conditions specified above at temperatures corresponding to the optimum amounts are compared in the cases of copper and zinc oxide catalysts. With copper at temperatures not exceeding 275° the yield was 1100—1150 g., and with zinc oxide at 375°, 300—325 g. The effect of the pressure (*p* in atm.) on the yield, *R*, for a copper catalyst is represented approximately by $R = 0.050p^2$, and for zinc oxide by $R = 0.014p^2$. When the rate of flow over a zinc oxide

catalyst is increased from 5000 to 10,000 the yield rises by 55%, and from 10,000 to 20,000 by 35%. Small amounts of sulphur present as carbon disulphide or thiophen in an otherwise pure gas mixture poison the copper, but are without effect on the activity of the zinc oxide catalysts. Besides the desired reaction which produces methyl alcohol, a number of side reactions occur which result in the formation of water, carbon dioxide, and methane. From a study of the relative rates of the main and side reactions it is shown that the useful conversion of carbon monoxide is much greater with a copper catalyst than is the case with one of zinc oxide. In deciding the relative value of the two types of catalyst, the poor yields from zinc oxide have to be considered in conjunction with the robustness and indifference to small traces of poisons, whilst the extra yields from copper catalysts have to be off-set against the much greater sensitiveness to poisoning.

H. INGLESON.

Analysis of mixtures containing acetone, ethyl alcohol, and isopropyl alcohol. C. A. ADAMS and J. R. NICHOLLS (Analyst, 1929, 54, 2—9).—Tables are given of the immersion refractometer readings of various concentrations of acetone and some of the lower alcohols, and it was found that in aqueous mixtures of such substances, provided the total proportion does not exceed 10% by vol., the apparent proof strength and the refraction of the mixture are practically the sum of those due to each ingredient, and up to about 17% of apparent proof spirit the sp. gr. and refractions may be regarded as additive factors. A rapid method for the detection and colorimetric determination of acetone is given, based on Penzoldt's method (Z. anal. Chem., 1885, 24, 149), in which indigo is formed as a condensation product when sodium hydroxide is added to a mixture of *o*-nitrobenzaldehyde and acetone. *iso*Propyl alcohol may be determined by oxidising with bromine water to acetone, and proceeding as for acetone, and for a mixture of acetone, ethyl alcohol, and *isopropyl* alcohol, the conditions for complete oxidation of each of these constituents are given, and a method for their determination.

D. G. HEWER.

Sp. gr. and immersion refractometer readings of dilute mixtures of acetone and water. J. R. NICHOLLS (Analyst, 1929, 54, 9—11).—Two samples of acetone were prepared: (a) by carefully fractionating commercial acetone, distilling twice from dichromate and sulphuric acid, shaking the distillate with anhydrous potassium carbonate, again distilling, shaking the constant-boiling fraction with fused calcium chloride for several days, refractionating, and repeating the process until the sp. gr. was constant; (b) by similarly fractionating and drying a sample prepared from the sodium iodide compound. Weighed quantities of each were diluted with water to 100 c.c. and the sp. gr. and refractometer readings taken, and from the plotted results the equivalent proof strength and refractometer readings are tabulated for 0 to 10 g. of acetone per 100 c.c.

D. G. HEWER.

Routine catalytic preparations in an organic course. S. GOLDSCHMIDT and L. ORTHNER (Z. angew.

Chem., 1929, 42, 40—42).—Apparatus and analytical methods suitable for students' use are described and illustrated with reference to acetone from acetic acid over iron turnings, methyl or ethyl acetate or *iso*amyl propionate from acid and alcohol, aniline from nitrobenzene, acetaldehyde from alcohol, keten from acetone, and ethylene from alcohol. C. HOLLINS.

Technical importance of adipic acids and their derivatives. W. SCHRAUTH (Chem.-Ztg., 1929, 53, 41—43).—Attention is drawn to possible technical applications of adipic acid, now rendered readily accessible by new methods for oxidation of *cyclohexanols*. It is completely non-toxic, and is an advantageous substitute for tartaric or phosphoric acid in baking-powder. The more soluble β -methyladipic acid is suitable for lemonade crystals. Various applications of the acids in textile, leather, and dyeing industries are mentioned. The esters, *e.g.*, methyl*cyclohexyl* β -methyladipate ("Sipalin MOM"), are used as plasticisers for cellulose esters, and in printing of textiles, whilst with glycerol etc. adipic acids give resins. The sodium salts of acid adipates of higher alcohols are powerful emulsifying and wetting-out agents, and the neutral higher esters are fixatives for perfumes. C. HOLLINS.

[Preparation of] derivatives of *s-m*-xlenol. E. NOELTING (Sealed Note 2333, 4.1.15. Bull. Soc. Ind. Mulhouse, 1928, 94, 648—649). Report by M. BATEGAY (*Ibid.*, 650—651).—Brief reference is made to the preparation of a number of substances from 5-hydroxy-*m*-4-xylyl methyl ketone and 2-amino-*m*-5-xlenol. A brown sulphur dye was obtained by treatment of the last-named substance with sodium sulphide and sulphur, and a green dye by condensation with tetramethyldiaminobenzhydrol and subsequent oxidation. Battagay draws attention to a number of other researches relating to similar substances.

A. J. HALL.

Potentiometric and conductometric analysis. CALLAN and HORROBIN.—See I. **Carboxylic acids in peat tar** (STADNIKOV and SABAWIN). **Pityrol.** KOMATSU and others. **ψ -Cumene derivatives in wood spirit.** HOLMBERG.—See II.

PATENTS.

Catalytic preparation of oxygenated carbon compounds. E. I. DU PONT DE NEMOURS & Co., Assees. of W. A. LAZIER (B.P. 272,555, 13.6.27. U.S., 12.6.26).—For the hydrogenation of carbon oxides a chromite catalyst is used, obtained, *e.g.*, by heating a chromate or dichromate of zinc, copper, cadmium, magnesium, manganese, silver, or iron at 800—900°.

C. HOLLINS.

Vaporisation of formamide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,974, 17.9.27).—Formamide is vaporised with only slight decomposition by bringing it, preferably in a finely-divided condition, into contact with surfaces heated above its b.p. so that it is vaporised immediately without accumulating in liquid form in the vaporiser. B. FULLMAN.

Manufacture of solid polymerised formaldehyde. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,258, 2.12.27).—Aqueous formaldehyde, preferably over 30%, is treated with sodium carbonate (0.5 pt.

per 100) below 50° in absence of gelatinising agents such as alkaline soaps. With more dilute formaldehyde solutions cooling below 0° is necessary. A filterable product is obtained.

C. HOLLINS.

Production of acetaldehyde. H. S. HIRST, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 302,515, 25.1.28).—Gases poor in acetylene, *e.g.*, those derived from methane by thermal decomposition, are scrubbed with a countercurrent of dilute sulphuric acid containing mercuric and ferric sulphates at 60—70° and at pressures up to 10 atm. Acetaldehyde and a gas richer in hydrogen are produced. A continuous process is described.

C. HOLLINS.

Catalytic oxidation of organic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,268, 21.12.27).—Organic compounds (*e.g.*, mineral oil, neutral oxidation products of higher paraffins, paraffin wax) are oxidised in the liquid phase by air in presence of manganese acetylacetone or other metal enolates.

C. HOLLINS.

Production of aliphatic ketones. HOLZ-VERKOHLUNGS-IND. A.-G., and K. RÓKA (B.P. 302,759, 18.10.27).—Alcohols having at least two carbon atoms, the corresponding aldehydes, or esters are converted into acetone or other ketones by passage with steam at about 500° over a catalyst made, for example, by evaporating a stirred mixture of calcium acetate solution and rusty iron shavings. Acetaldehyde, ethyl alcohol, or ethyl acetate yields acetone.

C. HOLLINS.

Manufacture of isothiocarbamide ethers. SCHERING-KAHLBAUM A.-G. (B.P. 296,782, 28.10.28. Ger., 9.9.27).—Cyanamides are treated with thiol derivatives in aqueous or alcoholic solution in presence of an acid, or the vaporised thiol may be passed into the cyanamide solution. With disubstituted cyanamides the reaction is effected at 100°, dimethyl- or diethyl-cyanamide and methyl mercaptan yielding, *e.g.*, NN-dimethyl- or -diethyl-S-methylisothiocarbamide. R. BRIGHTMAN.

Manufacture of substituted guanidines. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,044, 26.9.27).—Thiocarbanilide, or other substituted thiocarbamide, is treated with a zinc compound and an alkali or alkaline-earth in presence of ammonia or a substituted ammonia. Thiocarbanilide with aniline gives triphenylguanidine, with benzylamine *diphenylbenzylguanidine*, m.p. 102—103°; the methyl- (m.p. 108—109°) and dimethyl-compounds (m.p. 199°) are similarly obtained. With phenylhydrazine *anilindiphenylguanidine*, m.p. 160°, and its oxidation product, an azo compound, m.p. 111°, are produced, whilst phenylhydroxylamine yields N-hydroxytriphenylguanidine, m.p. 154°.

C. HOLLINS.

Manufacture of higher alkylated guanidine derivatives. SCHERING-KAHLBAUM A.-G. (B.P. 279,884, 27.10.27. Ger., 28.10.26).—Cyanamide, an alkyl cyanamide, or a monoalkal salt of cyanamide in slight excess is treated, *e.g.*, in aqueous or alcoholic solution, with the salt of an alkylamine, the alkyl group or groups of which together contain more than four carbon atoms. *isoAmylguanidine sulphate*, m.p. 266° (decomp.), *hexylguanidine*

sulphate, m.p. 255°, heptylguanidine, pentamethylenediguanidine sulphate, decomp. above 320°, diguanylhexamethylene sulphate, decamethylenediguanidine sulphate and hydrochloride, N-methyl-N'-hexylguanidine picrate, and amyguanidine sulphate, m.p. 130°, are described.

R. BRIGHTMAN.

Recovery of volatile organic solvents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,297, 12.9.27).—The vapours containing the solvent to be recovered are scrubbed in a countercurrent of products, b.p. above 200°, obtained by the catalytic hydrogenation of oxides of carbon.

C. HOLLINS.

Preparation of sodium nitrogen compounds. DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 293,040, 26.6.28. Ger., 1.7.27).—Sodium hydride reacts with arylamines, acid amides or imides, etc. to give N-sodio-derivatives, often at temperatures below 100°. Examples are aniline, methylaniline, p-chloroaniline, naphthylamines, diphenylamine, acetamide, carbamide, guanidine, phthalimide, succinimide, diacetamide, phenylthiocarbamide, carbazole, aminotriazole, and aminothiazole. For acid amides a solvent is necessary to moderate the reaction.

C. HOLLINS.

N-Aminoalkylation of amines. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,401, 24.5.27).—Amines are heated with the hydrochloric acid salts of aminoalkyl p-toluenesulphonates. Aniline and the ester hydrochloride of ε-diethylaminopentan-β-ol give ε-diethylamino-β-amyraniline, b.p. 150—154°/6 mm. From 8-aminoquinoline and the γ-ester hydrochloride of N-β-γ-dihydroxypropylpiperidine 8-(β-hydroxy-1-γ-piperidylpropylamino)quinoline, b.p. 212—213°/1 mm., is obtained.

C. HOLLINS.

Manufacture of aromatic mercaptans [thiophenols]. I. G. FARBENIND. A.-G. (B.P. 279,136, 18.10.27. Ger., 18.10.26).—Diazo compounds, not containing solubilising groups, are converted smoothly into thiophenols or thionaphthols by treatment with a metal polysulphide and subsequent reduction of the disulphide so formed. The polysulphide must contain more sulphur than is required by the formula M₂S₂ or M'S₂. The preparation of 5-chloro-o-thiocresol is described.

C. HOLLINS.

Manufacture of alkylated phenols and their hydrogenated products. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 274,439, 17.6.27. Ger., 14.7.26. Addn. to B.P. 254,753; B., 1928, 740).—The process of the prior patent is applied to the condensation products of aldehydes (formaldehyde, acetaldehyde, or benzaldehyde) to give phenol and p-cresol, phenol and p-hydroxyethylbenzene, or phenol and p-hydroxydiphenylmethane, or hexahydro-derivatives of these.

C. HOLLINS.

Manufacture of anthraquinone derivatives. I. G. FARBENIND. A.-G. (B.P. 282,004, 9.9.27. Ger., 9.12.26).—2:3-Benzbenzanthrone, m.p. 229°, is oxidised with chromic-acetic acid to 1-o-carboxyphenylanthraquinone, m.p. 236°; the dichloro-derivative (from 2:6-dichloroanthraquinone) behaves similarly.

C. HOLLINS.

Reduction of nitroanthraquinones. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 302,964, 22.9.27).—

Nitroanthraquinones are smoothly reduced when heated with tetrahydro-quinoline or -quinaldine in a solvent (pyridine, quinoline, quinaldine, o-dichlorobenzene, acetic acid, etc.). At moderate temperatures (125°) the nitro-groups are reduced to the hydroxylamino-stage, at higher temperatures to the amino-stage.

C. HOLLINS.

Manufacture of aminated sulphurised [i.e., sulphur-containing] benzanthrone derivatives. I. G. FARBENIND. A.-G. (B.P. 275,271, 2.8.27. Ger., 2.8.26).—Benzanthronyl sulphides, disulphides, thiol compounds, thio-ethers, etc. are heated with hydroxylamine and sulphuric acid in presence of ferrous or copper sulphate to give amino-derivatives. Amino- and diamino-3:3'-dibenzanthronyl sulphides, a bromo-derivative of the former, and an aminated oxidation product of dibenzanthronyl sulphide are described.

C. HOLLINS.

Separation of hydrocarbons (B.P. 302,203).—See II. **Diazo compound and dyes therefrom** (B.P. 288,572). **Stable diazo compounds** (B.P. 280,945). **Azo intermediates** (B.P. 302,770 and 302,773).—See IV.

IV.—DYESTUFFS.

Electrochemical reduction of azo dyes to their respective amino-compounds. L. H. HUBBUCH [with A. LOWY] (Amer. Electrochem. Soc., May, 1929. Advance copy. 13 pp.).—Six azo dyes, Methyl Orange, Metanil Yellow, Acid Scarlet, Crimson, Congo Red, and Fast Brown O, representing six different types, have been reduced electrolytically to the corresponding pairs of amino-compounds in a sodium carbonate solution, using a mercury cathode. The successful reduction of Methyl Orange and Metanil Yellow indicates that the statements of Stohr and Schneider, that only azo compounds having an amino- or a hydroxyl group in the o- or p-position to the azo group can be electrolytically reduced to amines, are erroneous. The conditions generally most favourable for the reduction are: 95°, 0.885 amp./dm.², and 7.5 g. of dye and 7.5 g. of sodium carbonate in 200 c.c. of solution. Current efficiencies obtained were often above 80% under these conditions.

H. J. T. ELLINGHAM.

Potentiometric and conductometric analysis. CALLAN and HORROBIN.—See I. **Derivatives of s-m-xyleneol.** NOELTING; also BATTEGAY.—See III.

PATENTS.

Manufacture of condensation products of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 275,636, 4.8.27. Ger., 4.8.26).—Aminoanthraquinones are condensed with chlorohydrins to give dyes for acetate silk, oils, fats, fatty acids, etc. Examples are: 1-β-hydroxyethylaminoanthraquinone, m.p. 148—150° (red); 1-β-γ-dihydroxypropylaminoanthraquinone, m.p. 192° (red to orange-red); 1-β-hydroxyethylamino-4-hydroxyanthraquinone (violet); 1:4-di-(β-hydroxyethylamino)anthraquinone (blue); 1:5-isomeride (bluish-red).

C. HOLLINS.

Manufacture of dyes of the anthraquinone series. L. CASSELLA & Co. G.M.B.H. (B.P. 295,600, 13.8.28. Ger., 11.8.27. Addn. to B.P. 260,998; B., 1928, 225).—

Chlorination of anthraquinone proceeds smoothly in 90% sulphuric acid at 45–50° in presence of a chlorine carrier. The process can be conveniently combined with the cyclisation of 1:1'-dinaphthyl-8:8'-dicarboxylic acid in monohydrate, which may be diluted to 90% for chlorination without isolation of the anthranthrene.

C. HOLLINS.

Manufacture of brown vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,489, 5.12.27. Addn. to B.P. 289,980; B., 1928, 517).—The brown vat dye of the prior patent is reduced to leuco-compound and halogenated in presence or absence of catalysts to give, after re-oxidation, fast brown vat dyes. The $\cdot\text{N}:\text{N}\cdot$ group is probably converted into $\cdot\text{NH}\cdot\text{NH}\cdot$.

C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. SOC. CHEM. IND. IN BASLE (B.P. 279,479, 20.10.27. Switz., 20.10.26).—A dibenzanthrone is sulphonated, fused with alkali, and finally alkylated and/or halogenated, to give bluish-grey vat dyes.

C. HOLLINS.

New vat dyes of the dipyrazolanthrone series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 302,772, 31.10.27).—Dipyrazolanthrone or one of its derivatives is condensed with aldehydes in nitrobenzene, in presence of a chloride of phosphorus, antimony, or aluminium. Dipyrazolanthrone with formaldehyde, acetaldehyde, benzaldehyde, *o*- or *m*-chlorobenzaldehyde, *m*-nitrobenzaldehyde, or *m*-hydroxybenzaldehyde, dichlorodipyrazolanthrone with benzaldehyde, tetrabromodipyrazolanthrone with benzaldehyde, and ethyldipyrazolanthrone with formaldehyde give yellow to orange vat dyes.

C. HOLLINS.

Manufacture of a new diazo compound and of new dyes therefrom. COMP. NAT. MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES, ÉTABL. KUHLMANN (B.P. 288,572, 23.3.28. Fr., 12.4.27).—Diazotised 4-chloro-*o*-nitroaniline is treated with an alkali (sodium acetate, carbonate, etc.) to give 4-chlorobenzene-1:2-diazo-oxide, which is coupled with naphthols, aminonaphthols, pyrazolones, sulphonic acids of these, or resorcinol to yield chromable dyes. The latter may be converted into copper or chromium complex salts.

C. HOLLINS.

Manufacture of stable diazo-compounds. SOC. CHEM. IND. IN BASLE (B.P. 280,945, 18.11.27. Switz., 18.11.26. Addn. to B.P. 238,704; B., 1925, 840).—Diazo solutions (other than those from aminoazo compounds) are evaporated with sodium 1:3:6- or 1:3:7-naphthalenetrisulphonate to give stable diazo preparations, to which a little sodium hydrogen carbonate may be added to aid re-dissolution if desired. Diluents such as salt, sodium sulphate, or aluminium sulphate may be added to the diazo solution before evaporation.

C. HOLLINS.

Manufacture of [azo] dyes containing chromium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,709, 15.9.27).—Sulphoarylpyrazolones are used in place of the sulphonamide derivatives of B.P. 210,669 (B., 1924, 412), and give valuable dyes when coupled, for example, with diazotised 1:2:4-aminonaphthol-sulphonic acid and pre-chromed.

C. HOLLINS.

Manufacture of new *o*-hydroxyazo dyes and an intermediate product. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 302,770, 28.10.27).—2:6-Hydroxynaphthoic acid is sulphonated and the product is fused with alkali to give 2:8-dihydroxy-6-naphthoic acid, which when coupled with diazotised *o*-aminophenols yields brown wool dyes becoming brown to black on chroming and having good milling fastness. Examples of diazo components are 4-chloro-*o*-aminophenol, 4-nitro-*o*-aminophenol, 5-nitro-3-aminosalicylic acid, picramic acid, 5-nitro-3-amino-*p*-cresol, 4-chloro-6-nitro-*o*-aminophenol, *o*-aminonaphtholsulphonic acids, and *o*-amino-hydroxyazo compounds.

C. HOLLINS.

Manufacture of new azo dyes and intermediate products. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 302,773, 31.10.27).—Arylamides of 2-aryl-amino-8-hydroxy-6-naphthoic acids are coupled in substance or on the fibre with a diazo compound. 2-Anilino-8-hydroxy-6-naphthoic acid, obtained from 2:8-dihydroxy-6-naphthoic acid (B.P. 302,770, preceding) by the action of aniline and bisulphite, yields an *anilide*, decomp. 170°, *p*-anisidide, m.p. 160° (decomp.), and β -naphthylamide, m.p. 170–175° (decomp.). The anilide is coupled with diazotised *p*-chloroaniline (dark olive), 5-nitro-*o*-toluidine (dark olive), *o*-aminoazotoluene (brownish-black), *o*-phenetidine \rightarrow α -naphthylamine (black), *p*-nitroaniline-*o*-sulphonic acid (dark brown on wool), *m*-aminobenzoic acid (similar), the *p*-anisidide with 4-chloro-*o*-toluidine (brown-olive), 4-chloro-*o*-nitroaniline (grey-brown), 2:5-dichloroaniline (reddish-brown), 4:4'-diaminodiphenylamine (black); the β -naphthylamide with dianisidine (blue-black, suitable for lakes).

C. HOLLINS.

Manufacture of azo dyes containing chromium and their application. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (B.P. 301,772, 4.8.27).—Loss of dye in the form of insoluble complex compounds during pre-chroming is avoided by adding an organic salt and/or an organic acid, or an inorganic salt and an organic acid; if the addendum also serves as an acid-binding agent, excess is added. Examples are: dye, chromium formate, sodium acetate, with or without formic acid; dye, chromium formate, sodium chloride, and formic acid.

C. HOLLINS.

Manufacture of a black tetrakisazo dye. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,722, 29.8.27).—Benzidine is tetrazotised and coupled successively with 1 mol. of H-acid in acid solution and 1 mol. of *m*-phenylenediamine in alkaline solution, the resulting disazo dye being then coupled alkaline with 2 mols. of diazotised *p*-aminoacetanilide. On alkaline hydrolysis a tetrakisazo dye is obtained which gives greenish-black shades on chrome leather. Alternatively, *p*-nitroaniline may be used in place of *p*-aminoacetanilide and the tetrakisazo dye reduced.

R. BRIGHTMAN.

Preparation of triarylmethane dyes. BRIT. DYESTUFFS CORP., LTD., F. W. LITCH, and E. H. RODD (B.P. 301,193, 17.11.27).—Condensation of tetra-alkyldiaminobenzophenones with aromatic chloro-compounds (B.P. 272,321; B. 1927, 598) is effected at 60°

and in presence of benzene as solvent, giving higher yields and purer products. R. BRIGHTMAN.

Oxidation of leuco-compounds of the triaryl-methane series. I. G. FARBERIND. A.-G. (B.P. 299,473, 26.10.28. Ger., 28.10.27).—Leuco-compounds of triarylmethane dyes are oxidised with air in presence of a copper salt and pyridine or other cyclic tertiary base, with addition of a diluent (glycerol) if desired.

C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Tensile strength of abacá fibres in relation to their acidity. P. L. and H. E. SHERMAN (Philippine J. Sci., 1928, **37**, 21—40).—An examination of various samples of abacá fibre indicates that the higher is the natural acidity of the fibre the lower are its elasticity and tensile strength; the same relationship holds in the main for Canton fibres, but with less uniformity. The loss of strength of abacá during storage is also affected by the acid content, but no definite relationship was found between the rate of loss of tensile strength and the acid content determined at the end of the storage period.

D. J. NORMAN.

Fermentation as affecting the quality of Philippine abacá. T. BASUELOS and P. L. SHERMAN (Philippine J. Sci., 1928, **37**, 41—65).—Abacá fibre as commercially produced is heavily infected with bacteria; drying the fibre immediately after stripping causes a partial sterilisation, which remains effective so long as the fibre is kept dry, but should the drying process be omitted, or should the dried fibre be wetted again, fermentation sets in, the immediate results of which are increased acidity, and loss of colour and tensile strength. This deterioration appears to be due to acid fermentation of the soluble constituents of the fibre and to bacterial attack of the fibre itself. D. J. NORMAN.

Heartwood of the pine. I. Adsorption and infiltration experiments with sapwood and heartwood of pine and spruce. II. Extraction by organic solvents and alkalis. C. G. SCHWALBE and A. AF EKENSTAM (Cellulosechem., 1929, **10**, 1—11, 11—18).—I. The experiments included measurements of the rate and amount of adsorption of water vapour and of infiltration by water and various solutions, and were designed particularly for comparison of the heartwood and sapwood of the woods examined. It was found that pine heartwood in all circumstances absorbed less liquid than the other samples. In infiltration experiments with both pine and spruce the absorption of water by green sapwood was greater than that by green heartwood, but with increasing dryness the two values converged. Pine heartwood adsorbed more sodium hydroxide than spruce heartwood, but this difference disappeared when the former had been extracted with ether. In pine heartwood the sodium salt of an ether-soluble acid is formed, and partly retained by the wood. In sealed tubes at 110° pine heartwood showed no difference in ability to take up liquid, but it differed particularly in absorbing Mg^{++} ions to a relatively larger extent than HSO_3 ions from magnesium bisulphite solution, so that the composition of the absorbed salt

corresponded to that of a neutral sulphite. This, which is the chief cause of the failure of acid disintegration processes, is ascribed to comparatively slow diffusion of HSO_3 ions, whilst the Mg^{++} ions appear to react with an acid of high mol. wt. in the wood.

II. The amounts extracted by ether were in the order: pine heartwood > pine sapwood > spruce heartwood > spruce sapwood. Since pine sapwood can be disintegrated by the usual process, failure in the case of heartwood is considered to be due to a difference in the nature rather than in the amount of resinous substances present. Examination of the ether extracts showed that pine sapwood contained a larger proportion of fats (soluble in light petroleum) than the heartwood. The similarity of acid values indicated the presence of an acid of high mol. wt. in pine heartwood. The amount extracted by ether from pine heartwood decreased with time of storage, the fats decreasing most rapidly. Again, the residues obtained by evaporation of the extracts became insoluble when heated or kept. These effects are explained by the precipitation of colloidal constituents. Benzene extracted smaller amounts from pine heartwood than ether, but alcohol extracted larger amounts, including the fraction which had become insoluble in ether on storage. Pine heartwood yielded the largest amount of extract with 1% sodium hydroxide solution, and this was largely precipitated by hydrochloric acid. The precipitate was extracted with alcohol, and the alkaline solution fractionally precipitated by acid. Similar results were obtained with pine heartwood and sapwood. Large amounts of very weakly dissociated acids were present, and an acid with an insoluble calcium salt was isolated. Spruce, on the other hand, when similarly treated, yielded acids which were more strongly dissociated and precipitated within narrower limits of hydrogen-ion concentration. Comparison of the ether extracts of the substances precipitated from the sodium hydroxide extracts of pine heartwood and sapwood showed that the former contained a quantity of an acid with a soluble calcium salt which was lacking in the latter. Carbohydrates, compounds containing nitrogen or sulphur, or tannins could not be recognised in the extract of pine wood by 1% sodium hydroxide.

R. K. CALLOW.

Lignosulphonic acid obtained from spruce wood by the action of sulphurous acid in presence of ammonia. C. DORÉE and E. C. BARTON-WRIGHT (J.S.C.I., 1929, **48**, 9—12 τ).—The acid obtained by the above process of resolution due to Cross and Engelstad (*ibid.*, 1925, **44**, 267 τ), in which 0.1—0.5% of ammonia is present, has been isolated and its properties are compared with those of the acid obtained in the original process, in which sulphurous acid alone was employed. The acid has the formula $C_{40}H_{44}O_9 \cdot 2H_2SO_3$ and is present apparently as a monoammonium salt. It was purified by dialysis, conversion into a β -naphthylamine derivative, and resolution of this compound by means of pyridine. Experimental results are summarised in the dissected formula $C_{35}H_{33}O_2 \cdot CO \cdot CH(OH) \cdot (OH)_2 \cdot (OMe)_5 \cdot (SO_3H)_2$. On oxidation with nitric acid a nitro-compound, containing 2 carbonyl and 2 nitro-groupings, is formed. The nitro-compound is reduced by magnesium, the nitro-groupings being eliminated

and replaced by carbonyl groupings. In its reactions the acid closely resembles the acid obtained by the action of sulphurous acid alone, for which a formula $C_{26}H_{30}O_{12}S$ was found (Dorée and Hall, *ibid.*, 1924, 43, 257 r). The C_{40} -unit formula of the acid now described brings it into line with lignosulphonic acids isolated by previous workers.

Composition of commercial artificial silks. A. WAHL and J. ROLLAND (Rev. Gén. Mat. Col., 1929, 33, 1—4).—The moisture contents of commercial samples of viscose, hollow viscose, nitro- and cuprammonium silks, as determined after exposure of the material over sulphuric acid (at R.H. not stated) lies between 10 and 12%, whilst their ash content is 0.1—0.56%. The sulphur content, determined by ashing the finely-divided material in the presence of magnesium oxide and sodium and potassium carbonates, with subsequent dissolution of the ash and precipitation of the sulphate in the usual way, generally varies from 0.24 to 0.42%. The copper numbers (Braidy) of Tubize and of Charbonnet nitro-silk are approx. 3, those of viscoses, including the hollow variety Celta, 0.7—1.2, and of cuprammonium silk 0.5—0.7. Values for other kinds of cellulose material such as cotton and wood pulp, bleached to different extents, are included for comparison. The high values recorded, particularly for the nitro-silks and for some samples of viscose, are attributed to the presence of oxycellulose in the material, and suggestions are put forward to explain its formation.

B. P. RIDGE.

Viscosity in relation to cellulose acetates. M. DESCHIENS (Chim. et Ind., 1928, 20, 1023—1033).—The different methods of determining viscosity are explained and the range of their suitability is pointed out. In France, the official (Aeronautic) standard practice for cellulose acetate solutions is to employ 6 g. of the ester in 100 c.c. of solvent, and to compare the viscosity, in the same apparatus, with that of glycerin at d 1.26 and at 15° taken as 100. This practice is compared with that given in Brit. Eng. Stand. Assoc., No. 83, 1918. Relative values of viscosity of some cellulose acetates of commerce suitable for varnishes and dopes are given. The effect of variations in the proportions of the constituents of a dope on the viscosity has been examined. Acetates suitable for use as dopes or varnishes should be among the most rapidly soluble, and should have a viscosity when dissolved in acetone of 12—30. Solvents which possess the greatest dissolving power are those which give the most rapid dissolution, and also give solutions of the desired viscosity with the smallest volume of solvent. In general, such solutions are limpid, and have the least tendency to coagulate. The heterogeneity of cellulose esters of commerce can be readily demonstrated.

H. INGLESON.

Determination of the transparency of paper to light. W. HOLWECH (Papier-Fabr., 1929, 27, 37—45).—A method of determining the amount of light transmitted by paper and the absorption coefficient of the latter is described which involves the use of a polarisation photometer. The influence of the wave-length of the incident light, of increasing the number of layers of the same

paper, and of different kinds of paper, on the results obtained has been investigated.

B. P. RIDGE.

Bleaching cellulose. ESCOURROU.—See VI.

PATENTS.

Treating or retting flax fibre. M. WADDELL and H. C. WATSON (B.P. 302,300, 14.9.27).—The flax fibre in the form of a rove is immersed in water containing yeast at 21—27° with or without addition of sulphurous acid; it is finally treated with a tar product to prevent further fermentation.

F. R. ENNOS.

Degreasing of raw wool. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,941, 20.8.27).—Raw wool is treated with a mono- or di-chlorinated hydrocarbon, b.p. 175—200°, the solvent being subsequently removed from the wool by washing and aeration and from the wool fat by steaming.

F. R. ENNOS.

Manufacture of powdered plastic material. SOC. CHIM. DES USINES DU RHÔNE (B.P. 285,829, 31.1.28. Fr., 22.2.27. Addn. to B.P. 275,558; B., 1928, 330).—A mixture of cellulose ester or ether with a plasticiser is precipitated from solution by distillation of the solvent in the presence of a liquid which does not dissolve the material and which is miscible with and less volatile than the solvent. The precipitating liquid may be added to the solution of the plastic material before or during distillation of the solvent, or to the solvent itself before forming the solution.

F. R. ENNOS.

Manufacture of copper oxide-ammonia cellulose solutions for artificial silk production. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 302,956, 21.9.27).—Raw cellulose is pressed into thin paper-like sheets, avoiding unnecessary mechanical and chemical treatment, and owing to the large surface area dissolution is effected in 10—12% copper oxide-ammonia solution in about 1 hr.

W. G. CAREY.

Manufacture of ethers of carbohydrates. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 302,191, 9.8.27).—High-molecular carbohydrates, e.g., cellulose and starch, are treated with a gaseous alkylating agent, e.g., ethyl or methyl chloride, in the presence of moisture and less than 20 mols. of an alkali hydroxide per 1 mol. of carbohydrate, calculated as $C_6H_{10}O_5$. The carbohydrate may be kept in motion during the treatment, or may be treated with a current of the gaseous agent, e.g., by circulating it, alone or mixed with a non-reacting gas, successively through a heater, through the treatment chamber, and through a cooler to condense and remove water, whence it is returned to the circuit.

L. A. COLES.

Manufacture of aminocellulose derivatives. I. G. FARBENIND. A.-G. (B.P. 279,801, 14.9.27. Ger., 30.10.26).—Sodium cellulose is esterified with benzene- or toluene-sulphonyl chloride, and then acetylated or alkylated and finally treated with a primary, secondary, or tertiary amine; alternatively, the amine treatment may precede the acetylation or alkylation. The "animalised cellulose" so obtained, as also its mixtures with ordinary cellulose esters or ethers, has affinity for acid dyes. Amines used in the examples are aniline, diethylamine, pyridine, and isoamylamine.

C. HOLLINS.

Composition of matter containing a cellulose derivative. H. M. WEBER, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,690,515, 6.11.28. Appl., 13.10.25).—Glycerol, or other polyhydric alcohol, is heated at 235–265° with an organic acid and a vegetable oil, or a blown vegetable oil, or fatty acids. Thus, glycerol, phthalic anhydride, and tung oil give a resin, m.p. 73°, acid value 56, miscible with nitrocellulose, whilst glycerol, phthalic anhydride, and the fatty acids from soya-bean oil yield a similar resin, m.p. 71°, acid value 17.

R. BRIGHTMAN.

Manufacture of artificial silk by the dry-spinning method. SOC. FABR. SOIE "RHODIASETA" (B.P. 288,618, 14.9.27. Fr., 14.4.27. Addn. to B.P. 238,842; B., 1926, 627).—The spinning operation takes place in an evaporating atmosphere richly laden with the solvent vapour, the required concentration of which is maintained by a gaseous exchange between the atmosphere of the heated spinning cell and that of the cooled solvent condenser through a single large aperture connecting one with the other.

F. R. ENNOS.

Manufacture of rayon, artificial horsehair, films, etc. DU PONT RAYON Co., Assees. of W. H. BRADSHAW (B.P. 271,517, 23.5.27. U.S., 21.5.26).—Artificial silk of high wet strength is prepared by using as parent material a cellulose containing 98–100% of α -cellulose and conducting all subsequent operations under such conditions that the degree of hydration of the cellulose is not materially increased. Thus, in the manufacture of cuprammonium silk, purified cotton linters (99% of α -cellulose) is dissolved in cuprammonium hydroxide solution and the solution is beaten in a closed vessel with just sufficient air or oxygen to reduce the viscosity to the required degree without affecting the α -cellulose content. *E.g.*, the partial pressure of air in the mixer may be reduced to less than 4 in. of mercury, or the air may be suitably diluted with inert gases. When ready, the solution, containing, *e.g.*, 6% of cellulose, 5.5% NH_3 , and 2.5% Cu, is stretch-spun into 22–27% caustic soda solution at 0–14° and is preferably collected in a centrifugal spinning box. After keeping it for about 15 min. to complete the coagulation, the thread is passed through 20% sulphuric acid at 20° and wound on a rotating cylinder, the lower part of which is immersed in 2½% sulphuric acid at 20°. The speed of reeling and the length of travel of the thread in the 20% acid bath should be so adjusted that the acid carried over is sufficient to maintain the weak acid bath at a constant concentration. The thread is finally washed with water, drained, and dried.

D. J. NORMAN.

Cyclic process for manufacture of kraft pulp. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,689,534, 30.10.28. Appl., 4.12.25).—Raw cellulose material is digested in a liquor containing sodium sulphide. After separation of the pulp from the spent liquor, the latter is evaporated and the inorganic constituents are smelted in a reducing atmosphere, the sodium and sulphur present as sublimed solids and sulphur dioxide in the products of combustion being absorbed in more spent liquor, which is also evaporated and smelted. The recovered solids are dissolved in water and used for the digestion of other material, any deficiency of sulphur or sodium being made

up by addition during the smelting and dissolution stages respectively.

F. R. ENNOS.

Manufacture of pulp board. H. T. PRICE (U.S.P. 1,696,896, 25.12.28. Appl., 13.4.27. Appl., 13.4.27. Austral., 14.6.26).—The board consists of finely-ground ti-tree wood.

H. ROYAL-DAWSON.

Saccharification of cellulose (B.P. 273,317).—See XVII. **Treatment of textiles** (B.P. 272,541).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Action of ultra-violet rays in bleaching cellulose. R. ESCOURROU (Chim. et Ind., 1928, 19, 989–997).—The effect of ultra-violet radiation in accelerating the bleaching of wood-pulp pastes (in conditions varying from the raw to bleached) has been examined by exposing the reactants to the radiation from a mercury-vapour lamp. When chlorine bleaching agents (*e.g.*, hypochlorite solutions) are used, the acceleration produced enables the reaction to be carried out at a lower temperature, but more chlorine is used up owing to increased formation of chlorates (which play no part in bleaching) and to the fact that under these conditions the cellulose is attacked as well as the non-cellulose impurities. The material thus obtained is rich in oxy- and hydro-cellulose, as shown by its enhanced copper number (Hägglund), its α -cellulose content falls, and the β - and γ -contents are increased. Whilst this may not be important in papermaking, it is a disadvantage when the material is to be used for making viscose and some kinds of cellulose nitrate. This effect is shown only when chlorine bleaching agents are used; it is not observed when bleaching with ozone or hydrogen peroxide. In this latter case the degraded forms of cellulose are not produced, and normally low copper numbers are found. Under these conditions α -cellulose remains unaffected, and for the viscose industry, which demands high α - and low β - and γ -cellulose contents, the use of oxidising agents such as ozone or hydrogen peroxide, either alone or in conjunction with hypochlorite solutions, is recommended.

B. P. RIDGE.

Behaviour of cellulose acetate towards amino-derivatives of anthraquinone. H. M. BURNS and J. K. WOOD (J. Soc. Dyers and Col., 1929, 45, 12–15; cf. Kartaschov, B., 1926, 49, 188; 1928, 812).—Experiments were carried out in order to test the hypothesis that the dyeing of cellulose acetate silk with amino-anthraquinones is attributable to adsorption. The bases employed were purified samples of α -, β -, and 1:4-diamino-anthraquinone suspended in 0.5% solutions of gelatin. To 100 c.c. each of a number of suspensions of various concentrations was added 0.2 g. of dried cellulose acetate silk, and the materials were kept at 26° for 8 days, being shaken at intervals. Portions of the suspensions were then removed and the amount of base remaining was determined. When the equilibrium values showing the concentration of the base on the fibre and in the liquid, respectively, were plotted against each other, curves approximating in shape to those of an ordinary adsorption isotherm were obtained. Of the three bases, the 1:4-diamino-compound was adsorbed most strongly and the β -compound least, but there did

not appear to be any definite numerical relationship between their degrees of adsorption. As the adsorption was irreversible the authors conclude that after the adsorption of the base by the fibre a secondary action occurs leading to the formation of a complex, possibly a solid solution. L. G. LAWRIE.

Application of Naphthol AS dyes to animal fibres. J. RATH (Textilber., 1928, 9, 585—586).—Priority is claimed for the successful application of Naphthol AS dyes to wool and silk. The fabrics may be satisfactorily "prepared" with a Naphthol AS compound by the usual methods (but using the minimum quantity of caustic alkali) without suffering deterioration. To obtain satisfactory Naphthol AS dyeings on animal fibres, coupling of the developer and naphthol should be effected under acidic conditions. It is suggested that the usual addition of formaldehyde to the "prepare" liquor should be omitted in the case of wool, but not of silk. Naphthol AS dyeings on animal fibres have excellent fastness, their fastness to light on wool being greater than that on cotton; dyeings on silk are resistant to processes of degumming. A. J. HALL.

Reserves under sulphur colours [in printing]. F. WOSNESSENSKY (Sealed Notes Nos. [A] 1920, [B] 1926, [C] 1945, [D] 1951, and [E] 1952, 3.8., 2.9., 30.10., and 19.11.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 657—660). Report by P. BINDER (*Ibid.*, 660—661).—(A) Khaki-coloured reserves under sulphur colours are obtained by means of thickened pastes containing nitroso- β -naphthol, zinc chloride, and a metallic mordant (salts of iron and cobalt for greenish and reddish shades of khaki, respectively). (B) Reserves coloured with Thioindigo Red B or Scarlet R, or brominated indigo dyes are obtained by means of a thickened paste consisting of the indigo dye, glycerin, Rongalite, zinc chloride, and sodium acetate. (C) For indigo reserves a paste is used prepared by adding indigo powder, glycerin, Rongalite solution, and anthraquinone paste to a thickened solution of zinc chloride and caustic soda or sodium acetate. (D) Blue reserves under sulphur colours cannot be obtained satisfactorily by means of Dianisidine Blue because the acid treatment of fabric after printing affects the blue coloured reserves; good reserves result if the β -naphthol usually employed is replaced by 2:3-hydroxynaphthoic acid. (E) A Sulphur Blue reserve may be obtained by means of a paste consisting of the leuco-derivative of Sulphur Blue (B.A.S.F.), glycerin, sodium hyposulphite, and a thickened solution of zinc chloride. The leuco-derivative is prepared by concentrating a mixture of Sulphur Blue, in alkaline sodium hyposulphite solution, to which ice and acetic acid have been added. BINDER reports favourably on the processes. A. J. HALL.

PATENTS.

Dyeing of textile fibres, films, etc. made with or containing cellulose esters or ethers. BRIT. CELANESE, LTD. (B.P. 282,036, 23.11.27. U.S., 11.12.26).—Tightly wound, knitted, knotted, or woven goods of cellulose acetate etc. are evenly dyed by applying suitable dyes in solution in an organic solvent, e.g., benzene, trichloroethylene, alcohols, or mixtures of these.

C. HOLLINS.

Treatment [loading, mordanting, or dyeing] of materials made of or containing cellulose derivatives [esters or ethers]. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 302,775, 2.11.27).—Acetate silk etc. is treated with alkaline tin solutions under conditions which bring about partial hydrolysis, with or without addition of swelling agents. Material so treated may be dyed with direct, mordant, or vat dyes as well as with the usual basic or insoluble colours.

C. HOLLINS.

Printing with vat dyes. E. C. R. MARKS. FROM E. I. DU PONT DE NEMOURS & Co. (B.P. 302,252, 8.6.27).—Textiles are printed with vat dye pastes containing, besides reducing agent, tri-(β -hydroxyethyl)amine or a similar substance.

C. HOLLINS.

Coating, impregnating, dressing, sizing, and printing of fibrous material. L. LILIENFELD (B.P. 302,115, 20.6.27).—A viscose solution into which air has been injected is applied to the fabric, and the cellulose is regenerated to produce a dressing containing hollow spaces distributed throughout its mass; this gives to the dressing a downy texture. Gas generated within the viscose solution from sulphites or carbonates has a similar effect.

C. HOLLINS.

Treatment [delustring] of artificial silk. H. A. GARDNER (U.S.P. 1,692,372, 20.11.28. Appl., 13.5.27).—The silk is impregnated with 4% of its weight of a soluble titanium salt, which is hydrolysed at 65—100° to give a matt deposit of insoluble titanium compound in the silk.

C. HOLLINS.

Aminocellulose derivatives (B.P. 279,801).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

B.p. of electrolytic caustic solutions. C. C. MONRAD and W. L. BADGER (Ind. Eng. Chem., 1929, 21, 40—42).—The b.p. of solutions of sodium hydroxide of varying concentration and saturated with sodium chloride were redetermined to an accuracy of $\pm 0.2^\circ$ for pressures varying from 90 to 760 mm. The boiling flask was of monel metal and the thermometer tube used was nickel-plated, whilst an automatic device kept the pressure constant to within 1—2 mm. The slope of the Dühring line for solutions of increasing concentration shows several discontinuities when plotted, the form of the curve closely resembling the f.p. curve of similar solutions.

C. IRWIN.

Synthetic ammonia plant at Ostend. F. A. F. PALLEMAERTS (Ind. Eng. Chem., 1929, 21, 22—29).—This plant is one of a number recently erected obtaining hydrogen from coke-oven gas treated by the "U.C.B." process and then liquefied. Under conditions in Belgium such hydrogen costs less than half that made from water-gas. The cost of production of ammonium sulphate is given as 9 American cents/kg. of nitrogen. The U.C.B. process consists in treating the gas with successive washes of ammonia solution, water, sulphuric acid, and caustic soda. The purified gas is then compressed in two stages to 9 atm., refrigerated, warmed again, and subjected to a pressure water-wash mainly for the purpose of removing acetylene. The ammonia wash

reduces the carbon dioxide of the gas to below 0.1%. The pressure water-wash also removes any benzene which may not have been recovered previous to the water-wash by the refrigerator. The pure gas is cooled to -45° and fractionated. Ethylene, methane, and carbon monoxide are in turn condensed, and the final gas mixture is further purified by washing with liquid nitrogen. This removes methane entirely and leaves only 0.001% of carbon monoxide. The rich residual gas is returned to the ovens, but the possibility exists of other uses, particularly for ethylene. The nitrogen required comes partly from the gas and partly from the liquid nitrogen produced in a Linde liquid air plant. The Casale plant at 750 atm. produces all its ammonia in a liquid form, and it is therefore available for refrigeration. The Casale catalysis tube, which has a life of 1 year (after which the tube should be tested and the catalyst changed, though the tube may last much longer), is insulated from the catalyst space by the incoming cold gases. The reaction is kept slightly endothermic by allowing some ammonia to remain uncondensed in the gas mixture and regulated by an electrically-heated resistance. The ammonia is at present converted into sulphate with sulphuric acid, but it is intended to replace this by converting into ammonium carbonate with flue gases and then proceeding in a way similar to the gypsum process. C. IRWIN.

Solid solutions of lime and arsenic acid. A. T. CLIFFORD and F. K. CAMERON (Ind. Eng. Chem., 1929, 21, 69—70).—Varying quantities of arsenic acid solution were added to a saturated solution of calcium hydroxide with stirring. A precipitate formed in every case and the whole was kept for 2 months at 25° with occasional shaking. The ratio of calcium oxide to arsenic acid in the liquid and solid phases was then determined. The ratio in the solid phases showed a continuous variation according to the quantity of arsenic acid added. It is found that for concentrations up to 25 g. per litre of arsenic oxide, solid solutions are formed, and there is no evidence of the real existence of tricalcium arsenate. C. IRWIN.

The system ferrous oxide-silica. C. H. HERTY, JUN., and G. R. FITTERER (Ind. Eng. Chem., 1929, 21, 51—57).—When silicon is used for the deoxidation of steel ferrous silicate inclusions are produced. A series of m.p. determinations were made for various compositions of the system ferrous oxide-silica, using slags prepared in the laboratory. For each the softening point was also recorded. The m.p. of ferrous oxide was determined as 1355° . Minimum m.p. were observed with mixtures containing 22% of silica and 34% of silica, but the softening points scarcely follow the m.p. curve. The curve is nearly straight, from the second eutectic up to 60% SiO_2 , at which point it intersects the transition line from cristobalite to tridymite. Micro-analysis confirmed the existence of the first eutectic as a mixture of fayalite ($2\text{FeO} \cdot \text{SiO}_2$) and ferrous oxide. Photomicrographs are given and the system is represented in a three-dimensional diagram including the heat content together with temperature and composition. C. IRWIN.

Preparation of artificial white earths. M. NEK-

RITSCH (Z. anorg. Chem., 1928, 177, 86—90).—By the interaction of aluminium sulphate and sodium silicate solutions a precipitate of high adsorptive power is produced, which contains much crystalline material and does not yield up all its aluminium on treatment with acids. The composition, although somewhat variable, is approximately that of hydrated aluminium silicate, $\text{Al}_2\text{O}_3 \cdot 7\text{SiO}_2 \cdot n\text{H}_2\text{O}$. The material may be used for the adsorption of gases and for the purification of mineral and vegetable oils, and resembles closely natural bentonite. H. F. GILLBE.

Sublimation of sulphur between 25° and 50° . R. P. TUCKER (Ind. Eng. Chem., 1929, 21, 44—47).—The sublimation of sulphur at temperatures of 25° and upwards was observed in flasks with sulphur in the bottom, crystals in the upper part appearing in 2—4 weeks. The rate of sublimation was observed by consideration of the time necessary to produce a black film on silver foil. It increased very rapidly with temperature. An admixture of 10% of lampblack increased the rate of sublimation in sunlight owing to the higher temperature reached. Attempts were made to compare the sublimation rates of different forms of commercial sulphur by the silver-foil method, by weighing, and by microscopical measurement. Whilst results were variable no definite differences could be detected, and a mixture of equal parts of sulphur and calcium carbonate ground together sublimed as readily as pure sulphur. The rate of sublimation at temperatures below 35° decreases with time. The insecticidal value of sulphur is considered to depend on sublimation. C. IRWIN.

Mists and dusts. REMY. **Potentiometric and conductometric analysis.** CALLAN and HORROBIN.—See I. **Ammonia from coke-oven gas.** LIEPATSCHIK, also DENISOV.—See II. **Phosphates for plants.** UNGERER. **Manuring with iodine.** DOERELL.—See XVI.

PATENTS.

Acid-concentrating chamber. O. C. TRAUTMANN (U.S.P. 1,695,619, 18.12.28. Appl., 15.4.27).—A series of concentrating chambers has horizontally projecting tubular parts joined in stairway fashion so that only these horizontal parts are located in the inclined flue from a firebox, the joints being on the outside of the flue. Means for preheating the acid and connecting the preheater with the uppermost chamber are provided, and an aperture is arranged in each chamber for escape of the vapours. W. J. BOYD.

Catalytic apparatus for the synthesis of ammonia. F. C. REED (U.S.P. 1,689,684, 30.10.28. Appl., 2.8.27).—A number of concentric members are connected as a unit to the cover of a pressure vessel forming a catalytic chamber above which is an annular heating chamber having a heat exchanger in the upper portion and a heater in the lower portion. Gases are admitted to the heating chamber and are led into the catalytic chamber at points midway in its wall, being directed in an axial direction by an annular baffle. They then pass from the centre of the catalytic chamber to the heat exchanger. A cylindrical shell is attached to the cover of the vessel and

is spaced therefrom, and a circulating pipe and condenser connect the upper and lower portions of the vessel.

W. C. CAREY.

Manufacture of alkali cyanides. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,565, 6.9.27).—A mixture of ammonia and carbon monoxide is caused to react at 400–800° with the hydroxide, oxide, or salt of an alkali metal diluted with an indifferent filler, *e.g.*, magnesium oxide, and with the addition of a suitable catalyst, *e.g.*, an iron compound not reduced during the reaction.

W. G. CAREY.

Production of nitrogenised metallic compounds [cyanides, cyanamides, and nitrides]. R. B. GOLDSCHMIDT and S. COULIER (B.P. 277,714, 20.9.27. Belg., 20.9.26).—Mixtures, containing excess carbon, of raw peat with alkali, alkaline-earth, or earth-metal compounds for the production of cyanides or cyanamides, *e.g.*, sodium carbonate, or with magnesium or aluminium compounds for the production of the nitrides of these metals, together with catalysts for fixing nitrogen, *e.g.*, iron, manganese, nickel, or cobalt, or their oxides, or alkali halides, and catalysts for activating the carbon, *e.g.*, zinc or magnesium chloride, oxide, or phosphate, or titaniferous compounds, are briquetted, dried if desired, carbonised, and nitrogenised. After the product has been extracted, the residual carbon is purified, and part of it may be added to a fresh charge. L. A. COLES.

Production of nitrogenised metallic compounds. R. B. GOLDSCHMIDT and S. COULIER (B.P. 277,715, 20.9.27. Belg., 20.9.26).—The compounds are obtained by the carbonisation and nitrogenisation of mixtures of carbonaceous material, *e.g.*, wood, spent malt, oil cake, etc., with alkali, alkaline-earth, or earth-metal compounds, together with catalysts, if desired. Activating gases, *e.g.*, steam, carbon monoxide, carbon dioxide, hydrogen, nitrogen, or chlorine, are passed over the material during the carbonisation, and excess of active carbon, obtained in a previous operation, is added to the mixture before and/or after the carbonisation, the excess of carbon being separated from the nitrogenised product after the treatment. Both processes may be effected in the same vertical or inclined retort in which a descending mass of the mixture is treated with an ascending stream of the gases.

L. A. COLES.

Production of calcium cyanamide or mixtures containing it. N. CARO and A. R. FRANK (B.P. 279,419, 30.9.27. Ger., 23.10.26).—Calcium carbonate is treated with ammonia or mixtures of it with non-reacting gases, at above 450° and at 3–10 atm., the apparatus being constructed of material which does not cause dissociation of ammonia, *e.g.*, quartz, ceramic material, or copper or its alloys. Means are provided for condensing and removing the steam liberated during the reaction.

L. A. COLES.

Freezing mixtures. L. A. and B. GACHEY (B.P. 298,623, 4.8.28. Austr., 14.10.27).—Ammonium nitrate and crystallised sodium carbonate are mixed in equal proportions at the time of use and water is added.

W. G. CAREY.

Producing a mixture of calcium nitrate and ammonium nitrate. KUNSTDÜNGER PATENT-VER-

WERTUNGS A.-G., Assecs. of F. G. LILJENROTH (B.P. 301,486, 23.10.28. Sweden, 1.12.27).—A mixed fertiliser of calcium and ammonium nitrates is produced by acting on calcium sulphate with ammonia and carbon dioxide, treating the resulting calcium carbonate, together with a suitable amount of ammonium sulphate left therein, with nitric acid, and removing the precipitated calcium sulphate.

W. G. CAREY.

Reduction of [barium] sulphate minerals and briquettes therefor. J. E. BOOGE and J. P. KOLLER, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,685,772, 2.10.28. Appl., 10.11.24).—Barytes (4 pts.) and coal (1 pt.) are mixed with a binder, *e.g.*, starch, molasses, or a crude oil, and the mixture is formed into briquettes having a diameter of 1–4 in. On heating the briquettes at 950–1150° a high yield of barium sulphide is obtained even with ore containing up to 10% of silica, owing to the low porosity of the briquettes preventing ingress of furnace gases.

A. R. POWELL.

Production of anhydrous zinc chloride. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 288,253, 1.3.28. Ger., 9.4.27).—Zinc oxide is treated at 400–500° with chlorine in the presence of excess of a mixture of hydrogen and carbon monoxide.

W. G. CAREY.

Production of water-softening silicate. E. W. SHAWEN and A. C. ARNETT (U.S.P. 1,693,873, 4.12.28. Appl., 9.2.25).—Sodium bicarbonate is incorporated with porous vitreous material, the mixture is heated, and while hot is treated with sodium chloride.

W. G. CAREY.

Separation of gases [helium] from [natural] gaseous mixtures. M. H. ROBERTS, Assr. to AIR REDUCTION Co., Inc. (U.S.P. 1,693,052, 27.11.28. Appl., 25.1.19).—Natural gas is compressed, cooled, and expanded to liquefy a portion of it, and the residual gas is subjected to successively lower temperatures, at the pressure to which it was expanded, to condense constituents other than helium, the condensed fractions being used separately and successively for cooling the gas to such a temperature that only the helium remains uncondensed.

L. A. COLES.

Production of hydrogen of great purity. A. W. BURWELL, Assr. to ALOX CHEM. CORP. (U.S.P. 1,689,858, 30.10.28. Appl., 22.8.27).—Hydrocarbon material is heated at 1050–1150° to produce a mixture of hydrogen, carbon monoxide, carbon, and hydrocarbons, from which carbon is removed by settlement and aromatic hydrocarbons by scrubbing with water. Hydrocarbons similar to methane are converted into carbon monoxide by heating the moist mixture at 1230–1280°, and the carbon monoxide is converted into dioxide by adding excess of oxygen to the dried gases and passing them over a manganese dioxide catalyst below 100°. Any oxygen remaining is removed by passing the gases over heated copper, and carbon dioxide by caustic alkali solution.

W. G. CAREY.

Purifying hydrogen and gases containing hydrogen. GEWERKSCHAFT DER STEINKOHLENZECHHE MONT-CENIS (B.P. 276,687, 26.8.27. Ger., 30.8.26. Cf. B.P. 276,668; B., 1928, 894).—Organic compounds detrimental to contact processes are removed by passing the

gases at high pressure and temperature over catalysts, e.g., iron, cobalt, nickel, chromium, manganese, zinc, or vanadium oxides or metals, with the addition of alkaline-earth oxides, so that inorganic compounds and saturated hydrocarbons are formed, the former being absorbed by basic or oxidising substances added to the catalyst. Copper is excluded from the process.

W. G. CAREY.

Production of sulphur dioxide and hydrogen. C. H. MACDOWELL and H. H. MEYERS, Assrs. to ARMOUR FERTILIZER WORKS (U.S.P. 1,693,244, 27.11.28. Appl., 21.11.27).—A mixture of sulphur vapour and steam is heated at a suitable temperature in the presence of iron oxide as catalyst for a sufficient time to yield a mixture containing sulphur dioxide and hydrogen, and these gases are recovered separately from the product.

L. A. COLES.

Contact sulphuric acid process. SELDEN Co., Assces. of A. O. JAEGER (B.P. 294,975, 23.1.28. U.S., 3.8.27).—See U.S.P. 1,675,308; B., 1928, 783.

Sodium nitrogen compounds (B.P. 293,040).—See III.

VIII.—GLASS; CERAMICS.

New type of tunnel kiln for firing pottery. J. WILLIAMSON (Trans. Ceram. Soc., 1928, 27, 290—296).—A tunnel kiln of the combined direct-flame and muffle type is described. It consists of preheating, equalising, furnace, and cooling zones. In the furnace zone the flames take a transverse course, either by up- or down-draught. Six burners are provided on either side, spaced a truck length apart. The flames from the burners meet a stream of hot air at the top of the bag walls. The equalising zone is a double-vaulted muffle which completely encircles the trucks. The flue systems in the various zones are described. F. SALT.

[Effect of heat on] the crystalline break-up of kaolin. J. F. HYSLOP and H. P. ROOKSBY (Trans. Ceram. Soc., 1928, 27, 299—302; cf. B., 1928, 605).—The β -phase previously reported has been identified as α -alumina. The α -phase formed at 550° has not been identified. This breaks down at 870° with the formation of γ -alumina and sillimanite. There is no evidence of the formation of α -alumina above 870°, though between this temperature and 1060° the γ -alumina decreases and finally disappears. Mullite is formed above 1060°.

F. SALT.

Drying of clay. R. S. TROOP and F. WHEELER (Trans. Ceram. Soc., 1928, 27, 303—319).—The contraction of clay and its relation to rate of drying and shape of piece, and the effect of the physical conditions of the drying medium on the rate of removal of moisture from the clay and on the distribution of moisture in the piece have been studied. Measurement of linear contraction is rejected as a basis for comparing the drying behaviour of clays because it is vitally affected by compression and extension stresses associated with the method of making the test pieces. Rectangular test pieces showed irregular contraction on the different faces, and also at different points along the same face. Volume contraction was measured in a mercury volumeter. Results are given for fireclay, china clay, and ball clay. With china clay an expansion occurred between a moisture

content of 11% and the dry state. The rate of drying appeared to have no direct effect on contraction. A "main contraction" occurs in the early stages, when the contraction is equivalent in volume to the amount of water removed by evaporation; at a "critical point" the rate of contraction decreases suddenly, and "residual contraction" then proceeds at a much slower rate. The "plasticene coating method" of measuring porosity was also applied to the measurement of contraction. Measurement of the rate of drying of clay cylinders by air of definite humidities and temperatures (with apparatus previously described; B., 1928, 569) indicated lower water gradients in ball clay than in fireclay samples. An increase in the drying temperature reduced the water gradients in clays, and thus ensured safer drying. F. SALT.

Crushing strength of unfired fireclay bodies. W. C. HANCOCK and J. G. COWAN (Trans. Ceram. Soc., 1928, 27, 243—246).—A Stourbridge clay, and four mixtures of this clay with 20% and 40% of coarse and fine grog prepared from another hard-fired Stourbridge clay, were tested in the dry state for crushing strength, and the results were compared with the tensile strength. Three shapes of test-piece were used in determining the crushing strength. The highest figures were obtained with a mixture of 80% of clay and 20% of fine grog. Three types of fracture observed in the crushed pieces are illustrated. F. SALT.

Refractory material used as mortar for laying-up refractories. D. A. MOULTON (Trans. Ceram. Soc., 1928, 27, 329—333).—Bentonite is a good "floating" medium to prevent settling of a refractory cement when mixed to a thin consistency. A satisfactory refractory cement is made from ground fireclay grog, of similar chemical composition to the refractories, mixed with a colloidal material such as bentonite or aquacryptite. A monolithic lining for various types of furnaces may be made from this cement, using coarse grog.

F. SALT.

Artificial white earths. NEKRITSCH.—See VII.

PATENTS.

Manufacture of glass threads. F. POLLAK (CHEM. FABR. STOCKERAU DR. F. POLLAK) (B.P. 288,978, 14.4.28. Austr., 16.4.27).—The glass is fused by an electric resistance heating member suitably distributed in the melting pot, and the fused glass is discharged through nozzles.

W. G. CAREY.

Prevention of ageing in ceramic materials. F. SINGER (B.P. 282,404, 14.12.27. Ger., 18.12.26. Addn. to B.P. 282,403; B., 1928, 712).—The ceramic materials previously described are treated with oxides, carbonates, silicates, or aluminates of cerium, zirconium, chromium, manganese, phosphorus, tungsten, or vanadium in such amount that the product is practically entirely micro-crystalline.

W. G. CAREY.

Bonded refractory. R. E. LOWE, Assr. to DOHERTY RES. Co. (U.S.P. 1,694,924, 11.12.28. Appl., 28.4.25).—A graded refractory aggregate is bonded by less than 1% of a hydroxide precipitated in intimate contact with the particles of aggregate by such means that practically no other substances are formed during the precipitation.

L. A. COLES.

IX.—BUILDING MATERIALS.

Weathering [of the building stone] of the Bremen Town Hall. E. BLANCK and A. RIESER (Chem. Erde, 1928, 4, 137—144).—Analyses are given of the weathered red sandstone of the building erected in 1609—1612.

L. J. SPENCER.

Reaction of water on calcium aluminates. L. S. WELLS (U.S. Bur. Stand. Res. Paper No. 34, 1928, 59 pp.).—A study has been made of the mechanism of the reaction of water on a commercial high-alumina cement and on four anhydrous calcium aluminates ($\text{CaO}:\text{Al}_2\text{O}_3$ ratios of 1:1, 3:5, 5:3, 3:1, respectively). The reaction of water with the last-named compound is very vigorous, and the changes in composition of the resulting aqueous solutions could not be followed. The rapid setting is probably due to the formation of a hydrate occurring directly or by the reaction of the excess of lime on a calcium aluminate produced in the very early stages of setting. The activity of the other aluminates and of the high-alumina cement towards water is distinctly less than that shown by tricalcium aluminate. During the early periods of the reaction a portion of the lime and alumina in each case dissolves as the calcium salt of monobasic aluminic acid, the total concentration depending on the composition of the anhydrous aluminate or cement from which it is formed as well as on the time of contact. These solutions are metastable and soon decompose with precipitation of a portion of the lime and alumina. The molar ratio of the former to the latter remaining in solution after precipitation shows an increase, attended in each case by an increase in the p_{H} from approx. 11.1 to 11.75. Identical changes occur in the metastable solutions after filtration from the reaction mixture. The "equilibrium" solutions which result when the filtered, metastable solutions are kept are similar to those obtained after prolonged contact of the reaction mixture with water, and contained 0.35—0.50 g. of lime and 0.15—0.30 g. of alumina per litre. The establishment of equilibrium requires several weeks. As equilibrium is approached hydrated alumina and hydrated tricalcium aluminate are precipitated. Calculations based on electrometric measurements and chemical analyses indicate that the alumina exists in solution as the calcium salt of monobasic aluminic acid, and that the excess of calcium hydroxide determines the p_{H} of the solution. The conclusion of Slade (A., 1913, ii, 54), Blum (A., 1913, ii, 963), and Heyrovsky (A., 1922, ii, 771) that aluminic acid is a relatively strong monobasic acid is confirmed by these calculations, which, however, are only approximate. As the concentration of calcium hydroxide is raised, increasing precipitation of the alumina occurs until at $p_{\text{H}} \geq 12$ it is complete and *tetracalcium aluminate*, $4\text{CaO}.\text{Al}_2\text{O}_3.12\text{H}_2\text{O}$, appears to be formed. Analogous changes occur in aluminate solutions when mixed with tricalcium silicate. The electrometric titration of aluminium chloride with calcium hydroxide shows that precipitation of aluminium is complete between p_{H} 6.0 and 7.5, but that the aluminium hydroxide begins to redissolve at p_{H} 9.0, dissolution being complete at p_{H} 10.9. At still higher values of p_{H} calcium chloroaluminate is precipitated. It may also be prepared by acting on an aluminate

solution with calcium chloride, and when the concentration of the latter exceeds 3% the precipitate approximates in composition to $3\text{CaO}.\text{Al}_2\text{O}_3.\text{CaCl}_2.10\text{H}_2\text{O}$. The corresponding barium compound appears to be more soluble than the calcium chloroaluminate. Ammonia and magnesium chlorides decompose calcium aluminate solutions, precipitating, respectively, aluminium hydroxide and a mixture of aluminium and magnesium hydroxides.

H. INGLESON.

Hardening of Portland cement and the liberation of lime. S. YAMANE (Bull. Inst. Phys. Chem. Res., Tokyo, 1928, 7, 1177—1190).—The lime content of Portland cement attains a value of over 5% within 7 days of mixing with water, and increases with the water content of the mixture. The strengthening process taking place in Portland cement is due to the reaction: $3\text{CaO}.\text{SiO}_2 + \text{H}_2\text{O} \rightarrow \text{CaO}.\text{SiO}_2.m\text{H}_2\text{O} + \text{Ca}(\text{OH})_2$, and the concentration of hydrated silicate might be taken as a measure of the strength of a given sample of cement. Sand has, in Portland cement mixtures, amongst other functions that of conserving water, whereby a more complete utilisation of $3\text{CaO}.\text{SiO}_2$ is attained. If hardened cement be powdered and extracted with warm glycerol-ethyl alcohol solution, an opalescent filtrate is obtained, the particles of which are coagulated by the addition of water. It follows that hardened Portland cement contains substances in a state of colloidal division.

R. TRUSZKOWSKI.

Setting of cement. II. H. GESSNER (Kolloid-Z., 1929, 47, 65—76; cf. B., 1929, 56).—An apparatus is described for the measurement of the viscosity of cement pastes. Measurements of viscosity were made with pastes containing 30—33 pts. of water to 100 pts. of cement. Immediately after mixing, such a paste has a viscosity comparable with that of glycerol, but the viscosity slowly increases with time and the change becomes rapid after 0.5—1.5 hrs. The change is considered to be due to the formation of a gel through hydration of the particles. The measurements were made on different test portions of the cement; if the same portion of paste is passed repeatedly through the viscosity apparatus, little change in the viscosity is indicated, since the gel structure is destroyed. The electrical conductivity of cement was also measured during setting, and was found to reach a maximum after about 40 min. The subsequent fall in conductivity is rapid at first and gradually slows down, taking some months for completion. With hydraulic mortars, the reduction in volume after mixing with water takes place rapidly during the first five days, then more slowly for about 20 days, and the change is not completed until after about 275 days. With Portland cement the decrease in volume amounts to 3% after 1 day, 4—7% after 3 days, 5—8% after 7 days, 5.5—9% after 28 days, and 6—9% after 3 months. A hydraulic lime under the same conditions showed a smaller decrease in volume, viz., 1% after 1 day, 2% after 10 days, and 4% after 250 days. An alumina cement showed a greater volume decrease than Portland cement, viz., 11% after 1 day, 13% after 5 days, 16% after 28 days, and 17% after 150 days.

E. S. HEDGES.

Refractory cement. MOULTON.—See VIII. **Dental materials.** COLEMAN.—See X.

PATENTS.

Manufacture of artificial stone of crystalline structure. G. CONOD and F. C. F. LECOULTRE (B.P. 288,202, 26.3.28. Belg., 4.4.27).—The stone (artificial marble) is obtained by mixing a binding agent, *e.g.*, calcium sulphate, aqueous colloidal silica solution, and powdered calcium carbonate obtained synthetically in a crystalline form by heating limestone at about 1290° under 100 atm. pressure and dropping it into water while still hot, or by adding sodium carbonate to calcium bicarbonate solution, colouring matter being added if desired.

I. A. COLES.

Wet process for manufacturing cement. W. P. ECKDAHL (U.S.P. 1,693,644, 4.12.28. Appl., 24.5.26).—The setting of a slurry of finely-divided silicious material having a basic reaction is prevented by bringing gases with a high content of carbon dioxide into contact with it.

A. R. POWELL.

Hardened plaster product. Plaster board. W. II. KOBBE, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,693,715—6, 4.12.28. Appl., [A, B] 14.3.27).—(A) Dehydrated, set gypsum is impregnated either wholly or in part with sulphur. (B) The board comprises an interior of hardened plaster covered in part at least with fibrous material which is bonded to the interior by sulphur.

W. G. CAREY.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Protection of underground pipe from corrosion. Method used in Southern California. E. O. SLATER (Ind. Eng. Chem., 1929, 21, 19—21).—The deterioration of underground pipe lines in relation to the capital value involved is discussed. The rate of corrosion is determined to a major extent by the character of the soil and soil water in contact with the pipe. Any protection against corrosion apart from corrosion-resistant metal must therefore prevent effective contact between the moist soil and the pipe line. A coating successfully used in Southern California consists of a paint produced from native bitumens. The bitumen coating on previously sand-blasted metal usually proves a satisfactory protection, but where the soil conditions are known to be severe a bitumenised cotton wrapping is applied with a subsequent "paint" coating.

C. A. KING.

Effect of additions of lime and soda ash to brackish water on the corrosion of iron and steel. H. O. FORREST, J. K. ROBERTS, and B. E. ROETHELI (Ind. Eng. Chem., 1929, 21, 33—35).—As a protection against the corrosion of iron and steel particularly in cooling systems situated along sea coasts and using brackish water, the formation of a calcium carbonate scale was tried with success. Small quantities of calcium bicarbonate in distilled water greatly accelerate corrosion, but higher concentrations assist in building up a carbonate film. In waters containing 50% or more of sea-water the protection afforded by treatment with calcium hydroxide is relatively poor, being about

30—40% as a maximum. This is due largely to the presence of magnesium salts which probably lower the hydrogen-ion concentration of the water, and which also produce a non-adherent precipitation of magnesium hydroxide. The further addition of sodium carbonate corrects this tendency, and when the p_H of the solution was 8.5 or above a satisfactory protective coating was obtained.

C. A. KING.

Influence of nitrogen on the solubility of ferrous materials in hydrochloric acid. II. Effect on the carbon content. III. Further experiments. H. H. GRAY and M. B. THOMPSON (J.S.C.I., 1929, 48, 21—25 τ , 25—28 τ ; cf. B., 1926, 919).—II.—The ferrous material was heated for a period (usually 6 hrs.) in pure dry nitrogen at temperatures up to 1100°. Nitrogen is found to decarburise ferrous alloys with the formation of a volatile compound, probably cyanogen. This reaction is found to be reversible. The loss of carbon depends on temperature and on the initial carbon content of the alloy. The importance of the results in connexion with case-hardening and nitrogen fixation is indicated. The literature of the subject is reviewed.

III. The solubility of the nitrogenated material is found to be affected by the initial carbon content and by the temperature of nitrogenation. Microscopical examination shows the nitrogenated metal to be covered with a transparent, crystalline layer which is practically insoluble in dilute hydrochloric acid. This is the chief obstacle to dissolution in acid, but the underlying mass of the ferrous material is also rendered less soluble by the nitrogenation. The "resistivity" shown to hydrochloric acid is confirmed by *E.M.F.* experiments. The appearance of the nitrogenated material is illustrated by means of a photomicrograph showing a well-developed crystalline structure without the use of etching reagents.

Illustration of the transformations of brasses containing 57.5—63.5% Cu by the study of their mechanical properties. P. DEJEAN (Compt. rend., 1929, 188, 169—172).—The temperature-plasticity curves of these alloys show two points of inflexion corresponding with the transformation temperatures observed by other workers. The first change occurs at 480° for each alloy, and is considered to be the conclusion of a progressive transformation starting at 250—300°. The second represents the transformation of the ($\alpha + \beta$) into the β -constituent, and occurs at a temperature (685—800°) which increases with the copper content of the brass.

J. GRANT.

Internal transformations of a cupro-aluminium. A. ROUX and J. COURENOT (Compt. rend., 1929, 188, 172—173).—The structural transformations of a 90:10 cupro-aluminium have been studied by means of X-rays before and after annealing for 20 min. at 900° and slow cooling, quenching in cold water after 5 min. at 900°, and reheating of the quenched metal for 20 min. at 650° or 200°. Only the spectrum of the α -solution appears in that of the annealed metal; in the case of the quenched metal this spectrum shows with additional lines, but without the lines corresponding with the [100] plane. A solid solution of Cu_3Al in the α -solution is indicated.

J. GRANT.

Determination of traces of antimony in copper and its alloys. S. G. CLARKE and B. S. EVANS (Analyst, 1929, 54, 23—28).—5 g. of sample are dissolved in 30 c.c. of 1:3 sulphuric acid and 15 c.c. of concentrated nitric acid, and evaporated to fuming point. After boiling, the residue is dissolved in 150 c.c. of water, 150 c.c. of hydrochloric acid (*d* 1.18) and 10 g. of sodium hypophosphite are added, and the mixture is boiled for 10 min. If a brownish-black precipitate due to arsenic appears, the liquid is boiled for another 20 min., and the precipitate coagulated by 20 c.c. of benzene and filtered off. If a tin bronze is being tested, 10 g. of oxalic acid are dissolved in the hydrochloric acid solution before adding the hypophosphite. In the absence of any appreciable amount of arsenic, the Reinsch test is at once proceeded with. A flat spiral of electrolytic copper foil is cleaned and then boiled in the solution for 2 hrs., quickly rinsed in water, and placed without delay in a small beaker, covered with distilled water, and about 1 g. of sodium peroxide added, and after 5 min. the beaker is warmed until the coil becomes darkened with oxide. The antimony should now have been completely removed from solution together with any bismuth and arsenic and a little copper. The coil, after rinsing, is immersed in dilute sulphuric acid, which removes the oxide and shows up any antimony which may have escaped stripping. The antimony solution is treated with a rapid current of hydrogen sulphide for 15 sec., and after a short while the precipitated copper and bismuth sulphides are washed with 1% ammonium nitrate solution, and 5—6 c.c. of concentrated sulphuric acid are added to the filtrate, which is evaporated to fuming point after addition of a few drops of nitric acid. The solution of antimony is diluted with 15 c.c. of water, heated to b.p., cooled, and the antimony determined colorimetrically. 10 c.c. of 1% gum arabic, 5 c.c. of 20% potassium iodide, 1 c.c. of 10% aqueous pyridine, 1 c.c. of dilute solution of sulphur dioxide, and 60 c.c. of cold 1:3 sulphuric acid are consecutively added to a 10 c.c. Nessler tube, followed by the antimony solution. Standard antimony solution (0.0001 g. Sb per c.c., 0.2764 g. of tartar emetic in 1 litre of 10% sulphuric acid) is run into another glass containing the same reagents, except that 80 c.c. of sulphuric acid instead of 60 c.c. are used, until the colours match. The concentration of the acid used in the quantitative separation of the antimony on copper in the presence of cuprous chloride is of great importance.

D. G. HEWER.

Lead-tin-cadmium as a substitute for lead-tin wiping solder. E. E. SCHUMACHER and E. J. BASCH (Ind. Eng. Chem., 1929, 21, 16—19).—The replacement of part of the tin in solder by cadmium would lower costs, not by a difference in the intrinsic value of the two metals, but because it allows the use of a higher proportion of lead. As judged by the working qualities, porosity of joints, and tensile strength, certain of the ternary alloys, though within narrow limits of composition, produced joints as satisfactory as those from the standard alloy (62% Pb, 38% Sn). The most satisfactory composition was 68% Pb, 23% Sn, and 9% Cd, and increase to the extent of even 1% in lead or cadmium showed markedly in an adverse direction. C. A. KING.

Physical properties of dental materials (gold alloys and accessory materials). R. L. COLEMAN (Bur. Stand. J. Res., 1928, 1, 867—938).—The physical properties of dental gold alloys, in the wrought and cast states, investments, and pattern waxes were investigated. 18 wrought alloys of commercial manufacture for use as wire, clasps, and plate were selected. They vary widely in composition, but the chief constituents are gold, silver, copper, and palladium or platinum, with small amounts of zinc, tin, nickel, manganese, and iron. The fusion temperature for wires varies from 895° to 1000°, and for plate from 950° to 1400°. The alloys are softened by heating to 700° and quenching in water, or hardened by cooling from 450° to 250° during 30 min., and are more resistant to corrosion in the hardened state. Static flexure, Brinell hardness, tensile, and alternating stress test results are given for all the alloys. The best physical properties were shown by an alloy containing Ag 5.1%, Au 64.5%, Pt 17.6%, Pd 2.1%, Cu 10.4%, and Zn 0.8%, which had an ultimate strength of 153,000 lb./in.² and elongation of 7.5% on 8 in. The fatigue-resistance is greatly increased by hardening. 35 commercial alloys representative of casting golds and solders were investigated, and showed the same variation in composition as the wrought alloys. The casting alloys melt at 900—1000°, and the solders at 750—800°. The alloys show no serious change in composition on remelting 3 or 4 times. Castings showed porosity due to occluded gas and localised shrinkage. The former can be eliminated by using vacuum-melted alloys, and the latter by using a short, wide sprue. Gold foil fillings were harder than an inlay cast of 24-carat gold, and nearly as hard as 22-carat gold. The net casting shrinkage is unaffected by the pouring temperature, the average value being 1.25%. The thermal expansion of 5 inlay waxes was determined. The shrinkage on cooling from 40° to 25° was 0.75%. 11 investment materials were analysed and dimensional changes caused by setting and heating measured. They contained calcined gypsum 20—55%, silica 80—45%, and expanded on setting up to 0.5%. In general, an expansion of 0.2—0.3% is obtained by heating to 300°. Data on compressive strength and porosity of investments are given. Accurate casting to dimensions can be achieved by suitable selection and manipulation of alloy, wax, and investment.

C. J. SMITHHELLS.

System ferrous oxide-silica. HERTY and FITTERER.—See VII. **Electrodeposition of copper.** JULIARD and LEDRUT.—See XI.

PATENTS.

Metallurgical furnace. D. CUSHING (B.P. 303,042, 26.9.27).—The base of a cupola furnace is hinged and movable at will, the body having an annular sealing rim. The refractory lining is tongued and grooved or otherwise ribbed to retain its position in the flanged base, and is inclined down to the tapping spout. C. A. KING.

Operation of blast furnaces. A. E. WHITE. From S. G. ALLEN (B.P. 303,206, 30.9.26).—The blast to a furnace is oxygenated to a degree that would cause excessive smelting capacity as compared with the shaft

reduction capacity, and the ratio of ore to coke is increased, additional heat and reducing gases being supplied above the mantle in the form of oil, gas, or pulverised fuel. C. A. KING.

Treatment of ores with gases in rotary furnaces.

C. P. DEBUCH (B.P. 303,072 and 303,096, 27.7.27).—(A) In a rotary tubular furnace heated by a number of gas ports distributed along the length of the furnace, the wall is increased gradually in thickness from the main reaction zone. Particular zones may be defined by internal rings, and the speed of movement of the charge is regulated by the number and disposition of prongs which also act as protectors against ore falling into the gas ports. (B) When roasting weak ores, fuel is introduced at various points along the furnace; the fuel may consist of the finely-divided ore.

C. A. KING.

Furnace for treating ores. F. M. SIMONDS (U.S.P. 1,693,702, 4.12.28. Appl., 26.10.26).—The hearth is formed in sections each of which consists of a floor and a perforated confining wall at one end forming an angle with the floor.

M. E. NOTTAGE.

Treatment of iron ores. T. P. CAMPBELL (U.S.P. 1,696,188, 25.12.28. Appl., 19.5.27).—The comminuted ore is brought into a spongy state by reduction, and after being partially reoxidised is then passed over a magnetic separator.

H. ROYAL-DAWSON.

Manufacture of steel. F. C. LANGENBERG and M. A. GROSSMANN (U.S.P. 1,695,594, 18.12.28. Appl., 1.6.27).—A non-hot-short steel contains <0.05% C and <0.03% O.

H. ROYAL-DAWSON.

Heat treatment of steel. LE B. W. KINNEY and G. H. BIERMAN, ASSRS. to WHITE MOTOR CO. (U.S.P. 1,695,922, 18.12.28. Appl., 27.9.28).—A composition consisting entirely of kieselguhr, water, and borax serves to prevent decarbonisation during heat-treatment of the metal.

H. ROYAL-DAWSON.

Carburisation of metals. G. W. HEGEL and G. R. BROPHY, ASSRS. to GEN. ELECTRIC CO. (U.S.P. 1,696,603, 25.12.28. Appl., 26.8.26).—A carburising liquid containing bone oil is used.

H. ROYAL-DAWSON.

Slag and gas eliminator for molten steel. F. H. MOYER (U.S.P. 1,690,748, 6.11.28. Appl., 24.7.26).—The steel is tapped from the furnace into a ladle in the shape of an inverted, truncated cone divided by a baffle parallel to the side of the furnace and extending nearly to the bottom of the cone. The purified metal overflows from the far side of the cone into a larger receptacle for transfer to the moulds.

A. R. POWELL.

Hardening of iron, mild steel, and iron alloys. H. LINDHORST (B.P. 302,740, 28.9.27).—Cementation of iron is effected by heating it at 720–820° in a closed chamber together with a small quantity of powder out of contact with the metal and which evolves carbon dioxide and other gaseous hydrocarbons.

C. A. KING.

Pickling of iron and steel. V. BERTLEFF (B.P. 293,701, 9.7.28. Austr., 9.7.27).—To the bath of usual pickling liquor a protective agent is added consisting of sulphonated products of coal-tar oil, made by heating the latter with not more than an equal weight of con-

centrated sulphuric acid until complete solubility in water has been attained; or salts, *e.g.*, those of calcium or sodium, or the purified sulphonic acids prepared from such sulphonated oils may be used.

M. E. NOTTAGE.

Zirconium-treated iron-chromium alloy. F. M. BECKET, ASSR. to ELECTRO METALLURGICAL CO. (U.S.P. 1,689,276, 30.10.28. Appl., 8.5.26).—Addition of small quantities of zirconium, added as silicon-zirconium alloy, to iron-chromium alloys containing 10–60% Cr greatly increases the ductility and renders them more easily rollable. *E.g.*, a low-carbon steel with 9–30% Cr, less than 3% Si, and up to 0.5% Mn is treated with zirconium sufficient to leave 0.02% in the alloy.

A. R. POWELL.

Manufacture of metal articles and alloys therefor. W. H. HATFIELD and H. GREEN (B.P. 302,812, 31.12.27).—A steel alloy resistant to corrosion and to the staining effects of foodstuffs contains 11.5–13% Ni, 11.5–13% Cr, and not more than 0.2% C. The alloy is rendered ductile for cold-working by heating it at 950–1090°, preferably 1000–1050°, and cooling it rapidly at first and then at a slower rate.

C. A. KING.

Preparation of the surfaces of iron, steel, and copper articles for nickel-plating. W. A. BURFORD (G.P. 451,620, 7.10.26).—The articles are pickled anodically in a bath containing 45–55% of sulphuric acid, using a current density of 10 amp./dm.² A higher current density may be employed if nitric acid is added to the bath, an extra 5 amp./dm.² being used for every 2% of nitric acid. The articles are rinsed after treatment, dipped into concentrated nitric acid, and plated directly.

A. R. POWELL.

Copper alloys. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 288,314, 4.4.28. Ger., 9.4.27. Addn. to B.P. 286,616; B., 1928, 898).—The workability and capability of being strengthened of these alloys can be considerably increased if the iron content be replaced wholly or partially by cadmium, tin, or manganese. Before being shaped the alloys are rolled hot, annealed above 600°, quenched to room temperature, and mechanically worked to a cross-section slightly greater than the final; the metal pieces are then re-annealed either at 300–600° and cooled slowly, or at temperatures above 600°, rapidly cooled to 300–400°, and afterwards slowly cooled.

M. E. NOTTAGE.

Erratum.—B., 1928, 898, col. 2, line 21 from bottom, for “286,615” read “286,616.”

Leading-in wires for evacuated [glass] containers. J. A. YUNCK (U.S.P. 1,695,791, 18.12.28. Appl., 6.8.27).—A sheath of silver alloy composed principally of silver is welded to a core composed of approx. 40% Ni and 60% Fe.

J. S. G. THOMAS.

Manufacture of bodies [*e.g.*, wire] from metals [*e.g.*, tungsten] having a high m.p. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 284,990, 24.1.28. Holl., 7.2.27. Addn. to B.P. 200,879; B., 1923, 934A).—In addition to the volatile metal compound, the atmosphere in which the metal crystal is heated also contains nitrogen; preferably a current of nitrogen is caused to flow along the heated metal crystal.

M. E. NOTTAGE.

Treatment of ores (B.P. 277,660).—See II. **Reduction of barium minerals** (U.S.P. 1,685,772).—See VII. **Annealing furnaces** (B.P. 273,320). **Heat-treatment and forging furnaces** (B.P. 303,222).—See XI.

XI.—ELECTROTECHNICS.

Effect of different electrolytes on the electro-deposition of copper. A. JULIARD and J. LEDRUT (Bull. Soc. chim. Belg., 1928, 37, 377—384).—Economy of time and electrical energy in the deposition of copper is effected by the addition of small amounts of potassium chlorate or perchlorate to an acid solution of copper sulphate. At 20° the presence of 1% of potassium chlorate results in 75% increase in the current for a given voltage, and reduces the time and electrical energy required for the deposition of a given amount of copper by 30% and 50%, respectively. The deposition of copper in the presence of sulphites, persulphates, permanganates, chromates, dichromates, nitrites, fluorides, and alkaline tartrates is not quantitative, there being either wastage of electrical energy or incomplete deposition.

F. G. TRYHORN.

Detection of coal tar in mouldings for cable parts. E. KINDSCHER and P. LEDERER (Chem.-Ztg., 1928, 52, 1014).—10 g. of the moulding—an insulating, fusible material used for covering the ends and joints of cables—is heated for $\frac{1}{4}$ hr. with 40 c.c. of *N*-sodium hydroxide solution under a reflux. A positive reaction of a small portion of the filtrate with benzenediazonium chloride may indicate the presence not only of coal tar, but also of oxidation products of colophony. The bulk of the solution is then treated with dilute sulphuric acid, and the separated acids and phenolic substances, after extraction with ether and removal of the solvent, are heated with 5—10 c.c. of 10% sodium hydroxide solution until evolution of carbon dioxide ceases, when the carboxylic acids from colophony are destroyed. The liquid is then extracted with ether, the solvent is removed, and the residue is dissolved in sodium hydroxide solution, and tested with benzenediazonium chloride.

F. R. ENNOS.

Potentiometric and conductometric analysis. CALLAN and HORROBIN.—See I. **Reduction of azo dyes.** HUBBUCH and LOWY.—See IV. **B.p. of electrolytic caustic solutions.** MONRAD and BADGER.—See VII.

PATENTS.

Electric furnace. C. B. FOLEY, Assr. to C. B. FOLEY, INC. (U.S.P. 1,694,536, 11.12.28. Appl., 13.3.20. Renewed, 14.4.27).—A tapping device, with means for closing it, is located above the surface of the melt, when the furnace is in its working position. Below the normal level of the melt is a gas inlet. F. G. CLARKE.

Electric furnace. A. N. OTIS and C. L. IPSEN, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,696,728, 25.12.28. Appl., 10.9.25).—A heating resistor is hung in a series of depending loops from parallel bars extending across the furnace chamber, and having their ends secured in opposite walls.

J. S. G. THOMAS.

[Operation of] electric annealing furnaces of the resistance type. A.-G. BROWN, BOVERI & CIE. (B.P.

273,320, 23.6.27. Ger., 23.6.26).—Continuous current is supplied to the furnace at least until a desired temperature is attained, and is then automatically regulated so that, after the maximum permissible surface temperature of the material to be treated is reached, the energy supply is periodically reduced so as to be always equal to the energy loss from the furnace, together with that soaking into the material at that temperature.

J. S. G. THOMAS.

Electric furnaces for heat treatment and forging.

A. IMBERY (B.P. 303,222, 10.10.27).—Non-metallic heating elements, in the form of rods, are arranged in a refractory chamber near tubes extending through the chamber between the heating elements and receiving parts of the bars or rods to be heated. Electrical connexion with the heating elements is made by metal alloy terminals of low resistance.

J. S. G. THOMAS.

Induction electric furnace. High-frequency induction furnace.

E. F. NORTHRUP, Assr. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,694,791—2, 11.12.28. Appl., [A] 14.2.25, [B] 29.11.24. Renewed, [B], 28.7.28).—(A) In inductive heating free from inter-threading by transformer iron, using alternating currents within the range of frequency of generation by rotary, multipolar generators, the current path is confined to a single layer of turns about the charge, and the relation of the frequency to the diameter of the charge is such that the induced electromagnetic energy does not penetrate appreciably beyond the axis of the charge. (B) The loss of power in the inductor in apparatus free from transformer iron is rendered practically independent of the size of the inductor by providing a single inductor layer of greater conductor depth than thickness, and having a depth bearing radially a definite proportion to the diameter of the charge.

L. A. COLES.

[Seal for heater-leads of] electric furnaces. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of R. B. PRINDLE (B.P. 282,741, 12.12.27. U.S., 29.12.26).—Each opening in the furnace chamber for the heater-leads is provided with a glass sleeve surmounted by a metallic conducting cap to which the lead is connected. The sleeve is surrounded by a rigidly mounted protective member spaced apart from the glass and carrying the conductor from each conducting cap.

J. S. G. THOMAS.

Catalytic agent in storage battery. D. W. DAVIS, Assr. to A. L. NEW (U.S.P. 1,694,530, 11.12.28. Appl., 5.8.27).—Gases evolved from the electrolyte are caused to combine by means of a catalyst which is placed in such positions that at least one portion is unsubmerged when the battery is tilted or inverted.

F. G. CLARKE.

Production of storage-battery separator elements. N. FALLEK (U.S.P. 1,690,573, 6.11.28. Appl., 4.5.25).—A pulp of vegetable fibre and water is boiled, washed to remove dirt and impurities, and subjected to the direct action of steam. After boiling with caustic soda solution, it is washed to remove all traces of soda, mixed with a small quantity of pulverised rubber and sodium silicate, formed into sheets, and allowed to dry.

F. R. ENNOS.

Manufacture of cathodes for electric discharge tubes. VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G.

(B.P. 289,762, 18.11.27. Austr., 30.4.27).—An alloy of an alkaline-earth metal, *e.g.*, a barium-magnesium alloy, is decomposed *in vacuo* and partially or completely vaporised so that the vapour of the alkaline-earth metal is deposited on the outer surface of the core of the cathode which contains, at least on its surface, a substance which combines with the alkaline-earth metal to form an alloy or oxide. J. S. G. THOMAS.

Manufacture of cathodes for electric discharge tubes. M-O. VALVE Co., LTD., and J. W. RYDE (B.P. 302,503, 23.12.27).—A highly electropositive metal, *e.g.*, barium, is deposited upon a support, *e.g.*, tungsten, which has been exposed to cyanogen. J. S. G. THOMAS.

Manufacture of cathodes for discharge devices. ALLGEM. ELEKTRICITÄTS.-GES. (B.P. 286,650, 6.3.28. Ger., 7.3.27. Addn. to B.P. 268,815; B., 1928, 646).—A paste consisting of a metal oxide in a viscous fluid, *e.g.*, paraffin oil, sugar, and collodion solutions, is applied to an oxide base which is heated in a reducing atmosphere to a temperature at which the metal oxide is reduced to metal which is sintered upon the base. J. S. G. THOMAS.

Electron-discharge device. K. II. STARK, ASSR. to SPERRY GYROSCOPE Co. (U.S.P. 1,695,746, 18.12.28. Appl., 13.5.19).—The device comprises a gas-tight container, an anode, and a cathode element formed of a photo-electric metallic coating sputtered upon a glass member, a discharge-controlling device, and a spark-gap for causing the cathode to emit electrons. J. S. G. THOMAS.

Electron-emitting element [coating]. L. McCULLOCH, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,899, 18.12.28. Appl., 27.9.22).—A conductor coated with an aqueous solution of one or more compounds of at least one metal of the alkaline-earth group is heated in carbon dioxide to ignition temperature. J. S. G. THOMAS.

Alloy base for oxide-coated cathodes. G. W. HALLOCK, ASSR. to WESTINGHOUSE LAMP Co. (U.S.P. 1,695,845, 18.12.28. Appl., 27.10.27).—The cathode core consists of a nickel-silicon alloy. H. ROYAL-DAWSON.

Activation of [tungsten] filaments. G. D. O'NEILL, ASSR. to WESTINGHOUSE LAMP Co. (U.S.P. 1,695,819, 18.12.28. Appl., 1.4.27).—Thoriated tungsten filaments are activated by intermittently incandescing them in an atmosphere containing carbon. H. ROYAL-DAWSON.

Protection from oxidation of caps consisting of brass or similar alloys, such as bronze, for electric incandescence lamps etc. GEN. ELECTRIC Co., LTD., ASSEES. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 289,440, 25.4.28. Ger., 27.4.27).—The caps are heated at 500–600° in a reducing atmosphere between the process of cleaning by pickling and the setting of the cement. M. E. NOTTAGE.

Centrifugal apparatus for recovery of vapours from air drawn off from chromium[plating] baths. W. G. POETZSCH (B.P. 298,226, 13.9.28. Ger.,

6.10.27).—The air current is caused to pass through one or more systems of vanes arranged in the exhaust, and to rotate either by the air current itself or mechanically. J. S. G. THOMAS.

[Plates for] secondary or storage batteries. CHLORIDE ELECTRICAL STORAGE Co., LTD. From ELECTRIC STORAGE BATTERY Co. (B.P. 302,472, 21.11.27).

Argon-mercury discharge tubes. RAINBOW LIGHT, INC., and R. R. MACHLETT (B.P. 293,683, 23.9.27. U.S., 9.7.27).—See U.S.P. 1,680,271; B., 1928, 760.

Luminous electric discharge tube of high candle power. RAINBOW LIGHT, INC., ASSEES. of R. R. MACHLETT (B.P. 293,682, 23.9.27. U.S., 9.7.27).

Furnace regulation (B.P. 283,903).—See I. Carbonisation of marine algæ (B.P. 284,583). **Manufacture of hydrocarbons** (B.P. 301,949).—See II. **Preparation of articles for nickel-plating** (G.P. 451,620). **Leading-in wires** (U.S.P. 1,695,791).—See X. **Electrodeposition of rubber** (B.P. 303,214).—See XIV. **Pasteurisation of milk** (U.S.P. 1,695,300).—See XIX.

XII.—FATS; OILS; WAXES.

Formation of glycerides and their isomerides. I. T. L. GARNER (J.S.C.I., 1928, 47, 278–279 r).—The effect of various gases and reduced pressure on the preparation of glyceride from oleic acid was found to be largely mechanical with the exception that sulphur dioxide gave the isomeride trielaidin. The action of sulphur dioxide is briefly discussed and the synthesis of trielaidin with this gas is shown to be reversible. The m.p. of the trielaidin, 39°, was somewhat higher than that found by previous investigators.

Identification of fats and mixtures of fats. I. B. LUSTIG and G. BOTSTIBER (Biochem. Z., 1928, 202, 81–90).—Illustrations show the difference in microscopic appearance of cocoa butter, hardened train oil, palm-kernel fat, beef tallow, etc. when a drop of the melted fat is pressed between microscope slides and cooled in ice. Tables show for a variety of such fats the different degree and colour of fluorescence, together with its variation in different solvents, and, according to the manner of preparation of the fat, also the amount, iodine value, and m.p. of the solid fatty acid fraction, together with the normal variations of these in different samples of the fats. P. W. CLUTTERBUCK.

Oil seeds from British Guiana. [Crabwood seed oil; awarra palm oil.] (Bull. Imp. Inst., 1928, 26, 411–416). I. Crabwood seeds and oil (*Casapa guianensis*, Aubl.) have been examined. The kernels, which form 70.7% of the seeds and contain 6.4% of moisture, yield 55% of a cream-coloured oil, partly liquid and partly solid, having an intensely bitter taste. Oils extracted in British Guiana and at the Imperial Institute had, respectively, d_{4}^{20} 0.8689, 0.8572, n_D^{20} 1.4590, 1.4560, solidifying point of fatty acids 37.8°, 35.8°, acid value 36.9, 75.8, saponif. value 198.5, 197.4, iodine value (Hübl, 17 hrs.) 57.3, 64.5, unsaponifiable matter 1.1%, 0.7%. The oil is suitable only for soap making and possibly as a germicide. The residual cake

is useless as a feeding-stuff on account of the bitter taste.

II. The oil (45.5%) of awarra palm fruits (*Astrocaryum*, sp.), extracted by light petroleum from the pericarp, was orange-red and partly solid at 15.5°, with a slight odour resembling that of palm oil. The kernels yielded 36.3% of a cream-coloured hard fat with a faint odour of coconut oil. The oils from the pericarp and kernel, respectively, had d_{15}^{20} 0.8573, 0.8660, m.p. —, 30.8°, n_D^{20} 1.458, 1.4505, acid value 38.4, 0.4, saponif. value 195.8, 241.9, iodine value (Hübl, 17 hrs.) 68.0, 14.6, unsaponifiable matter 0.5%, 0.5%, soluble volatile acids 0.4, 2.4, insoluble volatile acids 0.2, 6.8, solidifying point of fatty acids 36.7°, 27.0°. The residual meal contains less protein than palm-kernel cake.

E. LEWKOWITSCH.

New oil-seed from Brazil [Arara nuts] (Bull. Imp. Inst., 1928, 26, 416—418).—Arara nuts (*Joannessia heveoides*) contained 44.8% of kernel which, on extraction with light petroleum, yielded 58.6% of a pale yellow liquid oil with a pleasant nutty odour having d_{15}^{20} 0.9239, n_D^{20} 1.467, acid value 2.1, saponif. value 188.5, iodine value (Hübl, 17 hrs.) 129.8, unsaponifiable matter 0.48%. A film of oil exposed on glass dried in 11 days (linseed oil, 8 days). The cream-coloured residual meal had a faintly bitter taste, contained substances of an alkaloidal nature, and gave on analysis: moisture 8.0%, crude proteins 47.4%, fat 0.7%, starch etc. (by diff.) 25.1%, crude fibre 6.5%, ash 12.3%, nutrient ratio 1:0.56, food units 145. Both meal and oil may possess toxic properties; the oil has semi-drying properties, and is soluble for soap-making.

E. LEWKOWITSCH.

The Wijs method as the standard for iodine absorption. J. J. A. WIJS (Analyst, 1929, 54, 12—14, and Chim. et Ind., 1928, 20, 1043—1044).—The criticism of Schmidt-Nielsen that substitution as well as addition takes place in the Wijs method for determining iodine absorption is refuted, the break required by Schmidt-Nielsen as showing a cessation of iodine absorption being in fact present. Further, Weiser and Donath's failure to obtain theoretical values with the Wijs reagent is shown to be due to the fact that their solution contained free iodine trichloride owing to 7.2 instead of 10.2 g. of iodine being used for 9.4 g. of iodine trichloride. The reagent should be made by dissolving about 9 g. of iodine trichloride in 1 litre of acetic acid of at least 99% concentration, or in a mixture of 300 c.c. of carbon tetrachloride and 700 c.c. of acetic acid. In exactly 5 c.c. of this solution the halogen content is determined, the bulk of the solution treated with 10 g. of pulverised iodine, and when nearly all is dissolved the halogen content is again determined on 5 c.c., and as soon as this is one half the previous value the solution is filtered into a stoppered bottle. No oxidisable matter must be present in the acetic acid or carbon tetrachloride, and the high coefficient of expansion by heat must be allowed for. Not more than 30% of the halogen present should be absorbed by the fat or oil.

D. G. HEWER.

Composition of Italian beeswax. D. CORTESE (Atti II Cong. Naz. Chim. pura appl., 1926, 1347—1352; Chem. Zentr., 1928, ii, 678).—Values are: d 0.9597—

0.9680, m.p. 63—64°, f.p. 60—61°, refractive index (Zeiss butyrefractometer, 70°) 27—30 (calc. at 40° 42.9—45.9), acid value 19.03—20.92, saponif. value 92—96.2, ester value 72.20—77.13, iodine value 8.0—10.3.

A. A. ELDRIDGE.

Emulsions of fatty substances and of hydrocarbons and their industrial applications. L. MEUNIER (Chim. et Ind., 1929, 21, 3—19).

Transformation of fatty acids. STADNIKOV and others. **Water-miscible mineral oil preparations.** HART.—See II. **Dental materials.** COLEMAN.—See X.

PATENTS.

Manufacture of fatty acid and soap derived from mineral oil. Manufacture of mineral oil derivatives. G. ALLEMAN, ASSR. to SUN OIL CO. (U.S.P. 1,694,461—2, 11.12.28. Appl., [A] 18.7.25, [B] 26.3.27).—(A) A mixture of fatty acids derived from mineral oil is claimed, the mixture having m.p. below —30°, being insoluble in water, practically free from mineral oil and petroleum resins, and readily converted into soaps resembling those of animal and vegetable origin, the soaps being miscible in all proportions with mineral oil and water and capable of forming homogenous emulsions with mineral oil and water together. (B) A water-soluble soap practically free from mineral oil and containing a reduced petroleum resin content is separated from an emulsion of soap, petroleum resin, and mineral oil, and is subsequently converted into a soap insoluble in water by double decomposition with a suitable metal salt.

L. A. COLES.

Removal or elimination of fatty acids, resins, and bitter and mucous substances from oils and fats. K. F. WILHELM (B.P. 289,801, 24.1.28. Ger., 2.5.27. Addn. to B.P. 226,767; B., 1925, 411).—To the solvent used in the process of the prior patent is added an equal amount of another solvent, which will dissolve fat, but which will not combine with the de-acidifying liquid, e.g., trichloroethylene, methylene chloride, or benzene. The reagent is completed by the addition of 1% of Glauber's salts, calculated on the weight of the crude oil to be treated.

E. HOLMES.

Treatment of cashew nut-shell and allied oils. HARVEL CORP., INC., ASSEES. of M. T. HARVEY (B.P. 275,574, 23.6.27. U.S., 6.8.26).—Non-drying cashew nut-shell oil, consisting mainly of anacardic acid and cardol, may be converted into a drying oil by interaction with copper, nickel, monel metal, lead, magnesium, calcium, or manganese, if necessary at elevated temperatures and pressures, followed by the addition of another vegetable oil such as linseed oil. Copper itself is the most effective reagent, although the oxides, hydroxides, carbonates, oleates, or stearates of any of these metals may be used. The resulting products are of use as impregnating and coating materials for waterproofing and insulating purposes.

E. HOLMES.

[Bar-forming device for] soap-making. L. H. NELLES (B.P. 299,960, 21.9.27).

Lubricating oils (B.P. 295,230).—See II. **Degreasing raw wool** (B.P. 300,941). **Compositions from cellulose derivatives** (U.S.P. 1,690,515).—See V.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Ageing processes of paints. A. V. BLOM (Farben-Ztg., 1929, 34, 892—895).—The ageing of paints is discussed with special reference to changes in colloid systems. Outside influences affecting a paint film at early stages of its life control its subsequent behaviour. Under unfavourable conditions of application etc. a paint very quickly enters the "danger zone" in which it is readily vulnerable to circumstances that would not affect the same paint applied satisfactorily. The chief properties, deterioration in which connotes "ageing," are elasticity, hardness, and adhesion. An accelerated test is detailed wherein dry paint films on suitable metal strips are tested in an extensometer. The film is observed under a lens and the experiment is stopped at the first appearance of cracks, when the extension of the strip is recorded. The particular form of cracking is noted and the film is rated by reference to a graded series of "standard cracking types," photomicrographs illustrating which are discussed. The "degree of cracking" and the maximum extension of the film must both be taken into consideration, since both adhesion and elasticity are concerned in the test. Hardness is measured by scratching tests carried out on the same films. Accelerated ageing is accomplished by subjecting the films to continuous cooling, intermittent cooling in dry and moist atmospheres, continuous heating and intermittent heating. The conclusions to be drawn from extensometer tests on these "aged" films at elevated, low, and ordinary temperatures are indicated.

S. S. WOOLF.

Behaviour of white pigments [and paints] under ultra-violet light. C. P. VAN HOEK (Farben-Ztg., 1929, 34, 833—834, 895—896, 951—956, 1006—1009).—Mainly a digest of the literature on the subject. The destructive influence of ultra-violet rays on paints is discussed with reference to the absorbing and transmitting powers of the various white pigments.

S. S. WOOLF.

Photometric determination of the colour of various lamp-blacks. L. HOCK (Kautschuk, 1928, 4, 266—268).—There is a close relationship between the specific surface, *i.e.*, the total surface exposed by the particles in unit weight, of different finely-divided forms of amorphous carbon and their blackness. The specific surface was determined by the heat of wetting with benzene. Blackness was determined by mixing with 100 pts. of lithopone in a medium, preferably rubber, and measuring the intensity of the light reflected from a smooth surface by means of a polarisation photometer or a leucometer.

D. F. TWISS.

Viscosity of cellulose acetate. DESCHIENS.—See V. **Selenium-red in rubber.** DITMAR.—See XIV.

PATENTS.

[Manufacture of] ink. B. KALISCHER (B.P. 303,200, 30.9.27).—An ink which penetrates almost immediately into ordinary paper contains water and/or glycerol or glycol together with 2—10% of an ester of a polyhydric alcohol, *e.g.*, glycol or glycerol acetate. Gelatin, dextrin, gum arabic, etc. may also be added to vary the fluidity of the liquid.

L. A. COLES.

Uninflammable nitrocellulose lacquers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,390, 15.9.27).—Alkylene chlorides are introduced into nitrocellulose lacquers in amount not less than that of the nitrocellulose solvents already present. S. S. WOOLF.

Manufacture of plastic compositions, lacquers, filling or priming compositions, etc. from nitrocellulose. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,961, 22.9.27).—Plasticising agents which do not volatilise and thereby render the products permanently elastic, comprising phthalic acid esters having one carboxyl group esterified with the radical of an aromatic or hydroaromatic alcohol and the other with the radical of an aliphatic or aromatic alcohol, *e.g.*, *n*-propyl benzyl phthalate, cyclohexyl benzyl phthalate, isoamyl cyclohexyl phthalate, etc., are used in the manufacture of the compositions. L. A. COLES.

Refining of wood rosin. G. E. JENKS, Assr. to HERCULES POWDER CO. (U.S.P. 1,694,179, 4.12.28. Appl., 15.5.23).—Wood rosin is externally heated *in vacuo* while superheated steam is injected into the body of the rosin. A portion of the distillate is condensed at not less than 174°.

S. S. WOOLF.

Manufacture of condensation products from phenols and aldehydes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,609, 14.7.27).—Phenols are condensed with formaldehyde etc. in presence of strong aliphatic or alicyclic bases, *e.g.*, *n*-butylamine, dicyclohexylamine, β -hydroxyethylcyclohexylamine, di-(β -hydroxyethyl)butylamine.

C. HOLLINS.

[Phenol-formaldehyde] condensation product. E. S. HOLE (B.P. 302,098, 20.6.27).—Water-soluble phenol-formaldehyde condensation products are obtained by boiling the reagents in the presence of a small quantity of an alkaline condensing agent (preferably an alkali hydroxide) until the requisite viscosity is attained, when water is added, the mixture is cooled, and a further quantity of water containing a suitable salt of chromic acid is added. The incorporation of alcohol and/or ammonia into the boiling mixture opposes any tendency to precipitation of the final water-soluble product.

S. S. WOOLF.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 278,698, 27.9.27. Ger., 8.10.26. Addn. to B.P. 258,289; B., 1928, 376).—The duration of the heating necessary to harden the condensation products is reduced by the addition, during or after evaporation, of substances giving acids during the heating (*e.g.*, magnesium chloride, formic esters, formamide).

B. FULLMAN.

Production of artificial masses. I. G. FARBENIND. A.-G. (B.P. 282,635, 29.10.27. Ger., 23.12.26).—Soluble formaldehyde-carbamide (etc.) products are mixed with fillers, such as wood-meal, lignin, etc., which have been impregnated with carbamide, thiocarbamide, phenol, etc., or non-sticky compounds of these with formaldehyde etc. (excluding soluble form-

aldehyde-carbamide products). The mixture is dried and hardened in the press. C. HOLLINS.

Composition from cellulose derivatives (U.S.P. 1,690,515).—See V. **Cashew nut-shell oil etc.** (B.P. 275,574).—See XII. **Perishable goods** (B.P. 302,447).—See XIX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Latices of *Euphorbia tirucalli*, *E. candelabro*, and *E. abyssinica*. E. DE'CONNO, S. CAPALBI, and L. FRUITIER (Annali Chim. Appl., 1928, 18, 540—549).—The latices of these plants, in the order named, which grow profusely in Erythrea, have the following percentage compositions: water 46.75, 30.05, 49.60; gum 7.25, 8.30, 8.44; euphorboresene 25.50, 40.50, 28.39; euphorbone 9.25, 10.20, 10.23; euphorbinic acid traces in each case; fat trace, —, 1.03; insoluble residue (inorganic) 7.40, 7.30, 0.29; (organic) 2.70, 2.50, 1.73. In *E. tirucalli* the gummy matter is perfectly elastic and resembles caoutchouc in its physical properties, the euphorboresene is soft and pale yellow, and the euphorbone has m.p. 112—113°; in the other two species the gummy matter is only slightly elastic and resembles Para rubber, the euphorboresene is solid and dissolves in carbon disulphide and acetic acid, and the euphorbone has m.p. 114—115°.

T. H. POPE.

Evaluation of anti-oxidants [for rubber]. T. L. GARNER (India-Rubber J., 1929, 77, 31).—The relative effect of various anti-oxidants is influenced considerably by the type of mixture used; for general comparative purposes the composition rubber 100, sulphur 3.5, zinc oxide 3, diphenylguanidine 1, and anti-oxidant 0.5—1 is recommended. The protective effect of anti-oxidants is much more marked with over-vulcanised rubber. Most anti-oxidants cause discoloration of white articles, and their presence may lead to staining of black goods.

D. F. TWISS.

Selenium-red [in rubber]. R. DITMAR (Gummi-Ztg., 1929, 43, 759—760).—Selenium-red forms a colloidal dispersion in rubber and increases the plasticity of the mixing. It has strong pigmenting action for hard or soft rubber vulcanised by the hot process, but is discoloured on vulcanisation with sulphur chloride. Its colour is not affected by organic accelerators and anti-oxidants.

D. F. TWISS.

Causes of variation in plasticity [of rubber]. REPORT FROM Ceylon Rubber Research Committee (India-Rubber J., 1929, 77, 32—36).—The addition of sodium bisulphite to latex before coagulation leads to a marked loss of plasticity in the finished rubber; the plasticity is also notably reduced by prolongation of the period between coagulation and crêpeing. Dilution of the latex before coagulation has little influence on the plasticity, but excessive machining of the coagulum on the crêpeing rolls increases plasticity, as also does drying the crêpe rubber in hot air. D. F. TWISS.

Influence of zinc oxide on the coefficient of vulcanisation [of rubber]. S. A. BRAZIER and L. A. RIDGWAY (J.S.C.I., 1928, 47, 351—356 T, 368 T).—The

effect of the combination of sulphur with zinc oxide on the coefficient of vulcanisation of various rubber mixings containing organic accelerators and compounding ingredients has been studied for different temperatures and periods of vulcanisation. No general rules can be deduced from the results obtained, as, although an appreciable amount of zinc sulphide is formed in the presence of organic accelerators in common use in the rubber industry, the amount is dependent on a number of variables. The extent of zinc sulphide formation is also shown to affect appreciably analytical work where specifications limiting the coefficient of vulcanisation are concerned. The paper includes preliminary results of experiments using cadmium oxide, where a similar effect is noted.

Effect of temperature on the stress-strain properties of vulcanised rubber. A. A. SOMERVILLE and W. H. CORE (Trans. Inst. Rubber Ind., 1928, 4, 263—286, and Kautschuk, 1928, 4, 271—272).—In view of the spontaneous heating of some rubber goods, e.g., tyres, during service, fuller investigation of the alteration in physical properties with temperature change is desirable. Tests are now described with ring test pieces at temperatures from 0° to 100°; the characteristics examined include the stress-strain curve, fatigue, permanent set, and adhesion, the variable factors examined in addition to temperature including time of vulcanisation, proportion of sulphur and accelerator, and presence of an anti-oxidant. D. F. TWISS.

Analysis and classification of regenerates [reclaimed rubber]. E. LINDMAYER (Kautschuk, 1928, 4, 278—280).—Determination of the proportion of caoutchouc hydrocarbon in reclaimed rubber is effected by extracting a comminuted sample successively with acetone, aqueous potassium hydroxide, and concentrated hydrochloric acid, and determining the carbon and hydrogen content of the dry, purified residue by a combustion method using lead chromate. From the hydrogen found, the proportion of rubber C_5H_8 is estimated, any carbon in excess of the requirement for this purpose being regarded as present as carbon black or lamp-black. Mineral rubber is estimated by dissolving the chloroform extract of the material in carbon tetrachloride and comparing the colour with that of a solution of "mineral rubber" of known concentration in the same solvent. On an assumption that the reclaiming process is dependent on a conversion of vulcanised rubber, thus: $(C_5H_8)_6 \cdot S \cdot (C_5H_8)_6 \rightarrow (C_5H_8)_3 \cdot S \cdot (C_5H_8)_3 + (C_5H_8)_6$, the latter formulæ represent the portions of the product respectively insoluble and soluble in chloroform; the limit of the reclaiming process will therefore be represented by a conversion of about 48% of the vulcanised caoutchouc into a condition soluble in chloroform.

D. F. TWISS.

PATENTS.

Electrodeposition of rubber. S. O. COWPER-COLES (B.P. 303,214, 3.10.27).—In the electrodeposition of rubber, inclusion of gas bubbles in the anode deposit is prevented by employing a cathode or cathodes bearing perforations which permit the escape of bubbles to the

side remote from the anode and its rubber deposit. The current may be intermittent, the cathode or cathodes may be rotatable, and the anode or anodes may be immersed only for intermittent periods. D. F. TWISS.

Production of rubber coatings. K.D.P., LTD. (B.P. 277,375, 12.9.27. Ger., 10.9.26).—Latex, if desired in admixture with filling materials such as lamp-black, pigments, sulphur, etc., is sprayed on the base to be coated, e.g., metal, wood, stone, leather, etc., provision being made for evaporation of the water by using a hot, gaseous medium for the atomising process. Rubber layers so produced on different bases may, if desired, be united by compression and vulcanisation.

D. F. TWISS.

Accelerator of vulcanisation [of rubber]. GOOD-YEAR TIRE & RUBBER Co., Assecs. of L. B. SEBRELL (B.P. 278,689, 19.9.27. U.S., 8.10.26).—Vulcanisation is accelerated by a reaction product of a mercaptan and an amine other than guanidine; compounds of mercaptobenzthiazole with an aliphatic amine, e.g., a dialkylamine, are indicated. [Stat. ref.]

D. F. TWISS.

Recovery of rubber. W. H. YEANDLE, Asst. to INTER-CONTINENTAL RUBBER Co. (U.S.P. 1,695,676, 18.12.28. Appl., 13.12.27).—Vegetable matter containing rubber enclosed within the plant cells is permeated with steam, the pressure being then increased and suddenly reduced. The material being thereby disintegrated and the cells disrupted, the rubber is then separated.

D. F. TWISS.

Jelutong products and their production. E. C. R. MARKS. FROM BEECH NUT PACKING Co. (B.P. 302,350, 29.3.28).—Oxidation of jelutong is prevented by introduction of an ionisable anti-oxidant, e.g., ammonium phosphate, tartrate, acetate, etc., or sodium phosphate or phosphoric acid. The jelutong may either be heated in an aqueous solution of the anti-oxidant, or the aqueous solution may be mixed into the molten jelutong.

D. F. TWISS.

XV.—LEATHER; GLUE.

Determination of tannin [in beers and worts]. HARTONG.—See XVIII.

PATENTS.

Tanning. P. PAVLOVITSCH (B.P. 302,408, 20.9.27).—Unaired hides are plumped with a tanning solution (e.g., quebracho liquor, *d* 1.075), which may be at 30–37°, and have p_H 6–12 (preferably obtained by adding sodium hydroxide), according to the tanning material used. After complete penetration of the hides (3–10 days) they are transferred to tan liquors of p_H 2–5. [Stat. ref.]

D. WOODROFFE.

Moulding of casein under pressure. P. HAESSLER (B.P. 302,545, 21.5.28).—Powdered casein, subjected in the pan of a spray-casting machine to a pressure of, e.g., 150–400 atm. at about 90–110°, is forced through a sieve and thence through an injection nozzle into a mould, where it is exposed to high pressure for a short time.

L. A. COLES.

Tanning agent (B.P. 272,541).—See XXIII.

XVI.—AGRICULTURE.

Catalytic properties of soils. K. SCHARRER (Z. Pflanz. Düng., 1928, 12A, 323–329).—The published literature of the catalytic properties of soils toward hydrogen peroxide and iodides is summarised and discussed, and the essential soil factors concerned are differentiated in the two cases.

A. G. POLLARD.

Properties of heavy alkaline soils containing different exchangeable bases. A. F. JOSEPH and H. B. OAKLEY (J. Agric. Sci., 1929, 19, 121–131).—Clays saturated with different bases exhibit very divergent physical and chemical properties. The commonly accepted clay-like properties are shown most definitely by lithium, sodium, and magnesium clays. The plasticity of a soil bears no relationship to the proportion of very fine material (remaining suspended in a 10 cm. column for 14 days) it contains. In base-exchange properties potassium and sodium clays resemble each other much more than calcium clay, but differ considerably in plasticity and permeability. Comparison of the absorption of calcium, sodium, and potassium by percolation with solutions containing two-base mixtures showed calcium and potassium to be absorbed in equivalent amounts, whilst only one sixth of the equivalent amount of sodium was absorbed.

A. G. POLLARD.

Dispersion and mechanical analysis of heavy alkaline soils. A. F. JOSEPH and O. W. SNOW (J. Agric. Sci., 1929, 19, 106–120).—Examination of soil fractions, of smaller ranges of mean diameter than the customary "clay" fraction, shows that after a single dispersive treatment the fractionation is affected by the nature of the bases with which the soil is saturated. The proportion of very fine particles ($< 0.5 \mu$) cannot be correlated with other important soil properties, and gives no measure of the colloidal properties of soil when the pipette method of analysis is used. Other conditions being similar, "sodium soils" show better dispersion than "ammonium soils." In no case examined was the "clay" fraction influenced quantitatively by pre-treatment with hydrogen peroxide. In the decantation process using sodium carbonate for dispersion, pre-treatment with acid did not affect the number of decantations required nor the proportion of clay determined. It is impossible to disperse soils in a single treatment as required for the pipette method, and for this process sodium carbonate gives better dispersion than ammonia. In Sudan soils decantation methods with sodium carbonate are recommended, with the omission of the hydrogen peroxide-hydrochloric acid treatment.

A. G. POLLARD.

Soil examination by means of conductivity measurements. W. BENADE (Z. Pflanz. Düng., 1928, 12A, 293–309).—Electrical conductivity measurements of aqueous suspensions of soils during the growth of seedlings mark the period of the intake of nutrients by remaining practically constant. Control soils without seedlings show a steady rise in conductivity with time. This increased conductivity is accompanied by a change of soil reaction towards the alkaline side. Conductivity of soil suspensions is influenced by the length of the dis-

solution period, atmospheric carbon dioxide, and the activity of micro-organisms. Soils which have been once extracted with water, filtered, and resuspended in water show an increased conductivity. The new value is characteristic for each soil, and is relatively greater for clays than for sandy soils. Mathematical relationships are obtained to express changes in conductivity in soils with continuous leaching. Soils from which practically all soluble salts have been leached show a general parallelism between conductivity and humus content. A. G. POLLARD.

Liming as a factor in the amelioration of deteriorated tropical soils. P. E. TURNER (J. Agric. Sci., 1929, 19, 83—89).—Liming trials with acid soils showed that finely-ground limestone was more effective than slaked lime as an ameliorant. Large single dressings were more quickly responded to than smaller annual dressings, there being little penetration of lime below the depth of cultivation. A satisfactory dressing must render the soil neutral in reaction and raise the degree of saturation to 80%. A. G. POLLARD.

Lime requirement of soils. S. GOY (Z. Pflanz. Düng., 1928, 12A, 317—318).—A brief outline of the chemical methods of examining acid soils, and their significance. A. G. POLLARD.

Rapid electrometric method for measuring "lime requirements" of soils. F. HARDY and A. H. LEWIS (J. Agric. Sci., 1929, 19, 17—25).—The soil sample (10 g.), is mixed with 40 c.c. of 0.2 *M*-calcium chloride solution (neutralised to p_H 7.0 with bromothymol-blue) and titrated with 0.03*N*-lime water in successive portions of 5 c.c. with shaking for 3 min. between each addition until p_H 7.0 is passed. From a curve showing the changes of p_H with lime added the exact amount of lime required to produce p_H 7.0 is determined. Comparison with the Hutchinson-MacLennan method (B., 1915, 565) shows the new method to be more rapid and reliable. A. G. POLLARD.

Determination of harmful soil acidity. B. TACKE and T. ARND (Z. Pflanz. Düng., 1928, 12A, 362—390).—Of the total soil acidity, the active and the base-exchange acidity are injurious to plants. The different sensitivities of various plant species to acidity cannot be fully described in terms of soil acidity alone. It is probable that the neutral point in respect of plant physiology is the same as the chemical neutral point. For the effective determination of injurious acidity, soils should be exhaustively extracted with neutral salt solutions by percolation. A shorter and rapid process consists in shaking soil for 1 hr. with *N*-potassium chloride and calcium carbonate. The carbon dioxide evolved is a measure of the injurious soil acidity. Both organic and mineral soils limed on this basis have a reaction of p_H 7. A. G. POLLARD.

Comparison of methods for determining the saturation capacity of soils. P. A. KUTSCHINSKY (Z. Pflanz. Düng., 1928, 12A, 392—411).—The methods of Hissink, Kappen, and of Gedroiz for determining the exchangeable bases in soils (*S*) give results in close agreement. For carbonate-free soils, Kappen's method

is the most rapid. The determination of the value ($T - S$) (Hissink) is unsatisfactory in soils of high p_H value, or of considerable buffer capacity, owing to the turbidity of the liquid to be titrated. Duplicate tests, however, show moderately close agreement. The value ($T - S$) can be calculated as $6.5 \times$ hydrolytic acidity (Kappen). The lime required to bring an acid soil to a definite p_H value can also be calculated from the hydrolytic acidity (Kappen), using the factors 3 for p_H 7.0, 4 for p_H 7.5, 5 for p_H 8.0, and 6.5 for p_H 8.5. Of the methods for determining the degree of saturation (*V*), those of Kappen and Gehring show closest agreement with each other and with field-trial results. Hissink's method gives low results and indicates only 50% of the saturation of alkali soils. Gehring's method is advantageous in that the final saturation of the soil with bases takes place at practically the same soil reaction as obtains under natural conditions. Kappen's method is quicker and more convenient in practice.

A. G. POLLARD.

Comparison between the culture methods of Mitscherlich and Wiessmann [for determining nutrient values of soils]. W. U. BEHRENS (Z. Pflanz. Düng., 1928, 12A, 412—415).—The basis of the methods is examined mathematically from the point of view of the dependence of the constants of the growth curves on the nature of the soils and experimental conditions. In Wiessmann's method the constants refer to one experiment only; those of Mitscherlich are of general application. The accuracy of the results of the pure sand cultures in Wiessmann's method is of vital importance, since they control the corrected values for plant growth in the experimental sand-soil cultures. A. G. POLLARD.

Influence of the carbon : nitrogen ratios of organic material on the mineralisation of nitrogen. H. L. JENSEN (J. Agric. Sci., 1929, 19, 71—82).—The relationship between the extent of nitrification of various organic materials in acid and alkaline soils to the carbon : nitrogen ratios is examined. In acid soil accumulation of nitrate and ammonia took place only when pea-pod meal with a C : N ratio of 13.3 was used. In alkaline soil materials with a C : N ratio less than 26 : 1 were not nitrified. Nitrification was the more rapid as the ratio narrowed below the limiting value. The residual unnitrifiable nitrogen amounted to 1.5—2.2% of the original material, and was greater with highly nitrogenous matter. All materials examined increased the α -humus content of the soil, but to varying extents. Alkaline soil favoured the production of α -humus from plant residues, but not from farmyard manure. The decomposition of straw, sweet clover, lupin, and farmyard manure is associated with their lignin contents, since they increased the nitrogen and methoxyl content of the humus, but the dried mycelium of *Polyporus* contained a "humic acid" fraction of high nitrogen content, but without methoxyl, which persisted in the soil. A. G. POLLARD.

Determination and characterisation of organic substances in soil. U. SPRINGER (Z. Pflanz. Düng., 1928, 12A, 309—317).—Existing methods for examining soil organic matter are discussed. Oxidation processes for determining organic matter may give fictitious

values if the usual relationship, $\text{humus} = 1.724 \times \text{carbon}$, is adopted. From 40 to 70% of the soil organic matter is humified, the balance being mainly cellulose, lignin, and pectin, with carbon contents of 44, 56, and 40%, respectively, and the humus content is more accurately regarded as $2 \times \text{carbon}$. Wet combustion methods for this type of analysis are recommended. In alkaline extraction processes for determining humified material values depend on concentration and temperature of the solvent and the period of extraction. Non-humified material may be partially extracted. Use of ammonia extraction leads to difficulty in the separation of deflocculated clay from the extract. 50% pyridine is not a specific solvent for humus. Where alkaline-extracted humus is determined by oxidation with permanganate, results vary somewhat with the concentration of permanganates used and the period of boiling. The ratio humus-carbon:total carbon is valuable in the characterisation of soil types; and in the examination of soil profiles the alkali-extractable carbon expressed as a percentage of the total carbon content yields information as to the process of soil formation.

A. G. POLLARD.

Relationship between stability of soil structure and its colloid and sand content. A. T. TIULIN (Rep. Dept. Agric. Chem., Perm, 1928, 2, 22; Bied. Zentr., 1929, 58, 7—8).—The comparison of colloid and sand contents in several soils with their stability of structure showed that the stability was related directly to the colloid and inversely to the sand content. Four tschernozem and two podsol soils were compared, stability of structure being measured directly by washing samples with a constant pressure of water, and colloid content estimated from adsorption capacity. The product of adsorption capacity and sand content varied as between the two types of soil, but was roughly constant within a type. Sodium acetate treatment was found to increase the amount of colloid and decrease sand; ferrous sulphate had the opposite effect.

H. L. RICHARDSON.

The second approximation of the theory of growth factors. E. A. MITSCHERLICH (Z. Pflanz. Düng., 1928, 12A, 273—282).—The theory of growth factors is extended to include the depression of crop yields resulting from the supply of nutrient in amounts greater than the optimum. Experimental work can be expressed in mathematical form. The growth-depression factor is influenced by the nature of the deficient nutrients and of the soil, the buffer capacity and water-retaining capacity of the soil, and by climatic conditions. Much of the experimental evidence of the opponents of the constancy of the growth factor is considered unsatisfactory owing to lack of consideration of the "depression factors."

A. G. POLLARD.

Sparingly soluble phosphates of physiological importance to plants. E. UNGERER (Z. Pflanz. Düng., 1928, 12A, 349—362).—The amount of phosphoric acid extracted from the tertiary phosphates $\text{Mg}_3(\text{PO}_4)_2 \cdot 22\text{H}_2\text{O}$, $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$, $\text{Ca}_3(\text{PO}_4)_2$, AlPO_4 , and FePO_4 is affected by the reaction of the extracting solution and the presence in it of electrolytes and univalent permutites. Neutral salts decrease the solubility

of iron and aluminium phosphate and increase the p_{H} value of the solution. Clay and potassium- and ammonium-permutites adsorb calcium and magnesium ions from the above phosphates, and an equivalent amount of phosphate appears in solution. In soils, iron and aluminium phosphates in the presence of neutral salts are involved in base-exchange activities, and titratable hydrated alumina is found in solution. The value of these phosphates as plant nutrients depends on the water and chalk content, and reaction of the soil. When the soil moisture rises to 90% of the maximum capacity, aluminium phosphate has a greater nutrient value than calcium monohydrogen phosphate. Soil reactions more acid than p_{H} 5 decrease the nutrient values of iron and aluminium phosphate.

A. G. POLLARD.

Physiology of urea in the higher plants. G. KLEIN (Z. Pflanz. Düng., 1928, 12A, 390—391).—Urea is present in plants in maximum amounts in seedlings, young buds, and leaves, and in active tissue. In old leaves and seeds none occurs. Urea in a nutrient medium for plants is partially decomposed outside the plant roots and partially absorbed and decomposed within the plant tissue, and the plant may be poisoned by ammonia produced. Urea in young plant tissue is to a large extent combined with aldehydes.

A. G. POLLARD.

The washing-out of chloride [added to soils]. H. J. WITTEVEEN (Landb. Tijdschr., 1928, 40, 528; Bied. Zentr., 1929, 58, 9—10).—In a field experiment in the wet winter of 1926—27, chloride added as heavy applications of 20% potash salts was soon washed out. Chloride was added at the rate of nearly 500 and 600 kg. per hectare, and samples were taken every four weeks to a depth of 20 cm. After two months only 100—130 kg. of chloride per hectare remained in this depth, and further washing out took place in the following months. It was concluded that by seed time most of the added chloride would be out of reach of all but deep-rooted plants, and thus would not be harmful.

H. L. RICHARDSON.

Influence of irregularities in the level of soil on its fertility. N. N. KOURTIAKOFF (Compt. rend., 1929, 188, 189—191).—Owing probably to the fact that carbonates are washed to a lower depth below depressions in a field than in the higher parts, the yield of barley is least in the portions of lowest level.

G. A. C. GOUGH.

Fertilising with iodine. E. G. DOERELL (Z. Pflanz. Düng., 1928, 12A, 344—349).—Application of iodine (as potassium iodide) at the rate of 3.2—4.3 kg. per hectare increased the yield of hops, but higher proportions (5.4 kg. per hectare) caused considerable crop reduction. In no case was the increased value of the crop as great as the cost of iodide used. The iodine present in Chilean saltpetre and in superphosphate is sufficient to show a small but definite effect in the crop. Iodides improve the colour of the hops and decrease the iodine number and tannin content.

A. G. POLLARD.

Field experiments with Zeotokol. E. BLANCK (J. Landw., 1928, 76, 317—326).—"Zeotokol" (powdered dolerite) applied at 5 and 10 cwt./acre had no good effect on growth or yield with sugar beet, nor any residual

value for oats in the following year. This result, agreeing with earlier pot experiments of the author, contradicts Popp's conclusion (1926) that Zeotokol improved yield and soil condition. His results are criticised on the basis of experimental error, whilst any beneficial action on soil condition could be secured more cheaply with other materials. H. L. RICHARDSON.

Sterilisation of green fodder by liquids. C. BRAHM [with G. ANDRESEN and R. PRILLWITZ] (Forts. Landw., 1928, 3, 769; Bied. Zentr., 1929, 58, 24—26).—Fingerling's suggestion for preserving fodder materials with dilute hydrochloric acid was tested, but it gave a product no better than ordinary silage at a greater cost. Analyses of the grass used, before and after treatment, and of the press liquors over a period of nine months, showed that the hydrochloric acid tended to disappear during storage, and was not able to prevent the occurrence of other processes than lactic acid fermentation, nor prevent the disappearance of lactic acid.

H. L. RICHARDSON.

Influence of clay on plant growth. E. BLANCK and H. KEESE (J. Landw., 1928, 76, 309—316).—In pot experiments with oats the addition of increasing proportions of clay to sand at first improved and then injured the plants. Quartz sand and a "pure clay" containing only traces of bases were used in mixtures of 10:0, 9:1, 8:2, 6:4, and 4:6, with the addition of a complete fertiliser. Height at harvest and dry weight were both greatest with the 9:1 mixture, about the same with either 10:0 or 8:2, and greatly reduced with 6:4 and 4:6 mixtures. Subsequently reaction measurements showed that whereas the sand was neutral the clay was acid (p_H 4.23); the injurious action of the clay was therefore attributed to its acid nature. The difference in the properties and action of individual clays was emphasised.

H. L. RICHARDSON.

Effect of sodium silicate in increasing the yield of barley. R. A. FISHER (J. Agric. Sci., 1929, 19, 132—139).—Sodium silicate increases the yield of barley most markedly in the absence of superphosphate dressings. Although the phosphate content of the barley plant is not notably increased by silicate manuring, the total phosphate removed by the crop is greatly increased and the silicate content of the plant ash is also increased. The higher yield of grain and straw following the use of sodium silicate can be quantitatively ascribed to the increased availability of soil phosphate under these conditions.

A. G. POLLARD.

A type of bacteria abundant in productive soils, but apparently lacking in certain soils of low productivity. H. J. CONN (N.Y. State Agric. Exp. Stat. Bull. No. 138, 1928, 26 pp.).—The organism (*Bacterium globiformis*), which consists of a rod in young cultures and a coccus in older ones, will not grow in certain unproductive soils which are acid, even after liming, unless addition is made of certain simple nitrogen and carbon sources, e.g., amino-acids, ammonium salts, or nitrates, and carbohydrates, polyhydric alcohols, or various organic acids even as simple as acetic acid. A further study of the nutritive requirements of the organism is being made in order to throw light on the unproductiveness of these soils.

F. R. ENNOS.

Lime status of soil in relation to an insect pest of sugar cane. P. E. TURNER (J. Agric. Sci., 1929, 19, 26—35).—The blight of sugar cane caused by the "frog-hopper" (*Monecphora [Tomaspis] saccharina*, Dist.) is associated with the acidity and calcium reserve of soils. The ratio exchangeable calcium : (clay + fine silt) averaged 0.28—0.63 in unblighted soils and 0.11—0.25 in blighted areas.

A. G. POLLARD.

Seed treatment for black-leg disease of crucifers. E. E. CLAYTON (N.Y. State Agric. Exp. Stat. Bull. No. 137, 1928, 58 pp.).—The efficiency of various methods of seed treatment for black-leg on cabbage and brussels sprouts were tested both in the greenhouse and in field plats. Dust disinfectants, e.g., copper carbonate, or the organic mercury disinfectants Semesan and Uspulun, or liquid chemical treatments, e.g., mercuric chloride, Germisan, etc., are considered unsatisfactory on account of their uncertain action or of their liability to injure the seed. The most effective control of the disease was obtained by hot water. Treatments of 25 min. at 50°, 10 min. at 55°, and 4½ min. at 60° were about equally effective; the lowest temperature is recommended since the chance of seed injury is least.

F. R. ENNOS.

Tar distillate trials in Kent and West Sussex in 1928. F. V. THEOBALD (J. Pomology, 1928, 7, 199—211).—Data are cited with reference to the effects of various tar distillate washes in freeing the following trees and bushes from certain blights, particularly aphid and capsid bugs: gooseberry, cob-nut, black currant, and apple. In the case of the apple tree, data are also given with reference to the effect of the spraying on the subsequent crop.

E. A. LUNT.

Plant colloids. XXIII. SAMEC.—See XVII. Tobacco products. POPP.—See XX.

PATENTS.

Products from marine algæ (B.P. 284,583).—See II. **Calcium nitrate-ammonium nitrate** (B.P. 301,486). **Calcium cyanamide etc.** (B.P. 279,419).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Plant colloids. XXIII. Soluble starches obtained by oxidation methods. M. SAMEC (Kolloidchem. Beih., 1929, 28, 155—165).—Methods of obtaining soluble starch by oxidation processes involving sodium peroxide, hydrogen peroxide, ammonium persulphate, potassium permanganate, sodium perborate, chlorine, and air in alkaline solution are described. 1% solutions of each of these preparations were made and the following properties were studied: viscosity, electrical conductivity, hydrogen-ion concentration, alkali-binding capacity, osmotic pressure, and behaviour towards a collodion membrane. The viscosity of all the preparations was lower than that of native starch and, although the viscosity of ordinary starch is raised by addition of bases, the soluble starches were unaltered, except for a few cases where a decrease was observed. The electrical conductivity of the soluble starches was about 10 times as great as that of native starch. This result, together

with the increase in potentiometrically active hydrogen ions and alkali-binding capacity, suggests that during oxidation some acidic groups are produced. The degree of dispersion of the soluble starches is higher. The phosphorus content remains unchanged.

E. S. HEDGES.

Effect of heat on tragacanth and its mucilage. L. F. GABEL (J. Amer. Pharm. Assoc., 1928, 17, 1206—1210).—The viscosity of mucilage of tragacanth prepared under different conditions has been examined. A boiling period of 1 or 2 min. produces an initial maximum viscosity which increases very markedly on ageing. A 3 min. boiling period produces a mucilage of high viscosity which, however, is not materially increased on ageing. The viscosity rapidly decreases with prolonged boiling, and previous heating of the powdered tragacanth causes a very high loss in the viscosity of the mucilage prepared from it. Deterioration on heating or drying over calcium chloride is partially due to chemical change, as is evidenced by the development of acidity.

E. H. SHARPLES.

Insect pest of sugar cane. TURNER.—See XVI.

PATENTS.

Saccharification of cellulosic materials. H. SCHOLLER (B.P. 273,317, 23.6.27. Ger., 23.6.26).—Diluted acid is forced through the cellulosic material heated under pressure, whereby the laevulose solution formed is immediately and continuously removed from the system and cooled to prevent decomposition. The yield is practically quantitative.

W. J. BOYD.

Esters of carbohydrates (B.P. 302,191).—See V.
Gums from tobacco (B.P. 302,863).—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Continuous pasteurisation of black beers. R. SELIGMAN (J. Inst. Brew., 1929, 35, 10—17).—Stouts or black beers are sterilised in a plate heat-exchanger through which they flow at high speed and with great turbulence between walls so close together that at no time is any particle $\frac{1}{8}$ in. distant from a heated surface. In the heating section of the exchanger the beers are raised in 20 sec. to 71°, at which temperature the yeast is instantaneously killed. At this temperature they re-enter the regenerative section in which they are pre-cooled to 40° by unpasteurised stout. After being cooled by water to 15° in the cooling section the beers pass into a tank in which they can be carbonated to the degree required. The use of the plate heat-exchanger is not an alternative to pasteurisation in bottle, as it gives a beer which is sterile when it leaves the machine, but is subject to the ordinary risks of the bottle and the bottling plant.

C. RANKEN.

Determination of tannin [in beers and worts]. B. D. HARTONG (Woch. Brau., 1929, 46, 11—15).—From a boiled aqueous extract of hops, previously extracted with ether, the adsorption of tannin was studied by titration with 0.01N-permanganate in presence of indigotin. From the results of titrations of such extracts before and after treatment with varying

quantities of adsorbents the adsorption curves (amount adsorbed per g. of adsorbent against amount unadsorbed per 100 c.c. of extract) were plotted. Norit gave a normal curve, and alumina one suggesting chemical combination, as already described by Wislicenus. Casein, hide powder, and gelatin gave parallel curves of regular form, but indicating relative weakening of adsorptive power from more dilute solutions. The adsorptive action of kaolin, asbestos, and cellulose pulp was negligible. The curves for the five active adsorbents examined were capable of extrapolation and at maximum adsorption met at the zero point, indicating complete adsorption of oxidisable matter. Gravimetric determination of tannin on three hop extracts by Chapman's cinchonine method indicated an equivalent of 45.0 for hop tannin as regards oxidation by permanganate. Tests on beers indicated that norit adsorbs matters other than tannin, but alumina and casein gave curves which on extrapolation showed the same amount of unadsorbable oxidisable matter. The authors therefore assume that these only adsorb tannin, and, since their adsorptive power appears lower in beer and wort, that the tannin in these liquids is adsorbed on the proteins. The tannin from the malt greatly exceeds that from the hops in normal beers, and in a case examined 80% of the tannin present before boiling was still present in the beer after 2½ months' storage. Since the proportion of tannin adsorbed from worts and beers by any definite proportion of casein appears to be constant, to avoid the construction of an adsorption curve for each determination, two proportions only are employed and empirical factors applied to the result. Two 50 c.c. portions of beer or wort are treated with 1 g. and 4 g. of casein, respectively, shaken every 5 min. for 1 hr. and filtered bright. A 5 c.c. sample with 100 c.c. of water and indigotin is titrated with 0.01N-permanganate, run in as a steady stream of drops, with constant shaking, till the colour changes. Several titrations are obviously necessary. Under these conditions, and using Hammarsten's caseinum purissimum, the proportions of total tannin adsorbed from worts and beers was 50% and 74%, respectively. By applying these factors the duplicate results so obtained agreed within about 3%.

F. E. DAY.

Cooling and drying of materials [yeast]. E. B. BROWN, Assr. to FLEISCHMANN Co. (U.S.P. 1,694,807, 11.12.28. Appl., 1.6.25).—A current of air at about 15.5° containing about 1.25 grains of moisture per cub. ft. is passed through a thick, comminuted mass of moist yeast at the rate of about 2 cub. ft. per min. per lb. of yeast.

W. J. BOYD.

Retting of flax (B.P. 302,300).—See V.

XIX.—FOODS.

Correlation of ash content of wheat and of flour. R. C. SHERWOOD and C. H. BAILEY (Cereal Chem., 1928, 5, 437—444).—Ash content of straight-grade flour is positively correlated, with a small proportion of exceptions, with the ash content of the wheat; the relation appears to be linear. Variations in weight per bushel do not cause variations in the relation between the ash content of the wheat and that of the flour. The ash

content of a highly refined middlings flour was correlated with the ash content of the wheat in the same degree as was that of the straight-grade flour. The ash content of a flour cannot be used as a measure of the percentage extraction of that flour unless the ash content of the wheat is known.

W. J. BOYD.

Aminodicarboxylic acid fraction in [wheat] gliadin. D. B. JONES and R. WILSON (Cereal Chem., 1928, 5, 473—477).—The greater portion of the glutamic acid was precipitated from the hydrolysate as the hydrochloride, and the remainder of the dicarboxylic acid fraction was obtained as the barium salts insoluble in 70% alcohol. From this glutamic acid was separated as the hydrochloride, aspartic acid as the copper salt, and hydroxyglutamic acid as the silver salt. The following yields were isolated: glutamic acid 43.0%, aspartic acid 0.5%, hydroxyglutamic acid 7.7%. Time of hydrolysis had no significant effect on the proportions isolated.

W. J. BOYD.

Viscosity of flour suspensions. G. VAN DER LEE (Cereal Chem., 1928, 5, 484—485).—Replying to Denham, Watts, and Scott-Blair (*ibid.*, 330), the author maintains his objections (B., 1928, 282) to the application of Einstein's equation for the viscosity of colloidal solutions to the viscosity of flour suspensions.

W. J. BOYD.

Determination of hydrogen-ion concentration of flour-water mixtures. P. HALTON and E. A. FISHER (Cereal Chem., 1928, 5, 445—460).—The electrometric method of determining p_H is much more reliable than the colorimetric method. The quinhydrone electrode gives results in agreement with those obtained by means of the hydrogen electrode if gold or gold-plated electrodes are used. With platinum electrodes errors in p_H of 0.4 may occur. Concordant p_H values are obtainable with centrifuged extracts and with decantates from suspensions, but filtered extracts usually give slightly low, and suspensions high, values. The p_H of flour extracts increases with increasing ratio of flour to water, and very slightly with increasing time of extraction, but the concentration- p_H curves differ widely for different flours. The p_H of flour extracts cannot be correlated with the p_H of the corresponding doughs. The apparent buffer action of a flour is $1\frac{1}{2}$ — $3\frac{1}{2}$ times that of the extract.

W. J. BOYD.

Staling and hydrogen-ion concentration [of bread]. L. P. KARÁCSONYI (Cereal Chem., 1928, 5, 477—481).—During ordinary keeping of bread at room temperature acidity remains constant or shows some decrease. The acidity of stale bread may increase during further storage owing to the activity of micro-organisms.

W. J. BOYD.

Volume displacement of salt-sugar solutions [for dough mixtures]. E. GREWE (Cereal Chem., 1928, 5, 470—472).—In order to facilitate the addition of the salt and sugar in the form of solution, in the standard baking test (cf. Blish, B., 1928, 463), data are supplied whereby the water contained in given volumes of solutions of different concentration can be ascertained.

W. J. BOYD.

Effect of variation in the method of manufacture on the baking quality of dry skim milk. E. GREWE and G. E. HOLM (Cereal Chem., 1928, 5, 461—469).—Baking tests were carried out on three flours, from hard spring wheat, hard winter wheat, and soft winter wheat, using six samples of dry skim milk prepared by the spray process, but held for 30 min. at 50°, 63°, 73°, 83°, 93°, and 100°, respectively, before drying. The greatest improvement was obtained with the soft winter wheat flour and the least with that from hard winter wheat (cf. Grewe, B., 1928, 798). The use of dry skim milk increases the range of permissible fermentation times. The best baking results were obtained with the samples held at 73°, 83°, 93°, and 100°, which showed little variation among themselves, and the poorest results in all-round properties were obtained with that held at 50°.

W. J. BOYD.

Gasometric determination of carbon dioxide [in baking powders, flours, etc.] by the Chittick method. R. HERTWIG and J. S. HICKS (Cereal Chem., 1928, 5, 482—484).—In the Chittick method (A.O.A.C. Book of Methods, 1925, 305) for the gasometric determination of carbon dioxide in baking powder, self-raising flour, etc., the omission of a correction for the influence of the vapour tension of dilute sulphuric acid on the carbon dioxide volume reading may cause considerable errors.

W. J. BOYD.

Chemistry of sour milk. L. I. VAN SLIKE (N.Y. State Agric. Exp. Stat. Bull. No. 140, 1928, 14 pp.).—Experiments in connexion with the factors involved in the manufacture, ripening, and digestion of cottage cheese, the chemical changes taking place in certain constituents of milk at different stages of souring, and the determination of lactic acid in sour milk are summarised.

F. R. ENNS.

Alkali number of milk ash of various animals. A. SCHNECK (Milch. Zentr., 1928, 57, 373—379).—The alkali number ($K_2O : Na_2O$ ratio) for the ash of milk of various animals differs according to the type of food normally consumed. In any one class of animal it varies with the stage of lactation and the method of feeding, which probably accounts for the variation in the available figures for cows' milk. Except for the initial and final stages of lactation, the alkali number for cows' milk is more or less constant at 2.5 provided the food contains sufficient soda; further addition of soda in whatever form causes very little change in this ratio.

F. R. ENNS.

Factors affecting the composition of dates. M. T. FATTAH and W. V. CRUESS (Plant Physiol., 1927, 2, 349—355).—Dates grown in Mesopotamia contained more sugar and less moisture than the same variety grown in California. Differences in sugar content exhibited by different varieties can often be attributed to arrested ripening by drying on the tree. Unripe dates contain much sucrose; except in the Deglet Noor variety, this decreased greatly during ripening. Soluble tannin decreased markedly during ripening under various conditions.

CHEMICAL ABSTRACTS.

PATENTS.

Pasteurisation of milk. J. O. TEMPLETON, Assr. to ELECTROPURE CORP. (U.S.P. 1,695,300, 18.12.28. Appl., 13.3.25.).—In pasteurising milk by passing an electric current through it, a fluid of the same conductivity as milk is first passed between the electrodes until it acquires the required temperature, and then the milk is introduced.

W. J. BOYD.

Preservation of perishable goods. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 302,447, 17.10.27).—Eggs, or other perishable goods, are treated with an aqueous solution of a urea-formaldehyde condensation product, such as dimethylolurea, to which small amounts of organic solvents such as ethyl alcohol, glycol, or glycerol, and/or other preserving agents may be added, and the goods are then dried.

W. J. BOYD.

Kneading machines [for dough]. ARTOFEX ENGINEERING WORKS, LTD. (B.P. 300,960, 10.11.28. Switz., 21.11.27).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Incompatibility of sodium salicylate and sodium bicarbonate mixtures. J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1928, 17, 1203—1206).—The rapid darkening of mixtures of sodium salicylate and sodium bicarbonate in aqueous solution has been investigated. The reaction is accelerated by oxygen and does not occur with the sodium salts of the *m*- and *p*-hydroxybenzoic acids. Exposure to ultra-violet light or changing the alkali ion does not influence the rate of darkening.

E. H. SHARPLES.

Testing of Liquor cresoli saponatus D.A.B.6. W. MEYER (Chem.-Ztg., 1929, 53, 43).—For the detection of fish oils in lysols a rapid test is proposed: 3 pts. of the lysol, stirred with 7 pts. of boiling water, should develop during 3 min. no smell of fish oils, but should retain the pure odour of cresol, together with the typical "alkaline" odour.

C. HOLLINS.

Determination of chloral in syrup of chloral. P. FLEURY and M. MALMY (J. Pharm. Chim., 1928, [viii], 8, 537—542).—A critical examination of François' method (cf. B., 1928, 243). Both the presence of sugar and the temperature have a considerable influence on the rate of decomposition of chloral by alkalis. If the conditions described by François be strictly followed, chloral can be accurately determined in presence of syrup only if the temperature is above 21°. At lower temperatures decomposition is very slow, but ultimately is complete (cf. Andron, B., 1929, 35).

E. H. SHARPLES.

Commercial civet. C. T. BENNETT and W. M. SEABER (Perf. Ess. Oil Rec., 1929, 20, 14—15).—The analyses of 19 commercial samples of civet are critically compared with previously published figures and standards. Four samples appear to be adulterated with petroleum jelly, and 3 others with butter fat.

E. H. SHARPLES.

Indian ephedras. B. E. READ and C. T. FENG (J. Amer. Pharm. Assoc., 1928, 17, 1189—1192).—The

amounts of ephedrine and ψ -ephedrine in the following ephedras from Northern India have been determined: *E. intermedia* (dry broken stems and unbroken stems), *E. Gerardiana*, *E. sinica*, and *E. equisetina*. The results are compared with other assays and their therapeutical significance is discussed. *E. intermedia*, containing 60—70% of ψ -ephedrine in the total alkaloid, and *E. Gerardiana*, containing 20—30%, are not likely to yield identical physiological results. In the sample of *E. intermedia* the length, weight, and number of nodes or joints has been critically examined.

E. H. SHARPLES.

Chemistry, pharmacology, and therapeutics of Agave Salmiana. G. G. COLIN (J. Amer. Pharm. Assoc., 1928, 17, 1182—1188).—A crystalline substance from the juice of *Agave Salmiana* has been examined for its alleged anti-syphilitic properties. Some therapeutic properties have been observed without the use of cathartics or sudorifics, but the results were not conclusive, and the effects on the Wassermann reaction were not consistent.

E. H. SHARPLES.

Examination of β -amino- α -phenylethyl alcohol sulphate. S. M. GORDON (J. Amer. Pharm. Assoc., 1928, 17, 1195—1199).—A crystallographic and chemical examination of β -amino- α -phenylethyl alcohol sulphate, m.p. 250—254°, are described. An aqueous solution, when treated with copper sulphate and sodium hydroxide, yields a purple colour which is discharged by acids, and is insoluble in ether. Benzaldehyde and the corresponding hydrochloride are formed when the base is extracted from alkaline solution with chloroform. On boiling the sulphate with chloroform and alcoholic potassium hydroxide solution a strong odour of carbylamine is produced. In aqueous solution it yields precipitates with phosphotungstic acid, palladium chloride, and with Millon's reagent a yellow precipitate which is unchanged on heating. The compound forms a *picrolonate*, m.p. 200—201° (corr.), and a *monobenzoyl* derivative, m.p. 149.5—150° (corr.). Other published reactions are outlined and a series of standards is suggested.

E. H. SHARPLES.

Odour and constitution among the mustard oils [thiocarbimides]. V. Blending of mustard oils. G. M. DYSON (Perf. Ess. Oil Rec., 1929, 20, 3—5; cf. A., 1928, 283, 514, 748, 1127).—4-Aldehydophenylthiocarbimide ("thiotrope base"), m.p. 32°, has an odour strongly resembling heliotropin, for which it may be substituted without alteration in certain types of floral oils. Examples of blending of both the above and mesitylthiocarbimide, which exercises a softening action in perfumes of the "new-mown-hay" type, are given. Neither compound has any value for perfuming toilet soap base, but *p*-tolylthiocarbimide, either alone or blended, is a useful soap perfume. Rideal-Walker coefficients of some aromatic thiocarbimides have been determined. E. H. SHARPLES.

Tobacco products poor in nicotine. M. POPP (Z. Pflanz. Düng., 1928, 12A, 334—344).—The nicotine content of tobacco and products is discussed, and the relative proportions of toxic material in tobacco smoke are examined. Fertiliser trials are recorded showing

their effects on the nicotine content of the crop and compared with the market value. A. G. POLLARD.

PATENTS.

Treatment of oils and fats [medicinal paraffin]. E. CANALS (B.P. 302,954, 21.9.27).—Liquid paraffin etc. (e.g., 90 pts.) is mixed with a solution of, e.g., 0.05 pt. of saccharin in 9.95 pts. of olive oil.

L. A. COLES.

Production of narcotics. E. HESSE, ASSR. to GEHE & Co. A.-G. (U.S.P. 1,695,656, 18.12.28. Appl., 17.11.27. Ger., 10.3.27).—Substances containing cocaine are treated with adonidine; the product is an anaesthetic containing both drugs.

F. G. CLARKE.

Preparation of tannic acid compositions. R. S. PATERSON (U.S.P. 1,690,175, 6.11.28. Appl., 4.2.26).—Tannic acid is ground with zinc oxide, mixed with glycerol, and after hardening with a little water the mass is dissolved in water and used for urethral injection.

R. BRIGHTMAN.

Extraction of essential principles or gums from tobaccos of all kinds. B. SEFERIADIS (B.P. 302,863, 25.5.28. Fr., 19.3.28).—Tobacco or tobacco residues are treated with acetone at normal or higher temperature and pressure. The gums obtained on evaporation of the solvent are incorporated in tobacco to impart an aroma.

B. FULLMAN.

Destruction of germs in and sterilisation of cigarette tobacco. "UNIVERSELLE" CIGARETTEN-MASCHINEN FABR. J. C. MULLER & Co. (B.P. 294,537, 12.7.28. Ger., 25.7.27).—Air containing ozone is used for the pneumatic conveyance of the cut tobacco to the tobacco-loosening and dust-removing plant. The introduction of injurious substances (such as nitrous oxide, obtained in the production of the ozone) into the air-feed pipe is avoided.

B. FULLMAN.

Cosmetic (B.P. 272,541).—See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photochemistry of silver halides. IV. New method of determining excess silver in unexposed photographic films, and studies on the processes involved in exposure. H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1929, 26, 259—274).—The new method consists in centrifuging a neutral solution. From analyses by this method and by that using dichromate-sulphuric acid, the "silver value" has been split into three components. The first and greatest part is dispersed in the gelatin and can be obtained by centrifuging or by dichromate-sulphuric acid solution without altering the photographic properties of the film. The second part is absorbed in the grain and can be dissolved out by means of dichromate-sulphuric acid. The third part is similar to the second, but is not soluble. This third component is believed to cause sensitivity. The emulsions used, in this case poor in gelatin, had a smaller "silver value" than those rich in gelatin which were used previously (B., 1928, 625). A better method of deter-

mining "free silver" is required before the correlation of "free silver" with sensitivity is proved.

W. E. DOWNEY.

Influence of the nature of the fixing agent on the development after fixation of inverted or solarised photographic plates. H. BELLIOU (Compt. rend., 1929, 188, 167—169; cf. B., 1929, 151).—Development after fixation of inverted or solarised plates resembles that produced by a chemical developer when the fixing agent has strong reducing properties, the evolution of the development being inverted if the latter is acidified. The image produced is attributed to molecules directly reduced by the action of light, and, in cases where the fixing agent has strong powers of reduction, to activated molecules and molecules in their immediate neighbourhood.

J. GRANT.

XXII.—EXPLOSIVES; MATCHES.

Decomposition of explosives. J. P. KOETTNITZ (Z. Elektrochem., 1928, 34, 768—783).—The author's previous view, that an explosive process may be regarded as the evaporation or sublimation of the liquid or solid explosive, with simultaneous, or almost simultaneous, dissociation and the evolution of large quantities of heat, is further developed. On the above assumption, an approximate value for the detonation temperature, T_r , can be calculated from $Q_v/\log_e 10RT_r = n_r(1.75 \log T_r + C_0)$, where Q_v is the heat of explosion at constant volume, n_r the number of gas molecules reacting as a result of the detonation, and C_0 is a constant. From this, $n_r = Q_v/(4.58 T_r)(1.75 \log T_r + C_0)$. If t_v is the detonation temperature in °C., and n_A the number of atoms at the end of the reaction, then, for mercury fulminate, by calculation, $n_r = 7 = n_A$ at $t_v = 150^\circ$; corresponding values for cyanuric triazide are 15, 193°; for nitrogen tetrasulphide S, 206°; and for nitroglycerin 80, 195°. Assuming that in the explosion of the following substances $n_r = n_A$, t_v (calc.) is 190° for pentaerythritol tetranitrate, 180° for mannitol hexanitrate, 170° for nitrohexamethylenetetramine (hexogen), 140° for methyl nitrate, and 190° for ethylene glycol dinitrate. In cases of incomplete combustion (i.e., when no water is formed among the reaction gases from a hydrocarbonaceous explosive), it is calculated that for picric acid $n_r = 18 = n_e$ (number of mols. at the end of the reaction) at $t_v = 424^\circ$; corresponding values for trinitrotoluene are: 18, 377°; for diazobenzene nitrate 10, 83°; for guncotton 38, 183°. Considering metallic explosives: for silver oxalate $n_r = 2 = n_g$ (number of gaseous mols. at the end of the reaction) at $t_v = 140^\circ$; the values of n_r and t_v are: for lead trinitroresorcinate 19, 260°; for lead azide 3.5, 337°; for silver acetylide 4, 200°. It is also calculated that for nitrogen trichloride $Q_v = 114.3$, and for ammonium nitrate $n_r = 7 = n_e$ at $t_v = 164^\circ$. The significance of these results is discussed at some length, with special reference to the classification and action of anti-knock materials. A number of experiments are recorded on the explosions of alkali metals with halogenated hydrocarbons. Numerous equations are developed, and a new theory of detonators is outlined, according to which the

detonator is regarded as a system consisting of a very large number of molecular elements or condensers.

L. L. BIRCUMSHAW.

Vaseline for smokeless powders. POGGI.—See II.

PATENTS.

Priming mixture for small-arms ammunition. H. T. PECK, Assr. to PETERS CARTRIDGE Co. (U.S.P. 1,694,890, 11.12.28. Appl., 27.6.27).—The mixture consists of mercury fulminate, barium nitrate, lead chromate, and lead thiocyanate. II. ROYAL-DAWSON.

Priming compositions (B.P. 302,961). See XIII.

XXIII.—SANITATION; WATER PURIFICATION.

Dissolved oxygen absorption test. III. E. A. COOPER and S. D. NICHOLAS (J.S.C.I., 1928, 47, 320—322 T).—Solutions of sodium permolybdate accelerate the rate of dissolved oxygen absorption by sewage effluents. The oxidation of sugars, glycine, and sometimes ammonium salts in dilute aqueous solution is also accelerated by the permolybdate activator. The increased oxygen uptake is due partly to stimulated bacterial activity, and also to an induced direct chemical oxidation. An improved method for preparing the active solution is described.

Hydrogen sulphide removal and water softening at Beverly Hills, Cal. R. L. DERBY (J. Amer. Water Works' Assoc., 1928, 20, 813—818).—In order to augment the water supply of Beverly Hills, California, a supply of well water was tapped, but the presence of hydrogen sulphide averaging 6—10 p.p.m. and an average hardness of 275 p.p.m. caused many complaints. Difficulty also arose owing to a luxuriant growth of beggiatoa in the settling basins and service pipes. Chlorination was fairly successful in removing the trouble, but the coating of beggiatoa in the mains was so thick that it was impossible for this to be effective at a reasonable distance from the station without having an objectionable quantity present for those nearer to the point of supply. A combination of aeration, alum, and lime precipitation, and chlorination followed by filtration, produced a water having a hardness of 160—170 p.p.m. and free from hydrogen sulphide.

C. JEPSON.

Composition and use of ferric hydroxide as a coagulant [in river water]. E. S. HOPKINS (Ind. Eng. Chem., 1929, 21, 58—60).—Laboratory tests carried out on litre samples of river water indicate that the normal working p_H value 9.4 is the most efficient value for quantitative precipitation of ferric hydroxide at all concentrations, less than 1 p.p.m. remaining in solution. The maximum turbidity removal takes place at p_H 8.4 with the average at 9.4. When 17—170 p.p.m. of ferrous sulphate ($7H_2O$) are used the floc consists entirely of ferric hydroxide, but with higher concentrations, 428 and 1710 p.p.m., definite evidence of adsorption of sulphates is obtained. A definite basic sulphate is not formed under these conditions as occurs with alum.

C. JEPSON.

Chlorine absorption as a substitute for oxygen consumed [in water analysis]. H. HALE, M. SULLI-

VANT, and C. B. DEWITT (J. Amer. Water Works' Assoc., 1928, 20, 847—853).—The oxygen consumed test gives an indication of the carbohydrate but not of the nitrogenous matter present, and is somewhat affected when the chloride content of the sample is high. Chlorine absorption gives a measure of the nitrogenous matter present, and in low concentrations is unaffected by carbohydrates. It is recommended that the test be carried out on 100 c.c. of the sample, heated just to boiling, with a chlorine dose of 2 mg. in the form of chlorine water. After 10 min. the excess chlorine may be determined in the usual way with *o*-tolidine.

C. JEPSON.

Corrosion of iron and steel by brackish waters. FORREST and others.—See X.

PATENTS.

Sewage treatment with the aid of a screen acting like a filter. K. IMHOFF (U.S.P. 1,696,846, 25.12.28. Appl., 18.6.27. Ger., 31.7.26).—Sewage is not allowed to pass through the screen until a mat of sludge has been formed on it. This acts as a fine filter when the sewage is passed through, and the deposited solids are finally removed.

C. JEPSON.

Production of a substance having disinfectant, cosmetic, tanning, or like properties, and suitable for the treatment of textile materials. K. KELECI (B.P. 272,541, 10.6.27. Hung., 12.6.26).—The product comprises a mixture of soap, an aldehyde other than acetaldehyde, and chlorine and/or bromine or substances yielding them, e.g., hypochlorites etc., with or without the addition of a heavy-metal compound, e.g., chromium sulphate, and carbon disulphide. [Stat. ref.]

L. A. COLES.

Distillation of water for feed water make-up. N. V. NECKAR WATERREINIGER MAATSCHAPPIJ, and H. NOORDENDORP (B.P. 301,666, 10.1.28).—Sludge water blown down from the boiler is passed direct into an expansion and pressure-reducing chamber, and the steam derived is conveyed to the make-up evaporator and there used for the distillation of make-up feed water which has been preheated and de-aerated by the sludge water remaining after the pressure reduction.

W. G. CAREY.

Purifying the water used for steam-boiler purposes. A. H. WHITE (U.S.P. 1,693,066, 27.11.28. Appl., 7.12.25).—Sufficient phosphoric acid to form insoluble phosphates with the calcium, magnesium, and iron carbonates present is added to the water together with sodium phosphate to precipitate other salts of these metals, the amounts added being such as to neutralise the water after the carbon dioxide has been removed.

W. G. CAREY.

Removal of easily-soluble salts from boiler feed water. Purification system for boilers. Clearing and purifying water for steam boilers of all kinds. J. OSTERTAG (U.S.P. 1,700,714—6, 29.1.29. Appl., [A, B] 7.7.22, [C] 10.7.22. Poland, [A—C] 8.2.22).—See B.P. 200,263 and 204,352; B., 1923, 905 A, 1195 A.

Water-softening silicate (U.S.P. 1,693,873).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 15, 1929.

L—GENERAL; PLANT; MACHINERY.

Approximate determination of the absolute size of pores. M. A. RABINOVICH and N. S. FORTUNATOV (J. Chem. Ind. Moscow, 1928, 5, 689—694).—Vapour pressure determinations lead to determinations of the absolute dimensions of pores. Minkovski's equation is modified: $r = k (\log P_0 - \log P_1)$, where k is a constant, P_0 the vapour pressure over the flat surface, and P_1 the vapour pressure over the meniscus in the capillary pore. For charcoal the order of decreasing relative volume of the pores is: birch, pine, activated; porosity determinations give the order: activated, birch, pine. Experiments were also performed with graphites, kaolin, and porcelain. Ignition at a high temperature leads to the same changes in the structure of kaolin as are observed in the ageing of gels.

CHEMICAL ABSTRACTS.

Apparatus for indicating the saturation of an absorption system. A. MACKERT (Chem. Fabr., 1929, 39—40).—The apparatus automatically gives an alarm when, e.g., an activated carbon or silica gel scrubber is saturated and is allowing benzol vapour to pass. A number of beads of activated carbon are hung on a light metal ring from a leaf spring within a vessel through which a portion of the exit gas passes. One end of the leaf spring is clamped and the other end bent at right angles above a mercury contact. Any adsorption by the beads leads to an increase in weight, which depresses the spring and closes an electric circuit. The apparatus is readily adjusted as regards sensitiveness, and will record a few g./m.³ if required. C. IRWIN.

See also A., Feb., 161, **Gas analysis apparatus** (BAHR). **Hydrogen-ion colorimeter** (GRADWOHL). 167, **Determination of Engler viscosities** (ERK). **Turbidimeter** (EWALD).

Slag wool. GUTTMANN.—See X.

PATENTS.

Regenerative furnace. W. MILLWARD (U.S.P. 1,695,199, 11.12.28. Appl., 5.7.23).—The furnace is provided with a gas uptake and an unobstructed air uptake. The former has a gooseneck terminal which stops short of the air uptake and can be supplied with air under pressure, so forming a primary mixing chamber. A secondary mixing chamber centred relatively to the first is provided, the whole arrangement forming a means for effecting sharp combustion in furnaces of the regenerative type. A. B. MANNING.

Desiccation of substances. F. H. DOUTHITT (B.P. 301,952, 8.9.27).—In the apparatus described in B.P. 260,453 (cf. B., 1927, 26) the drying air is admitted through a number of slit-like tangential nozzles which

are individually or collectively adjustable by hinged vanes. Another form of scraper is described.

B. M. VENABLES.

Apparatus for producing an intimate mixture of several media by means of centrifugal force. P. JAMES (B.P. 283,975, 21.1.28).—An intimate mixture of a gas and liquid or powder is created by a centrifugal rotor which draws in the gas through an upper axial opening and the other medium through a lower axial opening, the two being kept apart for about two thirds the radial distance they have to flow and being mixed in the outer third. The mixing is aided by corrugations formed on the interior of the outer third of the rotor, and the outermost rim deflects the mixture downwards, so that coarser particles are caught in a basin below and re-fed to the lower axial inlet; only the finer gas-borne particles leave the apparatus.

B. M. VENABLES.

Separation of substances of different sp. gr. W. R. CHAPMAN (B.P. 301,336, 24.6.27).—The coal or other material is passed down an inclined covered trough, a current of gas in the same direction being provided to promote stratification. The heavier materials are drawn off through unobstructed apertures the full width of the bottom of the trough, up which there may be auxiliary currents of gas to prevent the fall of lighter materials. The trough may be reciprocated in a plane inclined to it, and riffles may be provided.

B. M. VENABLES.

Apparatus for classifying or separating granular materials. A. FRANCE (B.P. 301,812, 1.9.27).—In a wet classifier of the type where a stream of pulp flows down an inclined trough with a transverse pocket for intercepting heavy material, the depth of the pulp stream, acting on a flap, is caused to vary the conditions of classification in a number of ways, through an elaborate mechanical gear.

B. M. VENABLES.

Separating constituents of loose mixtures particularly of liquids, vapours, and gases. F. BARTLING and F. LAWACZECK (B.P. 282,840, 2.1.28. Ger., 31.12.26).—When two surfaces are moving at different speeds and a current of gas is passed between them the coarser particles will move to the layers of lowest speed. The apparatus, based on the above principle, comprises a hollow cylindrical or conical member rotating between stationary walls, the heavier particles collecting on the stationary walls and the cleaned gas passing off through the interior of the rotating members. B. M. VENABLES.

Steam purifiers and like apparatus for separating impurities from fluids. M. R. PATTISON (B.P. 302,018, 1.11.27).—A number of open-bottom tubes are placed side by side so that the fluid passes downwards

between the tubes and then through them. The tubes are of aerofoil shape in section, so that the spaces between are of Venturi section. A sludge tank is provided to collect solid impurities. B. M. VENABLES.

Centrifugal separator. J. I. FRITZ (U.S.P. 1,694,972, 11.12.28. Appl., 30.6.26).—The apparatus is provided with a central supply feed below a perforated diaphragm almost closing the lower portion of the bowl, and with a superposed axial deflecting member. L. A. COLES.

Apparatus for the treatment of solid-bearing solutions. W. L. REMICK (U.S.P. 1,696,014, 18.12.28. Appl., 17.9.25).—A tank discharging into a filter is provided with a receptacle for the separated solids and with means to convey the solids from the receptacle to the tank or to return the filtered liquid to the tank. A. R. POWELL.

Plant for washing, drying, and chemical treatment of granular and crystalline materials. T. O. and N. WILTON, and CHEM. ENGINEERING & WILTON'S PATENT FURNACE CO., LTD. (B.P. 297,630, 28.1.28).—The material is spread on a conveyor of woven wire and there treated with sprays of reagents or washing fluids; thus, between the runs of the conveyor the material catches the effluent liquids, and on the return run of the conveyor is washed, after discharge of the solid material, by dipping into a trough. B. M. VENABLES.

Extraction from gases or liquids of substances suspended or contained therein. A. H. PEHRSON (B.P. 278,722, 6.10.27. Swed., 7.10.26).—The fluid to be cleaned is passed through a mass of granular material which is tumbled about in a rotary, perforated container. The granular material may be damped with water, tar, lye, etc. according to the constituent it is desired to remove. Superfluous liquid formed during treatment may be evaporated by passing heated gases. [Stat. ref.] B. M. VENABLES.

Filter. A. BOULADE, ASSR. to SOC. DU CARBURATEUR ZÉNITH (U.S.P. 1,688,326, 23.10.28. Appl., 16.9.25).—A filter for separating solids from oils comprises a series of flat filtering discs corrugated on the top and bottom so as to form a number of thin annular apertures through which the liquid passes to a central annular space surrounding a central supporting stem. A. R. POWELL.

Air filter. H. E. BIRKHOLZ, ASSR. to NAT. AIR FILTER CO. (U.S.P. 1,690,813, 6.11.28. Appl., 24.9.26).—An apparatus for removing dust from air comprises a rotating drum containing a metal wire screen coated with oil, which acts as a collector for the dust, and means for directing a small stream of oil in a helical path over the screen, so that only a relatively small area of the screen is in a newly washed condition at any time. A. R. POWELL.

Recovering [crystallisable] values by evaporation. H. W. CARR (U.S.P. 1,694,841, 11.12.28. Appl., 30.4.27).—Solutions containing crystallisable constituents are dropped on to residues obtained by the previous evaporation of similar solutions, maintained at a temperature sufficiently high to cause evaporation of the liquid. L. A. COLES.

Pasteurisation of liquids. A. JENSEN (U.S.P. 1,693,034, 27.11.28. Appl., 2.2.26).—The liquid, in a closed chamber, is caused to rise up on the side walls thereof by centrifugal action and brought to the required temperature by means of a heating fluid under pressure in contact with the walls. Deleterious gases are withdrawn centrally from the chamber by suction. W. J. BOYD.

Stirring apparatus for vacuum distillation vessels. G. SCHAUS (G.P. 450,978, 7.8.25).—The shaft of the stirring device is provided with an inverted conical protuberance which works in a suitably shaped oil-lubricated bearing. Below this is a small chamber which serves to collect the oil that percolates through; this chamber may be provided with a water-cooling arrangement to prevent the heat of the distillation vessel affecting the viscosity of the lubricating oil in the vacuum-tight bearing. A. R. POWELL.

Condensate removing systems. [Steam trap.] R. BELL-IRVING and P. SANDWELL (B.P. 301,213, 9.12.27).—The discharge for condensate from apparatus such as drum dryers comprises a main pipe extending nearly to the bottom and a shorter auxiliary pipe. The main pipe is normally closed by a valve which is opened by the steam pressure on a diaphragm. Whether or not the diaphragm receives pressure is determined by a float-operated valve, the float being raised and valve closed when the auxiliary pipe is discharging mainly steam and air, and sunk when the latter pipe is discharging water. B. M. VENABLES.

Diffusion reaction. H. WALTER, ASSR. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,685,759, 25.9.28. Appl., 1.5.26. Ger., 22.5.24).—Gases which react slowly in simple mixtures are passed in countercurrent on opposite sides of a porous partition under substantially the same pressure. Reaction takes place in the capillaries of the partition, and the reaction products diffuse out on both sides and are removed by the countercurrents and condensed. The partition may be impregnated with a specific catalyst for the reaction, e.g., for the production of methyl alcohol from carbon dioxide and hydrogen at 380° and 50 atm., with a solution of 80 g. of zinc nitrate, 10 g. of copper nitrate, and 20 g. of borax in 500 c.c. of water. R. BRIGHTMAN.

Treatment of liquids. C. BENEDICKS (U.S.P. 1,691,458, 13.11.28. Appl., 29.1.25. Swed., 2.7.24).—Molten glass or other liquid is freed from dissolved or occluded gas by agitating it in the presence of a device kept at a temperature higher than that of the liquid. F. G. CLARKE.

Digesters. J. W. PENMAN (B.P. 302,077, 31.1.28).—A false bottom of a digester is connected to the end plate of the digester in such a manner that it is slightly flexible between the rivets of the joint, e.g., by making fine saw cuts radially inwards. B. M. VENABLES.

Autoclaves. T. W. BARBER (B.P. 301,994, 30.9.27).—A continuously operating autoclave for such purposes as separation of oil from a magma by heating in an aqueous liquor is provided with outlet valves in the upper and lower parts which are controlled by floats; circulation of the charge is effected by convection.

currents and by the injector effect of the incoming liquid.

B. M. VENABLES.

Photometer. R. H. DAVIS (B.P. 301,665, 10.1.28).—A shadow photometer is provided with a holder for a standard candle which can be placed in any one of several fixed positions to suit different ranges of candle-power to be measured, and can be raised or lowered so as to be level with the illuminant to be measured. The latter, *e.g.*, a miner's lamp, is supported on a base slidable against a square-root scale and preferably formed in stepped circles to suit various diameters of lamp. The base may also be provided with a scale of degrees so as to determine the candle-power at various angles of lamps with reflectors.

B. M. VENABLES.

[Flat] glass bulbs for hydrometers and like instruments. F. F. W. FLAIG (B.P. 302,034, 16.11.27).—A flat glass bulb is provided with a stay between the flat surfaces comprising a tube or bridge, the space within the bulb becoming annular.

B. M. VENABLES.

Hydrometer device. S. P. WILBUR, Assr. to U.S.L. BATTERY CORP. (U.S.P. 1,696,618, 25.12.28. Appl., 26.5.20. Renewed, 16.1.28).—A float is adapted to co-operate with the vertically projecting stem of a sp. gr. device having a distinctive zone and presents a contrast to that zone.

J. S. G. THOMAS.

[Laboratory] crucible holder and stand. G. S. HARTLEY (B.P. 301,685, 13.3.28).—A laboratory crucible which has been ignited while on the usual pipeclay or silica triangle is removed therefrom and supported while cooling by a triangular stool of refractory material which, together with a wire handle, is small enough to pass through the pipeclay triangle. After cooling, the crucible is transferred (for weighing) to a light stand made of bent sheet metal which is large enough to slip over the refractory stool and small enough to retain the crucible. Direct contact with the hand or tongs is avoided in all the above operations.

B. M. VENABLES.

Apparatus for pulverising. W. R. WOOD, Assr. to INTERNAT. COMBUSTION ENGINEERING CORP. (U.S.P. 1,697,704, 1.1.29. Appl., 24.2.27. U.K., 1.4.26).—See B.P. 269,319; B., 1927, 575.

[Combustion apparatus for] furnaces. C. H. GLOVER (B.P. 302,757, 17.10.27).

[Door suspension and control system for] annealing and other furnaces, muffles, etc. BIRMINGHAM ELECTRIC FURNACES, LTD., and A. G. LOBLEY (B.P. 304,458, 11.1.28).

Refrigerating machines. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of C. STEENSTRUP (B.P. 284,730, 4.2.28. U.S., 5.2.27).

Refrigerating apparatus. N. V. "KODOWA" REFRIGERATOR CO. (B.P. 294,999, 20.10.27. Holl., 4.8.27).

Economical carrying-out of refrigeration on a large scale. BROWN, BOVERI & CO. A.-G. (B.P. 285,003, 15.11.27. Ger., 8.2.27).

Rectification by cooling of gases, gas mixtures, or vapours in absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS RE-

FRIGERATING SYSTEM AKTIEBOLAG (B.P. 274,840, 30.6.27. Ger., 26.7.26).

Combustion of fuels [for steam generators etc.] (B.P. 302,255). **Distillation of liquids** (B.P. 303,038).—See II. **Plant for cooling liquids** (B.P. 302,004).—See V. **Separation of gases etc. from discharge gases of furnaces** (B.P. 302,386). **Hardness testing** (B.P. 301,762).—See X. **Pyrometer** (U.S.P. 1,697,148).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Flotation of coal. K. YAMAGUCHI (J. Fuel Soc. Japan, 1928, 7, 126—128).—The separation of pyrites from coal by froth-flotation has been studied. A mixture of pyrites with a coal containing 3% of ash was successfully separated by taking advantage of the different rates of flotation of the two constituents. Less success was attained with a mixture of pyrites and a very high-ash coal.

A. B. MANNING.

Determination of the ignition point of liquid fuels. F. HOFFMANN (Arch. Wärmewirts., 1928, 9, 213—219; Chem. Zentr., 1928, ii, 726—727).—A comparative study of methods.

A. A. ELDRIDGE.

Firedamp explosions within closed vessels: "pressure piling." C. S. W. GRICE and R. V. WHEELER (Safety in Mines Res. Board Paper No. 49, 1929, 26 pp.).—When two vessels of different size, and communicating with one another through a short, narrow passage, are filled with an explosive mixture, and the latter is ignited in the larger vessel, a sudden and rapid production of pressure occurs in the smaller vessel. Photographs of the flame movement within glass vessels have shown that this is due to the turbulence in the smaller vessel produced by the explosion in the larger, and to the large flame which is projected through the communicating passage. If the mixture is ignited in the smaller vessel, the turbulence produced in the larger is inconsiderable, and the flame projected thereinto is smaller. If the ratio of the volumes of the vessels is greater than 10:1 and the communicating passage is narrow, then an explosion of methane and air initiated in the former can give rise to pressures in the latter considerably higher than those normally developed by methane-air mixtures initially at atmospheric pressure. This phenomenon, known as "pressure piling," is due partly to the causes already mentioned, but principally to the compression of the mixture before ignition.

A. B. MANNING.

Thermal decomposition of methane. T. KUSAMA and Y. UNO (Bull. Inst. Phys. Chem. Res., Tokyo, 1929, 8, 1—11).—Catalytic reduction of natural gas was tried, using as catalyst pure nickel, a mixture of nickel and kieselguhr, which was equally suitable after being deactivated by the reduction of naphthalene, and finely-divided iron. Carbon obtained as a decomposition product of methane was of graphitic form and contained nickel, which was recovered by digesting the deposit with hydrochloric acid with subsequent reduction of nickel chloride to active nickel. Dissociated hydrogen was always contaminated with considerable quantities of methane, and the mixture was used for the catalytic reduction of naphthalene.

C. A. KING.

Products and mechanism of the thermal decomposition of benzene. Y. KOSAKA (J. Fuel Soc. Japan, 1928, 7, 121—123).—The decomposition products obtained when benzene is dropped down through a tube packed with silica or coke granules and heated at 700°, 800°, or 900° are diphenyl, diphenylbenzenes, ethylene, methane, carbon, and hydrogen. The yields at each temperature have been determined and tabulated. No condensed-ring compounds are formed.

A. B. MANNING.

Products and mechanism of the thermal decomposition of phenol. Y. KOSAKA (J. Fuel Soc. Japan, 1928, 7, 123—125; cf. preceding abstract).—The products obtained when phenol is decomposed by being dropped down through a hot tube packed with coke or silica granules include benzene, naphthalene, diphenyl, anthracene, phenanthrene, ethylene, methane, carbon, and hydrogen. The yields of each product obtained at 700°, 800°, and 850°, respectively, are tabulated.

A. B. MANNING.

Rational production of mixed gas, including the use of composite producer gas ("Doppelgas"). E. LANGTHALER (Gas- u. Wasserfach, 1929, 72, 31—33).—Mixed gas, having a gross calorific value of 4200 kg.-cal./m.³, may be produced by mixing ordinary coal gas (gross cal. value 5000 kg.-cal./cub. m.) with suitable proportions of flue gases, ordinary producer gas, water-gas, or composite producer gas ("Doppelgas") obtained by carbonising coal and completely gasifying the coke in the same retort, the gross calorific values of these latter being, respectively, 0, 1150, 2750, and 3300—3600 (carburetted) kg.-cal./m.³. Considering these five gaseous admixtures in the above order, the percentage of each in a mixed gas containing coal gas as the only other constituent and having a gross calorific value of 4200 kg.-cal./cub. m. is 16, 20, 35, 47, and 58%, respectively. The coal required per 1000 cub. m. of mixed gas is 2580, 2340, 1980, 1980, and 1760 kg., respectively, and the available coke remaining is 1390, 1220, 785, 874, and 715 kg., respectively. The mixed gas obtained by diluting coal gas with flue gases or producer gas contains an excessive proportion of inerts (26—28%), but the possibilities of composite producer gas are noteworthy.

W. T. K. BRAUNHOLTZ.

Removal of sulphur from illuminating gas by activated carbon. E. W. TODD (Proc. Nova Scotian Inst. Sci., 1928, 17, 120—121).—The activated carbon "Rotite" (125 g.) reduced the sulphur content of 100 cub. ft. of gas passed through it to one eighth of its original value. The carbon, saturated with sulphur from 300—400 cub. ft. of gas, was considerably revived by heating with stirring in an open vessel. "Norit" carbon gave similar results.

B. W. ANDERSON.

Colorimetric determination of carbon disulphide in gas. G. G. DÉSY (Proc. Amer. Gas Co., 1927, 1440—1441).—The gas (100—1000 c.c.) is passed through 10% potassium hydroxide solution, and then through concentrated sulphuric acid, and its volume is measured; it is then passed from a capillary orifice through alcoholic potassium hydroxide, which is diluted in a Nessler tube with distilled water to 48 c.c., and made slightly acid (to phenolphthalein) with acetic

acid. Four drops of 0.05N-copper acetate solution are added, and the colour is compared with controls similarly prepared from ethyl xanthate. The method is accurate to about 10%. CHEMICAL ABSTRACTS.

Action of silent electrical discharge on mineral and vegetable oils. T. RABEK (Przemysl Chem., 1928, 12, 240—249; Chem. Zentr., 1928, ii, 512).—An improved polymerisation process is described.

A. A. ELDRIDGE.

See also A., Feb., 147, **Effect of anti-knock materials on flame of hydrocarbon-air mixtures** (NAGAI). 161, **Gas analysis apparatus** (BAHR). 168, **Base exchange and genesis of petroleum** (TAYLOR). 169, **Fushun coal** (IWASAKI). 184, **Naphthenic acids in Japanese petroleum** (KUWATA).

Indicator for absorption systems. MACKERT.—See I. **Conductivity of kerosene and gasoline.** MOREHOUSE.—See XI.

PATENTS.

Retort structures. E. G. BLACKWELL (B.P. 297,445, 18.1.28. U.S., 22.9.27).—A retort chamber of elongated cross-section is disposed between, and with its ends against, abutments; the side walls are constructed of bricks the adjacent abutment faces of which converge in the direction of the forces acting on the wall. By constructing the horizontal courses so that in some the brick abutment faces converge inwards and in others outwards, a structure capable of withstanding both external and internal pressures is obtained.

A. B. MANNING.

Vertical coking retort ovens. KOPPERS Co., Assces. of J. VAN ACKEREN (B.P. 283,191, 5.10.27. U.S., 8.1.27).—In order to facilitate the discharge of the coke by gravity the coking chambers of a vertical coke-oven setting are of progressively increasing width from top to bottom; to produce a corresponding gradient in the rate of heat supply the vertical heating flues are subdivided into superimposed sections to each of which the supply of fuel gas can be suitably adjusted. The flue sections increase progressively in size from the bottom to the top of the coking chamber, thereby attaining substantially uniform velocities of the products of combustion throughout the height of the heating walls. The upper ends of the vertical flues communicate with horizontal flues which are connected together in pairs by cross-over ducts. At the lower end of the vertical flues of each heating wall are two horizontal flues which can be connected directly to regenerators disposed outside and below the main oven structure. The bottom openings of the chambers are provided with doors the supporting frames of which form water-seals for the chambers.

A. B. MANNING.

Charging of coke ovens. L. MELLERSH-JACKSON. From KOPPERS Co. (B.P. 302,188—9, 11.6.27).—(A) A coke oven has an offtake near each end and charging openings in the roof between the offtakes. Means are provided for inducing a forced draft within the offtakes in order to remove the gas and smoke evolved during charging (cf. B.P. 278,012; B., 1929, 160). The oven may be charged first through openings near the ends and subsequently through an intermediate opening, the gas

and smoke being removed through the end offtakes. In another modification offtake pipes are provided which can be simultaneously connected to the mains and to additional offtake openings between the charging openings. Coal hoppers corresponding in number with the charging openings, the offtake pipes, and the mechanism for operating the connecting devices between these and the offtake openings and the mains respectively, and for operating similar connecting devices between the hoppers and the charging openings, are all mounted on a larry car, which traverses the top of a battery of ovens, charging each in succession. (B) The hoppers from which the oven is charged may be made of different holding capacity, one only being of such size that its contents when charged into the chamber will, until levelled, divide the chamber into two separate compartments. All the smoke and gases evolved during charging can then be withdrawn through one or other of two offtakes at either side of the oven and no further offtake openings are required.

A. B. MANNING.

Carbonisation of coal, wood, peat, and other ligneous material. GAS LIGHT & COKE CO., H. HOLLINGS, and J. NEATH (B.P. 302,437, 6.10.27).—In order to avoid losses of gas by leakage resulting from fluctuations of pressure within retorts in which coal, wood, etc. is being carbonised, the pressure is controlled in such a way as to suit the requirements of the material undergoing treatment and the other conditions of carbonisation. The pressure may be regulated (a) by varying the position of an adjustable dip-pipe in the hydraulic main, (b) by varying the level in the main by the use of an adjustable overflow pipe, or (c) by means of a gas governor. The adjustable dip-pipe, overflow pipe, or gas governor is controlled mechanically by a cam of suitable design rotated at the desired speed. The design of the apparatus for effecting the control is described in detail.

A. B. MANNING.

Carbonisation of low-grade fuel substances and apparatus therefor. M. MIGEON (B.P. 288,335, 5.4.28. Ger., 8.4.27).—The material after being dried by the waste heat of the plant passes down a vertical retort in which it undergoes a primary distillation. This retort is constructed of superimposed elements, each of which consists of a number of radial chambers stepped laterally in relation to each other; the material thus follows a helicoidal path, the heating surface being thereby increased and the production of dust reduced. The products of distillation from each element are condensed rapidly and are then redistilled by contact with a bath of molten metal. The lower end of the vertical retort communicates by means of inclined retorts with a number of other vertical retorts heated by burners and capable of acting as producers. The hot gases from the burners pass up and heat in succession the inclined retorts, the main vertical retort, and finally the dryer. During the primary distillation the material may be internally heated by the passage of hot distillation gases which have passed through a reheater in the lower part of the kiln and thence up a central duct provided with inlet openings into the vertical retort.

A. B. MANNING.

Combustion of fuels and the application and use of the heat produced thereby. A. MUSKER (B.P. 302,255, 11.8.27, 19.11.27, and 9.6.28).—Pulverised or gaseous fuel admixed with air is introduced into the upper end of a separate vertical combustion chamber, the flame of the burning fuel being then directed through horizontally-projecting ducts at a lower level to the furnace or flues of the apparatus (*e.g.*, a Lancashire boiler or other type of steam generator) to be heated. The chamber is lined with brickwork and is surrounded by an annular jacket in which the air for combustion is preheated. Some of this air is injected into the chamber at different levels through nozzles arranged to impart a whirling motion to the fuel-air mixture; some also is passed up through the ash-pit at the bottom of the chamber. If desired, other air nozzles may be provided at the end of the horizontal ducts. Below the open end of the burner delivering the fuel-air mixture into the chamber is an adjustable conical distributing device, preferably provided with spiral vanes.

A. B. MANNING.

Recovery of soluble products from coal and the like. J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 301,946, 1.9.27).—Coal is heated under pressure in a closed vessel and is then extracted with a solvent also under pressure and at elevated temperatures. A much greater yield of soluble products is obtained than by direct extraction under pressure. The products may be subjected to further treatment, such as cracking, hydrogenation, or separation by means of suitable solvents into resins, waxes, etc.

A. B. MANNING.

Apparatus for the distillation of fuels at low temperature. A. ABRASSART (B.P. 302,875, 5.7.28. Belg., 12.1.28).—The apparatus comprises a setting of three retorts of which the upper two serve for the dehydration and the partial distillation of the fuel. These retorts consist conveniently of horizontal tubes fitted with worm-conveyors. The partially distilled product is briquetted with a suitable binding agent, and the briquettes are then carbonised at 450–580° in the third retort, through which they are carried on a flexible movable conveyor. The conveyor is constructed of a metallic lattice and is so arranged that the idle strand and the members actuating the conveyor are located outside the retort. The conveyor enters and issues from the retort through a seal.

A. B. MANNING.

Production and activation of carbon. J. S. DANIELS, Assee. of E. H. MEEZE (B.P. 277,987, 22.9.27. U.S., 22.9.26).—Carbonaceous material is subjected to destructive distillation at 500–800°, preferably under reduced pressure and in the presence of steam or inert gases, and after being cooled and crushed under conditions encouraging the occlusion of atmospheric oxygen is further carbonised at 850–1000°. Compounds of zinc, magnesium, ammonium, etc. (up to 0.5% of the raw material) may be added to the material before it is subjected to the second heat-treatment.

A. B. MANNING.

Manufacture of activated carbon. R. DEFRIJS and R. WÄLDER (B.P. 303,669, 7.2.28).—Carbonaceous material, mixed with a binding agent and with 0.5–20% of suitable fibrous material, is briquetted, carbonised,

and activated. The fibrous material, *e.g.*, animal hair, is of such a nature as to produce a network of macrocapillary pores throughout the briquette, so permitting easy access of the activating gases to all portions of the material.

A. B. MANNING.

Production of active carbon. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 302,774, 1.11.27. Addn. to B.P. 238,889; B., 1925, 977).—Fine-pore, non-carbonised, vegetable substances of a preponderantly sclerenchymatic structure, *e.g.*, plum stones, coconut shell, etc., are treated with a concentrated solution of zinc chloride containing a weight of the salt less than that of the dry material. The mixture is dried and carbonised, and the product extracted with hydrochloric acid and water.

A. B. MANNING.

Production of lamp-black. J. L. MAJOR (B.P. 303,632, 6.12.27).—Lamp-black is produced by the incomplete combustion of a carbonaceous liquid which is fed on to trays mounted one above the other within a combustion chamber in such a way that the liquid cascades from tray to tray. Each tray projects further forward than the one immediately above, and has its front edge and sides notched to form a weir. Air is admitted through orifices over the trays and passes transversely across the cascading liquid.

A. B. MANNING.

Treatment of hydrocarbons to obtain ink or soot. AULT & WIBORG CO., Assees. of A. L. MILLER (B.P. 286,207, 3.8.27. U.S., 28.2.27).—A hydrocarbon oil is decomposed by incomplete combustion, 5–9 pts. of air (by wt.) being present to 1 pt. of oil, into free carbon and gases at 1093–1426°. These are removed rapidly from the zone of combustion at a minimum constant velocity of 500 ft./min., cooled to 121–593° while at the same speed, and the carbon particles separated from the products of combustion by causing the gases to impinge against an interposed film or sheet of an oily ink vehicle extending at an angle to their direction of flow. The oil vehicle is continuously withdrawn from contact with the gases, mixed with a larger quantity of the same vehicle, cooled, and returned into contact with the carbon-laden gases. Any unabsorbed oil-laden soot particles are removed by electrical precipitation after reducing the velocity of flow of the gases.

H. S. GARLICK.

Hydrogenation of coal and like carbonaceous material. C. H. LANDER, F. S. SINNATT, and J. G. KING (B.P. 302,629, 17.9.27).—Sulphur compounds in the gases or vapours formed during the hydrogenation of carbonaceous material under pressure are removed by withdrawing the gases through a by-pass on the reaction vessel, to which they are returned after passage through a treatment chamber. If desired, other constituents, *e.g.*, carbon dioxide or volatile hydrocarbons, may be removed from the gaseous phase in the same manner.

A. B. MANNING.

Production of liquid products from coal and other solid carbonaceous material. M. MELAMID (B.P. 285,879, 24.2.28. Ger., 24.2.27).—The finely-divided material is subjected to the action of hydrogen at a high pressure and a relatively high temperature in the presence of a catalyst, the material and catalyst, which

is also in a finely-divided form, being injected simultaneously into the reaction chamber by means of the hydrogen used for effecting hydrogenation.

A. B. MANNING.

Hydrogenation of organic substances, especially of the products of the distillation of coals and petroleum oils. G. HUGEL, M. PAUL, and M. BOISTEL (B.P. 286,206, 18.7.27. Fr., 28.2.27).—Tar, pitch, heavy petroleum fractions, etc. are hydrogenated under pressure in the presence of catalysts consisting of the hydrides of the alkalis or alkaline-earths. The catalysts are prepared by heating the metal with hydrogen under pressure and are then stabilised by the addition of an oil such as tetrahydronaphthalene or of a substance more easily oxidisable than the catalyst itself, *e.g.*, sodium amide.

A. B. MANNING.

Distillation of oils, coal tar, and other liquids. GAS LIGHT & COKE CO., W. G. ADAM, and F. M. POTTER (B.P. 303,038, 27.8.27).—The material to be fractionated is passed through one or more heating coils while being maintained under pressure, and is then allowed to expand suddenly, the liquid and vapour being withdrawn separately and passed in heat-exchanging relation with the material passing to the heating coils. The vapour is condensed directly, or is further fractionated in a suitable column, supplemented, if desired, by a reheating still. The soft pitch passing from the heating coils is treated with superheated steam in a column down which it passes countercurrent to the steam, producing a pitch residue of any desired m.p.

A. B. MANNING.

Gas-producing plant. O. Y. IMRAY. From P. B. MORGAN (B.P. 303,554, 5.10.27).—A gas producer of the type having a device for levelling the surface of the fuel bed is provided with a number of such devices operating at different levels of the fuel layer. These have counterweights of different sizes, or are otherwise adjustable as to their position.

A. B. MANNING.

Process for producing gas. L. P. BURROWS (U.S.P. 1,695,914, 18.12.28. Appl., 13.11.22. Renewed, 13.1.28).—Coal is heated in a retort enclosed within a furnace. A current of steam is passed through iron pipes embedded within the furnace walls, whereby part of it reacts with the metal of the pipes producing hydrogen. The mixture of superheated steam and hydrogen is then passed through the retort, which can be rotated or is provided with other means for agitating the fuel contained therein.

A. B. MANNING.

Generating gases for power development. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 299,792, 12.9.27. U.S., 25.10.26).—The hot, waste gases from a boiler plant are passed to a coal-distilling apparatus of the type described in B.P. 277,660 and 291,062 (B., 1929, 160, 4), wherein finely-pulverised coal is carbonised without any agglomeration of the particles taking place. The carbonised particles are then passed directly to a gas generator to which air and, if desired, steam also is admitted, and in which the fuel is converted completely into gas and ash. The ash is discharged and the gas is returned to the boiler plant to be consumed therein.

A. B. MANNING.

Process of combined absorption and fractionation for recovering gasoline from gas. E. R. Cox

and D. A. CUSHMAN (U.S.P. 1,695,139, 11.12.28. Appl., 31.8.26).—The oil used for the absorption of the gasoline is first cooled to air temperature and part is then further cooled to a considerably lower temperature. The refrigerated oil is fed in at the top of the absorption tower up which the compressed gas is passed, whilst the remainder of the oil is fed in at a lower level.

A. B. MANNING.

Determining the content of combustible gas in gas mixtures. SIEMENS & HALSKE A.-G. (B.P. 282,080, 8.12.27. Ger., 10.12.26).—Combustible gas is determined by the variation in resistance of an electrically-heated wire which brings about the catalytic combustion of the gas as the gas mixture passes over it. A uniform and constant position of the wire is attained by mounting it in such a way that it is stretched in the direction of the longitudinal axis of the chamber, and that all the insulated parts required for this mounting are arranged before the point in the current of gas at which the catalysis takes place. Thus two metal pins may be arranged close to the inlet opening for the gas current, and the wire may be stretched between one of these and a bracket supported by the other.

A. B. MANNING.

Joint manufacture of valuable liquid and other hydrocarbons and derivatives thereof and ammonia. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,620, 17.9.27).—The methane and other hydrocarbons in the waste gases from the hydrogenation of carbonaceous materials are converted into hydrogen, *e.g.*, by passage of the gas, admixed with air and steam, over a nickel catalyst at 600°, and the resultant mixture of hydrogen and nitrogen obtained after the further catalytic conversion of the carbon monoxide and elimination of the carbon dioxide is used for the synthesis of ammonia. A. B. MANNING.

Preparation of hydrocarbon products. N. E. LOOMIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,694,272, 4.12.28. Appl., 13.7.23).—A light condensate containing some normally gaseous hydrocarbons is introduced into the intermediate region of an absorber tower up which vaporising constituents pass through progressively cooler zones countercurrent to a flow of heavy naphtha under a pressure of 100–300 lb./in.², and a balanced gasoline is withdrawn from the bottom of the tower.

H. S. GARLICK.

Production of homogeneous mixtures of aliphatic mineral oils or distillates and aliphatic alcohols. J. KIRSCHNER (B.P. 277,357, 9.9.27. Ger., 9.9.26).—Mechanically prepared mixtures of the components at raised temperatures are subjected to the action of nascent hydrogen prepared by passing the mixtures with the addition either of organic acids or salts, *e.g.*, oxalic acid or oxalates, into contact with heated metals or alloys, m.p. above 100° (*e.g.*, lead and nickel mixture), or with hydrochloric acid they are passed in vapour form over a granulated metal such as zinc. On condensation a homogeneous mixture is obtained, the use of which is governed by the b.p. of the original fraction of mineral oil selected.

H. S. GARLICK.

Apparatus for treating emulsified oil. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,694,643, 11.12.28. Appl., 21.8.20. Renewed 28.5.28).—A horizontal cylindrical still is heated by a pair of burners of about the same length as the still and arranged to heat its walls in a plane above the highest liquid level in the still so that oil is distilled only from the surface. A rotating shaft fitted with propeller blades is provided in the lower part of the still, below the liquid level. The oil is simultaneously dehydrated and fractionated.

L. A. COLES.

Production of sulphonated products [emulsifying agents etc.]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,281, 13.1.28).—Oxidation products of paraffin waxes, separated or not from unsaponifiable matter, are sulphonated with oleum or chlorosulphonic acid to give emulsifying and wetting-out agents.

C. HOLLINS.

Apparatus for refining oils. J. PRIMROSE, Assr. to FOSTER WHEELER CORP. (U.S.P. 1,693,156, 27.11.28. Appl., 21.6.22).—An oil-heating apparatus consists of a combined furnace and tube chamber separated by a bridge wall over which heating gases enter the tube chamber at the top, leaving by an outlet at the bottom. Oil is pumped from an external tank through a bank of horizontal tubes disposed at successively higher levels in the tube chamber, and thence back into the chamber itself.

H. S. GARLICK.

Manufacture of hydrocarbons and substances containing adsorbed hydrocarbons. GEN. CARBONALPHA Co. (B.P. 278,745, 7.10.27. Holl., 7.10.26).—Hydrocarbons are produced by heating active carbon with hydrogen, or a gas containing hydrogen, at between 150° and 600° and under ordinary or slightly higher pressures. When an excess of active carbon is used the hydrocarbons are adsorbed thereon and a plastic or solid material is produced, which may be used as a fuel or illuminant. The active carbon is obtained by heating carbon monoxide at between 230° and 600°. If desired, catalysts may be used in the production both of the active carbon and of the hydrocarbons.

A. B. MANNING.

Revivification of [earthy] filtering material [used for filtering mineral oils]. L. W. NAYLOR, Assr. to CONTINENTAL OIL Co. (U.S.P. 1,693,206—7, 27.11.28. Appl., [A] 31.8.22, [B] 3.3.23).—The material is treated in the filtering chamber with a current of benzol flowing in the reverse direction to the mineral oil when the filter is in normal use, and, when the discharge is nearly colourless, the material is submerged in benzol for a period of time depending on the relative proportions of benzol and material. The benzol is finally drawn off and the residue treated with a current of steam.

L. A. COLES.

Treatment of spent clays [from oil-treating processes]. R. A. DUNHAM, Assr. to UNION OIL Co. OF CALIFORNIA (U.S.P. 1,694,971, 11.12.28. Appl., 30.10.26).—The spent clay is brought into intimate contact with the quantity of water, as determined experimentally, capable of causing the maximum separation of oil.

L. A. COLES.

Production of high-viscosity oils from mineral oils and tar oils of any origin. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,776, 8.9.27).—The initial materials are freed from alcohols, primary and secondary bases, phenols, or other ionisable substances, and subjected in the coherent liquid phase to the action of alternating currents of frequencies lying between 500 and 10^4 periods/sec. H. S. GARLICK.

Production of hydrocarbons of low b.p. from those of high b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,761, 3.9.27 and 25.2.28).—Readily volatile hydrocarbons are obtained from hydrocarbons of any origin, including tars, by subjecting them to the action of a gaseous halogen hydride at temperatures above 100° in the presence of metals such as aluminium, copper, lead, magnesium, iron, cobalt, chromium, or alloys of these, activated by a solution of a salt of a less electropositive metal, and with or without the addition of a metallic chloride. The speed of the initial material is chosen so low that considerable quantities of hydrocarbons of b.p. below 70° are formed. This is augmented by increasing the rate of flow of the halogen hydride. Introduction of the materials counter-current to one another is advantageous.

H. S. GARLICK.

Lubricants for low-temperature expansion machines in which benzol is separated from coke-oven gases. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 277,378, 12.9.27. Ger., 13.9.26).—A lubricant having a low temperature of solidification and preventing the separation of solid ice, benzene, or naphthalene in the cylinder or valves of the expansion machine is produced by the addition of toluene, other homologues of benzene, or a hydrogenated naphthalene, and a glycol, ketone, or alcohol to the ice-machine oil.

A. B. MANNING.

Oil-gas apparatus. A. J. BASSETT, ASSR. to BRUCE MACBETH ENGINE CO. (U.S.P. 1,694,910, 11.12.28. Appl., 31.5.24).—Oil is cracked by the heat produced by its partial combustion within a closed retort. The tube conveying the oil-air mixture into the retort projects into a wider retort tube which is closed at the further end. The gases and vapours formed therefore pass back through the annular space between the two tubes and then forward again between the outer tube and the retort to the outlet.

A. B. MANNING.

Treatment [cracking] of hydrocarbons. G. EGLOFF (U.S.P. 1,692,203, 20.11.28. Appl., 19.7.18. Renewed 21.4.28).—Hydrocarbon oil is cracked at 230 – 260° in presence of 1–2% of aluminium chloride (generated *in situ* by action of hydrogen chloride on aluminium powder in presence of mercuric chloride); the gasoline vapours escape to a condenser while the unvaporised oil and aluminium chloride are withdrawn continuously from the bottom of the still and returned through a spray nozzle at the top. R. BRIGHTMAN.

Production of gasoline and other light hydrocarbons from heavier hydrocarbons. B. ORMONT, ASSR. to B. ORMONT ASSOCIATES, INC. (U.S.P. 1,691,300, 13.11.28. Appl., 9.10.23).—Hydrocarbon oil and water are vaporised independently under pressure, *e.g.*, at 180° , and in such proportion that for the production

of gasoline the total latent heat of the oil is approximately equal to that of the water, *i.e.*, a ratio of oil/water of 7–10/1; for the production of lubricating oils the latent heat of water is in excess (oil/water ratio of 7–1/1). The unvaporised oil flows into the steam-coil where it is completely vaporised, and the resultant mixtures are passed through cracking coils. R. BRIGHTMAN.

Purification of benzol, petrol, and the like. K. COX and P. J. McDERMOTT (B.P. 303,991, 8.12.27. Addn. to B.P. 269,242; B., 1927, 436).—The oils or spirits are treated in the washer or agitator with ferric sulphate, and, after filtering, the filtrate is neutralised with alkali prior to distillation. H. S. GARLICK.

Purification of the wax residues of petroleum and shale oil distillation and apparatus therefor. STEEL BROS. & CO., LTD. From D. ARMSTRONG and G. C. G. DOBBIE (B.P. 303,812, 8.7.27).—An apparatus for sweating or purifying paraffin wax or scale comprises a chamber containing a tank or vat fitted with a steam inlet and having a bottom, inclined to one or more drain points provided with cocks, above which is a horizontal shelf of wire cloth. Above the shelf are situated multiple layers of horizontal piping arranged in a series of closely-spaced vertical supports for the wax, which is thus prevented from forming a compact mass. Suitable means are provided for circulating cooling or heating fluids through the coils.

H. S. GARLICK.

Bleaching of montan wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,080, 22.9.27).—The crude or deresinified wax is heated with an aqueous solution containing chromic acid and an organic acid such as acetic or benzenesulphonic acid. The chromic acid may be replaced by the equivalent amount of a chromate.

A. B. MANNING.

Bleaching of montan wax, fractions thereof, or wax alcohols or acids obtained therefrom. A. RIEBECK'SCHE MONTANWERKE A.-G., and T. HELLTHALER (B.P. 303,036, 26.8.27).—The materials are treated with an oxidising agent, *e.g.*, hydrogen peroxide or chromic acid, in the presence of aqueous sulphuric acid.

A. B. MANNING.

Treatment of oil-fuel refuse as obtained after cleaning and washing out of tanks or bunkers. H. W. WILDISH (B.P. 301,405, 28.7.27).—Oil-fuel residues are pumped through automatically cleanable strainers to remove sand and heavy sediment, then heated to 82° to separate water and dirt held in suspension (which is discharged through a sand filter), and the floated oil is finally passed through a centrifugal separator.

H. S. GARLICK.

Method of carbonisation or destructive distillation. S. McEWEN, ASSR. to INTERNAT. COAL CARBONISATION CO. (Re-issue 17,182, 1.1.29, of U.S.P. 1,481,140, 15.1.24).—See B., 1924, 166.

Decolorising carbon. E. URBAIN, ASSR. to URBAIN CORP. (U.S.P. 1,700,342, 29.1.29. Appl., 10.7.26. Fr. 25.7.25).—See B.P. 255,871; B., 1928, 6.

Purification of gases. W. H. FULWEILER, ASSR. to U.G.I. CONTRACTING CO. (U.S.P. 1,700,698, 29.1.29. Appl., 1.8.24).—See B.P. 237,920; B., 1925, 837.

Emulsification of tar, bitumen, creosote, petroleum, heavy oils, etc. W. E. BILLINGHAME (U.S.P. 1,700,581, 29.1.29. Appl., 21.11.25. U.K., 27.11.24).—See B.P. 252,449; B., 1926, 655.

Vertical-chamber coke oven. C. OTTO (U.S.P. 1,695,204, 11.12.28. Appl., 2.11.26. Ger., 14.10.25).—See B.P. 259,968; B., 1927, 436.

Pulverised fuel burners. H. E. YARROW (B.P. 304,079, 18.4.28).

Means [valve] for automatically controlling the temperature of a suction gas producer. H. C. READING and T. G. TULLOCH (B.P. 302,033, 16.11.27).

Charging furnaces [water-gas generators] with solid material. POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 302,852, 13.4.28. Addn. to B.P. 283,238).

Device for extracting impurities from lubricating oils. F. R. SIMMS and B. C. JOY (B.P. 303,076, 25.8.27).

Filter (U.S.P. 1,688,326).—See I. Lubrication of textile fibres (B.P. 277,637).—See V. Mixed fertiliser (B.P. 303,916). Manure and insecticide (B.P. 303,957).—See XVI.

III.—ORGANIC INTERMEDIATES.

Detection of isopropyl alcohol by means of mercuric sulphate solution (Denigès' reagent). H. MATTHES and P. SCHÜTZ (Pharm. Ztg., 1929, 74, 44—45).—A criticism of the work of Stainer and Lauwaet (B., 1928, 835). Mercuric sulphate solution gives with pure isopropyl alcohol a white, not a yellow, precipitate, which consists of mercurous sulphate and an indefinite compound of mercuric sulphate with the acetone formed by oxidation of the alcohol. Saturated hydrocarbons give no precipitate; unsaturated hydrocarbons and tertiary alcohols give yellow and red precipitates. The test is therefore not sufficiently specific. S. I. LEVY.

Colour reaction of diphenylamine and detection of this compound in Powder B. L. DESVERGNES (Ann. Chim. analyt., 1929, [ii], 11, 1—4).—A solution of diphenylamine in 95% alcohol gives a green coloration when treated with a few drops of chlorine water; the colour slowly changes to dark violet on keeping or on warming. Aniline under similar conditions gives a pale yellow colour, ethylaniline a pale rose to violet, and diethylaniline a pale mauve colour becoming yellow on warming. In testing Powder B for the presence of diphenylamine this substance should be removed by shaking the mixture with ether; the ethereal solution is then warmed with water to expel ether and the precipitated substance dissolved in alcohol for testing with chlorine. A. R. POWELL.

See also A., Feb., 140, Volatility with steam of water-soluble organic substances (VIRTANEN and PULKKI). 141, Synthesis of carbamide from ammonia, carbon dioxide, and water (TERRES and BEHRENS). 152, Autoxidation of benzaldehyde (KUHN and MEYER). 153, Catalysts for formation of alcohols from carbon monoxide and hydrogen (FROLICH and others). Oxidation of ethylene (REYERSON and

SWEARINGEN). 162, Titration of amino-acids and -salts. (KAPPELMEIER). 173, Catalytic hydrogenation under reduced pressure (ESCOURROU). 178, Analysis of carbamide (TAYLOR). 181, Reduction of nitro-compounds by aromatic ketols (NISBET). Metallic complexes of o-amino- and o-hydroxyazo-derivatives (CRIPPA). Action of phthalic anhydride on aminoazo-derivatives (CRIPPA and GALIMBERTI). Thio-carbamido-derivatives in the azo series (CRIPPA). 184, Naphthenic acids of Japanese petroleum (KUWATA). Catalytic reduction of nitriles and oximes (HARTUNG). Naphthalic acid derivatives (DZIEWONSKI and KOCWA). 200, Substituted μ -methyl-benzthiazoles and dyes therefrom (SCHULOFF and others). 205, Determination of anthraquinone in mixtures with benzantrones (SOKOLOV and GUREVICH).

Thermal decomposition of methane; and of benzene and phenol. KUSAMA and UNO; KOSAKA.—See II. Abietic anhydride. NAGEL.—See XIII.

PATENTS.

Manufacture of concentrated acetic acid. SOC. ANON. PROGIL (F.P. 622,680, 6.2.26).—Dilute acetic acid is mixed with a substance, such as butyl or amyl alcohol, mesitylene, methyl isobutyl ketone, butyl acetate, etc., which boils below 150°, which when mixed with more than 50% of water vapour boils below 95°, and which has a solubility in water of less than 10 g./100 c.c. The mixture is distilled through a rectifying column, and the mixture of water vapour and solvent vapour obtained is separated in a second column.

A. R. POWELL.

Manufacture of dioléfines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,323, 30.4.28).—Butadiene and other diolefines are separated from hydrogen, methane, etc. by absorption in aqueous solutions of salts of metals of groups I and II, and are recovered by heating or by reducing the pressure. With cuprous chloride solutions butadiene gives a solid precipitate, and may thereby be separated from ethylene, propylene, and butylene. C. HOLLINS.

Oxidation of hydrocarbons. Manufacture of organic acids from petroleum hydrocarbons. A. W. BURWELL, Assr. to ALOX CHEM. CORP. (U.S.P. 1,690,768—9, 6.11.28. Appl., [A] 13.9.22, [B] 31.5.23).—(A) Fluid hydrocarbons containing an aliphatic chain, e.g., petroleum distillates, are agitated in the liquid phase above 100° at 100—320 lb./in.² in a current of air or other oxidising gas until the total acid content is about 20%. (B) The initial products insoluble in the hydrocarbon mixture are separated and the oxidation is continued. As example, distillate, *d* 0.8139, from Pennsylvanian crude oil yields 5% of formic acid, 5% of acids $C_{17}H_{14}O_2$ to $C_{19}H_{24}O_2$, and 90—95% of liquid acids, mol. wt. above 300; wax affords 5—10% of formic acid, 5—10% of other volatile acids, and 100—110% of acids, m.p. 45°. R. BRIGHTMAN.

Manufacture of acyl derivatives of β -ketonic esters and β -diketones. IMPERIAL CHEM. INDUSTRIES, LTD., and S. COFFEY (B.P. 302,750, 6.10.27).—Sodio-derivatives of β -ketonic esters or β -diketones react

with carboxylic acid anhydrides to give in good yield mainly *C*-acyl derivatives. The preparation of ethyl diacetate, b.p. 104–106°/20 mm., ethyl *C*-propionylacetate, and *C*-butyrylacetate is described.

C. HOLLINS.

Hydrogenation of products containing phenol, benzene, or naphtha. C. LAILLET and J. GIUSTINIANI (F.P. 622,637, 1.2.26).—The substance is heated with hydrochloric acid and bituminous limestone. Hydrogen is stated to be evolved according to the reaction: $\text{CaCO}_3 + 2\text{HCl} = \text{CaCl}_2 + \text{CO}_2 + \text{O} + \text{H}_2$.

A. R. POWELL.

Manufacture of solid alkali-metal salts of organic compounds. K. DOBMAIER, Assr. to I. G. FARBENIND. (U.S.P. 1,685,191, 25.9.28. Appl., 19.1.28. Ger., 18.1.27).—The organic compound is ground with sodium phenoxide, and the phenol extracted with ether or alcohol. Sodium *p*-nitromercuriphenoxide, sodium cyanomercuriphenoxide, and sodium nitrophenylstibinate are thus obtained from the free phenolic compounds and sodium phenoxide. R. BRIGHTMAN.

Purification of benzoic acid and its derivatives. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,685,634, 25.9.28. Appl., 19.10.27).—Benzoic acid and its chloro- and nitro-derivatives are separated from phthalic acid and its corresponding derivatives by passing the aqueous solution or dispersion in countercurrent with an organic solvent.

R. BRIGHTMAN.

Catalytic oxidation of anthracene. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,685,635, 25.9.28. Appl., 23.12.27).—Anthracene is oxidised to anthraquinone in presence of a zeolite catalyst which may be diluted with a porous diluent, such as kieselguhr, siliceous powders, etc. The catalytically-active element may be present in chemical combination with or in the zeolite, or non-catalytic zeolite may be impregnated or incorporated during formation with suitable catalysts. Alkalinity of the zeolites may be avoided by effecting the zeolite reactions in only faintly alkaline solution or by treatment of the product with an acid gas at 400–500° in presence of air or other oxidising medium. If necessary, the zeolite catalyst may be strengthened mechanically with water-glass. Zeolite catalysts containing vanadium in non-exchangeable form as a metallic oxide component are preferred.

R. BRIGHTMAN.

Manufacture of wetting, cleansing, and emulsifying agents and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,258, 8.9.27).—Aromatic or hydroaromatic non-hydroxylated compounds or their sulphonic acids are condensed with polyhydric alcohols or their esters or ethers containing at least one free hydroxyl group, or with unsaturated alcohols, or with olefines containing several double linkings, with a subsequent sulphonation if necessary. Examples are: naphthalene with α -butylene glycol or glycol monopropyl ether.

C. HOLLINS.

Manufacture of thiocarbamide from cyanamides. COMP. DE L'AZOTE ET DES FERTILISANTS SOC. ANON. (Swiss P. 119,471, 11.2.26).—Calcium cyanamide and calcium sulphide in the theoretical proportions are added slowly to about five times their weight of water,

and the solution is treated with carbon dioxide until all the calcium is precipitated as carbonate. The mixture is then heated at 75° for 1–1½ hrs. with vigorous stirring, filtered, and evaporated for the recovery of thiocarbamide.

A. R. POWELL.

Liquid solution of thiocarbamide. H. GRAY, Assr. to B. F. GOODRICH Co. (U.S.P. 1,690,166, 6.11.28. Appl., 28.4.27).—Binary and ternary etc. mixtures of amines and diamines dissolve 20–40% of the thiocarbamide. Examples are: aniline with dimethyl-*p*-phenylenediamine; aniline with dimethyl-*p*-phenylenediamine and xylydine; aniline with *o*- and *p*-toluidines.

R. BRIGHTMAN.

Production of nitro-, amino-, and hydroxyl-amino-derivatives of cymene. J. SCHINDELMEISER and J. A. F. BEAVIS (B.P. 300,760, 18.10.27).—Cymene is dissolved, e.g., in an equal weight of sulphuric acid, and nitrated with the theoretical amount or a slight excess of an organic nitrate, particularly "diacetyl nitric acid," the nitrocymene being subsequently reduced by known methods.

R. BRIGHTMAN.

Hydrogenation of naphthalene. E. A. PRUDHOMME (F.P. 599,827, 19.5.24).—A mixture of naphthalene vapour and hydrogen or water-gas is passed through a tube packed with Laming's material and heated at 190–225°, then over nickel pumice. According to the working conditions the resulting product consists of more or less of the various hydrogenation products from di- to deca-hydronaphthalene.

A. R. POWELL.

Manufacture of *N*- ω -aminoalkylamino-naphthalenecarboxylic [-naphthoic] acids. I. G. FARBENIND. A.-G., and W. HENTRICH (B.P. 302,212, 12.9.27. Addn. to B.P. 230,457; B., 1925, 983).—Alkylenediamines are condensed with hydroxy- or amino-naphthoic acids (other than those containing the hydroxyl or amino-groups *ortho* to the carboxyl group) in presence of sodium bisulphite. 2- β -Aminoethylamino-6-naphthoic acid and 2- δ -aminobutylamino-6-naphthoic acid are described; they couple with diazotised *p*-nitroaniline-*o*-sulphonic acid to give a blue-violet and a reddish-blue dye, respectively.

C. HOLLINS.

Manufacture of 1:8-naphthoxyphenanthrophen [peri-naphththioindoxyl] compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,771, 25.10.27).—8-Carboxy- α -naphthylthioglycolic acid, or its nuclear substitution derivatives, is cyclised by heating with water at 150–200° in a closed vessel. *peri*-Naphththioindoxyl, m.p. 84–85°, and a bromo-derivative, m.p. 130°, prepared from brominated thioglycolic acid, m.p. 230°, are described.

R. BRIGHTMAN.

Purification of camphor and similar volatile substances. E. G. LOOMIS (U.S.P. 1,693,243, 27.11.28. Appl., 27.8.25).—Molten camphor is vaporised by blowing on to its surface a gas heated at below its b.p., and the gas saturated with camphor vapour is drawn by suction into a condensing chamber.

L. A. COLES.

Manufacture of acetic acid. H. DREYFUS (U.S.P. 1,697,109, 1.1.29. Appl., 27.5.26. U.K., 13.6.25).—See B.P. 259,641; B., 1926, 1013.

Recovery of concentrated acetic acid. H. SUIDA

(U.S.P. 1,697,738, 1.1.29. Appl., 16.3.27. Austr., 31.7.26).—See B.P. 275,158; B., 1927, 956.

Quantitative halogenisation of perylene and its derivatives. A. PONGRATZ and A. ZINKE, Assrs. to F. BENSA (U.S.P. 1,697,137, 1.1.29. Appl., 30.9.25. Austr., 19.12.24).—See B.P. 244,739; B., 1926, 974.

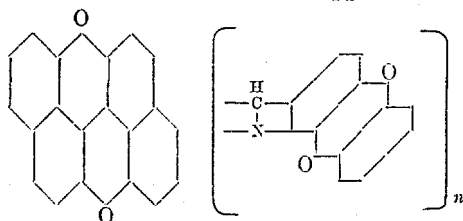
Cyanogen chloride (B.P. 303,469).—See VII.

IV.—DYESTUFFS.

See A., Feb., 183, **Triphenylcarbinol** or "aniline" dyes (MOIR). 190, **Colour of quinydrones** (WEITZ). **Indoquinonanthrene** (MEYER). 197, **Carbocyanine dyes** (HAMER). 200, **New heterocyclic polymethine dyes** (SCHULOFF and others).

PATENTS.

Manufacture of vat dyes of the anthanthrone series. I. G. FARBENIND. A.-G. (B.P. 296,373, 29.8.28. Ger., 30.8.27).—Fast bright-brown vat dyes (annexed formula; $n = 1$ or 2) of the anthanthrone series are obtained by condensing with 1-aminoanthraquinone-2-aldehyde the mono- or di-halogenated anthanthrones prepared, *e.g.*, according to the process of B.P. 287,020 (B., 1928, 398) in presence of an acid-fixing agent, *e.g.*, sodium acetate, a solvent, and copper or a copper



compound as catalyst. Dyes from mono- and di-bromo- and dichloro-anthanthrone are described.

R. BRIGHTMAN.

N- ω -Aminoalkylaminonaphthoic acids (B.P. 302,212).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Electrical conduction in textiles. I. **Dependence of the resistivity of cotton, silk, and wool on relative humidity and moisture content.** E. J. MURPHY and A. C. WALKER (J. Physical Chem., 1928, 32, 1761—1786).—Data showing the variation of the insulation resistance of cotton, silk, and wool with R.H. and moisture content are recorded. The logarithm of the resistance plotted against the logarithm of the moisture content gives a linear curve, the slope of which is independent of the form of the sample (short threads, individual fibres, and standard cotton-insulated wires) and the amount of impurity present, but which is characteristic of the kind of material investigated. The resistance at a given humidity is dependent on the amount of impurities, indicating that conduction occurs largely through aqueous paths. For samples of cotton, the insulation resistance at 1% R.H. is approx. 10^{12} greater than that at 99%, and in the range 20—80% is an exponential function of R.H. The conductance of a cotton thread is equal to the sum of the conductances of the constituent fibres, directly proportional to its

length, and is apparently independent of any contact resistances. Resistance decreases with a rise in temperature, but is little affected by small changes in tension of the fibres. Equations by which the resistances of cotton, silk, and wool can be calculated are given. The rate of change in resistivity with moisture content is greater for silk and wool than for cotton, and for a given moisture content the resistivity of silk or wool is greater than that of cotton, in spite of their greater hygroscopicity. This is explained by assuming that the effective conducting water channels in a textile consist of elementary filaments which are alternately expanded and constricted lengthwise, and that the resistivity is determined by the cross-sectional area of the narrowest parts of the channel, which are smaller in silk and wool than in cotton. L. S. THEOBALD.

Substances accompanying cellulose. I. F. W. KLINGSTEDT (Biochem. Z., 1928, 202, 106—114).—Cellulose preparations (sulphited cellular material of pine wood) can be completely freed from pentosans by treatment with sufficient 17—18% sodium hydroxide for 1 hr. With 6% sodium hydroxide, the first treatment of crude cellulose removes about half of the pentosans, and 90% is removed by two treatments. Saturated baryta at 50° removed only about one quarter of the pentosans. The cellular material obtained from rye straw by successive chlorinations contains large amounts of pentosans, which can be removed by treatment with 17% sodium hydroxide. P. W. CLUTTERBUCK.

Chemistry of the sulphite-cellulose cooking process. E. HÄGGLUND (Papier-Fabr., 1929, 27, 49—54).—Methods of determining the constituents of wood are discussed. Pine wood consists of cellulose about 42%, polysaccharides 24%, lignin 28%, acetic acid 2%, and rosin, ash, protein, etc. about 4%, the amounts varying somewhat with the method of determination. In the cooking of sulphite-cellulose, about three fourths of the hemicellulose originally present goes into solution as easily hydrolysed polysaccharides, whilst the other fourth remains in the cellulose. In the formation of lignosulphonic acids from the wood lignin, considerable amounts of sulphurous acid and lime are absorbed by the lignin and stable chemical compounds are formed. By treatment with hot dilute acids the greater part of the lignin is converted into lignosulphonic acids in solution. The ratio of sulphur to methoxyl in this material is about 0.5, whilst that found for ordinary sulphite waste-liquor is about the same, so that the lignin in the wood is already completely sulphonated at a relatively early stage of the cooking. The separation of the lignosulphonic acids in the dissolved state is dependent on the acidity and temperature of the cooking liquid, and this separation under the influence of acid is probably hydrolytic in nature, a solid lignosulphonic acid being first formed which subsequently goes into solution. Separation of the lignin is best produced by the use of cooking liquor of low hydrogen-ion concentration, *e.g.*, sodium bisulphite solution, but under these conditions longer cooking is necessary, although yields of 60% of easily defibreable, very white sodium bisulphite-cellulose can be obtained. With ordinary cooking liquor such high yields cannot be obtained as the greater

acidity causes dissolution of the hemicellulose. With calcium bisulphite yields of 55% of cellulose with 7–8% of lignin may be obtained. The lignin, hemicellulose, and cellulose are all affected by overcooking, and not only is the yield of cellulose poor, but its strength is reduced. The formation of sugars during cooking and the fluorescence and reddening of the cellulose due to the presence of lignosulphonic acids are discussed.

B. P. RIDGE.

Alteration of sugar-like substances in sulphite-cellulose cooking. I. E. HÄGGLUND and T. JOHNSON (Svensk Kem. Tidskr., 1929, 41, 8–11).—The variation of the sugar content of spent sulphite liquors from cellulose manufacture depends mainly on (a) the hydrolysing action of the acid used, which in turn depends on the hydrogen-ion concentration, and hence on the concentrations of sulphite and sulphur dioxide, and (b) the degrading action of the acid on dissolved sugar-like substances. The latter depends not on the acidity of the sulphite solution used, but on the oxidising action of the bisulphite ion which oxidises the sugars to the corresponding aldo-acids in accordance with the scheme $2\text{HSO}_3^- + 2\text{C}_6\text{H}_{12}\text{O}_6 \rightarrow \text{S}_2\text{O}_3^{2-} + 2\text{C}_6\text{H}_{12}\text{O}_7 + \text{H}_2\text{O}$. This oxidising action depends on the concentration of the bisulphite ion, which is governed by the proportion of lime in the sulphite liquor. If the latter is deficient in lime a point is soon reached when it is all used up in combination with the lignosulphonic acids formed, and the sulphite-ion concentration is greatly diminished, so that the sugars produced are not oxidised but are found in the spent lye. When a high proportion of lime and a longer heating period are used a considerable quantity of the aldo-acids are found in the spent lye, and their isolation as calcium salts is described. J. W. BAKER.

Factors affecting the sizing of paper pulp. R. ESCOURROU (Chim. et Ind., 1928, 20, 1039–1042).—A review of previous work in this field. D. J. NORMAN.

See also A., Feb., 175, **Constitution of flax cellulose** (RIGBY). **Acetylation of beech wood** (HORN).

PATENTS.

Treatment of plant stems to obtain fibres therefrom. C. E. BAHRE (B.P. 302,052, 14.12.27).—Stem fibres are disaggregated by boiling with alkali under pressure, with the addition of cholesterol or substances rich in cholesterol, such as fish oil, egg yolk, wool fat, etc., which protect the fibre from decomposition by the alkali. F. R. ENNOS.

Spinning of linen, hemp, and other fibres. G. PETROV and N. ALEXEV (F.P. 628,626, 5.2.27).—To accelerate the absorption of moisture the fibres are treated with water containing small quantities of aromatic sulphonic acids with or without the addition of hydrocarbons, sulphonic acids of aromatic hydrogenated hydrocarbons (octahydroanthracene), naphthasulphonic acids, aromatic fatty acids, alcohols, etc., or their derivatives (salts). F. R. ENNOS.

Lubrication of textile fibres. STANDARD DEVELOPMENT Co., Assees. of A. E. BECKER and DE V. STONAKER (B.P. 277,637, 22.8.27. U.S., 14.9.26).—Fibres are lubricated, previous to spinning, weaving, knitting, etc., with an emulsion containing a hydrocarbon having a

viscosity of 50–200 sec. Saybolt at 38° and 0.15–0.20% of an alkali soap as a dispersing agent. A. J. HALL.

Treatment of [pile] fabrics. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. E. WALKER (B.P. 302,208, 12.9.27).—For the production of ornamental effects in pile fabrics consisting of a cellulose acetate pile on a backing of other material, a solvent such as lactic, formic, or acetic acid, acetone, etc., with the addition of a suitable material to prevent spreading, is applied to the back of the fabric to weaken the loops of the pile filaments without damaging the other material. The pile filaments from the treated portion are afterwards removed by mechanical means. F. R. ENNOS.

Extraction of cellulose or paper pulp from fibrous vegetable matter containing the same. E. SPENCER (U.S.P. 1,690,954, 6.11.28. Appl., 10.2.28. Brit. India, 20.1.27).—The cellulosic material is introduced into a number of digestors which can be put into communication with one another. The digesting liquor is added to one and, after dissolution of part of the soluble matter in the material, it is blown down by means of the steam into the next digester containing the less extracted material and replaced by liquor of greater concentration, of higher temperature, and under greater steam pressure. This process of reverse extraction is continued so that the partially spent liquor from each stage is utilised in similar extractions of the less extracted material in another digester, whilst the most extractive liquor is applied to the material which has been most completely extracted. F. R. ENNOS.

Preparation of fibres or cellulose from graminaceous or other plants of high siliceous content. U. GENNARO and G. ROSSI (B.P. 301,226, 30.12.27).—The raw material is treated at not above 40° with a reagent, e.g., 3–4% caustic soda solution, which dissolves silica. After removal of the excess of reagent, by washing or by neutralisation, the material is retted, either by bacteria or enzymes, and allowed to season. D. J. NORMAN.

Decomposition of wood with nitric acid. E. STERZL (Austr. P. 107,590, 23.3.25).—Wood fibres are heated for about 1 hr. at 90–100° with about 8% nitric acid, removed from the acid, washed, bleached, and worked up as desired. The used acid is neutralised with potassium hydroxide and lime, volatile acids are removed by evaporation, potassium nitrate which crystallises on cooling is removed, and calcium oxalate is recovered from the mother-liquor by the further addition of lime. L. A. COLES.

Production of cellulose esters. E. ELÖD (B.P. 275,641, 4.8.27).—Cellulose is treated with 98–100% formic acid at 5–30° in the presence of an esterifying catalyst, e.g., gaseous hydrochloric acid or phosphoric anhydride, and at least 40% (on the weight of cellulose) of a swelling agent such as zinc chloride or calcium bromide. When esterification has reached the desired degree (1–4 hrs., depending on temperature) the reaction is immediately stopped by the addition of 25–33% (on the weight of the reaction mixture) of water, and the product is washed and dried. D. J. NORMAN.

Production of cellulose acetate and mixed cellulose esters containing acetyl and other carboxylic

radicals. C. RUZICKA (B.P. 303,432, 3.10.27).—Glacial acetic acid is treated with dry chlorine at below 30° in the presence of a lower fatty acid anhydride and/or one or more halogen compounds of acetic or other lower fatty acid. Dry cellulose is then treated with this chlorinated acid at not above 50° and, when chlorination and hydration of the cellulose have proceeded to the required degree, the product is acetylated at not above 30° by means of acetic anhydride and a suitable catalyst, e.g., zinc chloride or sulphuryl chloride.

D. J. NORMAN.

Cellulose esters containing unsaturated organic acid groups. H. T. CLARKE and C. J. MALM, ASSTS. to EASTMAN KODAK Co. (U.S.P. 1,690,620, 6.11.28. Appl., 4.5.27).—Cellulose is treated, e.g., in presence of chloroacetic anhydride and magnesium perchlorate at 60–65° with an unsaturated monocarboxylic acid, affording, e.g., *cellulose crotonate*, *cellulose cinnamate*. Mixed esters, such as *cellulose crotonate stearate*, *cellulose acetate undecenoate*, and *cellulose acetate crotonate* are similarly obtained. Esterification may be effected in chloroform or in chlorobenzene at 155–157°.

R. BRIGHTMAN.

Spinning of artificial silk and the like. J. HUEBNER and K. F. DIEHL (B.P. 303,421, 28.6.27).—Fine filaments of artificial silk (0.5 denier) are obtained by using a coagulating solution containing, e.g., 50% (or stronger) sulphuric acid, to exert a plasticising effect on the coagulated filaments, and spinning at high speed, e.g., 125 m./min. so that the friction between the filaments and the coagulating solution induces stretching. To prevent eddy currents in the bath partitions should be provided therein.

D. J. NORMAN.

Manufacture of artificial silk by the dry-spinning method. A. E. GULL (B.P. 303,778 and 303,821, [A, B] 5.10.27).—Air is drawn into one end of a drying chamber, consisting of a central tube surrounded by a larger annular space, and is heated by passing through a duct having (A) variable electrical heating elements, or (B) both variable and invariable elements. It then passes through the central tube in contact with and in the same direction as the extruded filaments, the exit end of which is maintained at a higher temperature than the inlet.

F. R. ENNOS.

Production of artificial filaments or threads. BRIT. CELANESE, LTD., E. KINSELLA, J. BOWER, W. I. TAYLOR, and H. DREYFUS (B.P. 300,998, 23.5.27).—In the production of artificial filaments by the dry-spinning process, collector devices are fitted at the top of the spinning chamber to cause the whole of the air or other evaporative medium to flow in close proximity to the spinning nozzle. In this way it is possible to utilise practically the whole of the evaporative capacity of the air, and at the same time to exercise a stricter control on the cross-section of the filaments. Various types of collector devices are described.

D. J. NORMAN.

Knitting of rayon fibres. H. WADE. From STANDARD OIL Co. (B.P. 303,841, 6.7.27).—A mixture of a low-viscosity mineral oil, oleic acid, an emulsifying agent, e.g., an oil-soluble alkali salt of a sulphonated mineral oil, and an anti-oxidant, e.g., β -naphthol, is applied to the fibres to soften them and for lubrication purposes.

F. R. ENNOS.

Preparation of artificial textile products with reduced lustre from viscose. G. BONWITT (B.P. 285,066, 7.9.27. Holl., 11.2.27).—Before spinning, the viscose is treated with a mixture of mono- and di-chlorobenzene having substantially the same density as itself, and the whole is then emulsified.

F. R. ENNOS.

Manufacture of sheets or films of composition containing cellulose esters or ethers. SPICERS, LTD., and H. J. HANDS (B.P. 301,439, 30.8.27).—The sheets or films are passed through a bath containing dilute solutions of caustic soda (0.5–1%) or of sodium hydrogen sulphate, the whole being electrically earthed. A softening agent, e.g., alcohol, and an oxidising agent, e.g., hydrogen peroxide, may also be added to the bath, or the oxidation may be carried out subsequently in a separate bath.

F. R. ENNOS.

Production of strips composed of cellulose derivatives carrying monochromatic or multi-coloured figuring. I. G. FARBENIND. A.-G. (B.P. 288,592, 2.9.27. Ger., 13.4.27).—Figuring is applied to a supporting surface, e.g., by printing or spraying with a pigment (preferably suspended or dispersed in a cellulose acetate solution), and after being dried the supporting surface is covered with a solution of cellulose acetate by pouring or spraying on to the moving surface. When the film is stripped from the support it carries the figuring with it. Other cellulose derivatives may be used.

C. HOLLINS.

Treatment of fabrics containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 302,363, 14.12.28. U.S., 16.12.27. Addn. to B.P. 274,074; B., 1928, 811).—Ornamental effects are produced on fabrics containing cellulose esters by locally applying a hydrolysing agent, e.g., a paste composed of caustic alkali thickened with gum, steaming, washing, and finally subjecting the fabric to a carbonising agent, such as an acid or an acid salt solution, to remove the cellulose.

F. R. ENNOS.

Regeneration of spent liquor in the preparation of sodium bisulphite cellulose. E. L. RINMAN (F.P. 627,691, 18.1.27).—The spent liquor is freed from a number of constituents, concentrated by evaporation, and the solid matter is ignited to convert sodium salts into sodium sulphide containing carbonate. The sulphide is dissolved in water and converted into carbonate by passing carbon dioxide, and finally into bisulphite by treatment with sulphur dioxide from the combustion gases or the cooker.

F. R. ENNOS.

Recovery of resinous by-products in the manufacture of wood pulp. E. H. FRENCH (U.S.P. 1,693,586, 27.11.28. Appl., 15.8.25).—The alkaline liquor from the manufacture of wood pulp is acidified with waste sulphur dioxide or nitre cake, and the precipitated resin acid is separated by filtration or decantation, dissolved in a hydrocarbon, e.g., benzol, kerosene, the solution distilled *in vacuo*, and the solvent and rosin are collected separately.

F. R. ENNOS.

Production of wood pulp. C. B. THORNE (U.S.P. 1,691,682, 13.11.28. Appl., 5.4.26).—Wood chips are heated in a digester with a preheated sulphite liquor under pressure of an external gas, which also causes the

liquor to circulate or pulsate through a tank and back again to the digester. F. R. ENNOS.

Manufacture of chemical pulp. J. S. BATES (U.S.P. 1,691,511, 13.11.28. Appl., 2.3.25).—In the making of kraft pulp the melt of black ash and sodium sulphate is dissolved in dilute alkali solution from a previous operation so that the concentration is above that which is advantageous for causticising. The liquid while hot (90–100°) is allowed to settle, and the precipitate is washed for recovery of the alkali, the washings being added to the clear liquid, which is then causticised. F. R. ENNOS.

Production of a cellulose product. W. W. CARTER, Assr. to H. T. NELSON (U.S.P. 1,693,573, 27.11.28. Appl., 27.12.22).—For use in the manufacture of toughened, waterproof paper the fibre stock is immersed in a sodium hydroxide solution containing a size emulsion. After being removed from the solution and allowed to age, the product is xanthated. F. R. ENNOS.

Treatment of fibrous material. J. MCINTOSH, Assr. to DIAMOND STATE FIBRE CO. (U.S.P. 1,697,077, 1.1.29. Appl., 30.1.26).—Fibrous sheets are passed through a solution of cellulose ester, the solvent is evaporated, and the dried sheets are superposed and heated under pressure. J. S. G. THOMAS.

Manufacture of a [mouldable] composition of matter [from cellulose]. E. C. R. MARKS. From STABILIMENTI CHIM. IND. (B.P. 298,793, 15.11.27).—A highly resistant material capable of being moulded under heat and pressure is made by treating cellulose with a solution of sulphuric and nitric acids. After washing and drying, the product is steeped first in phenol and then in formaldehyde, neutralised if necessary, drained, dried, and finally mixed with 2% of *p*-dichlorobenzene. F. R. ENNOS.

Manufacture of derivatives of cork. I. G. FARBERIND. A.-G. (B.P. 291,773, 8.6.28. Ger., 8.6.27).—Dry cork meal, suspended in an indifferent medium, *e.g.*, chlorobenzene, is esterified, whereby an insoluble plastic material is obtained. F. R. ENNOS.

Manufacture of waterproof paper board. E. BACHE, Assr. to WALDORF PAPER PRODUCTS CO. (U.S.P. 1,691,752, 13.11.28. Appl., 8.5.25).—Paper pulp is treated with ferric chloride (0.7% by wt. of the dry material) to render it electropositive, and mixed with emulsified bitumen, which bears a negative charge, by forcing the two at high velocity through an annular orifice the width of which does not exceed 0.05 in. F. R. ENNOS.

Treating papermaking fibres in the making of certain kinds of paper. R. MCNEILL (B.P. 301,682, 6.3.28).—In order to avoid the production of "fluff" in the manufacture of certain bulky antique and other papers, a jelly consisting of starch, glue, sodium silicate, and water is thoroughly mixed with the fibre in the beater before the addition of alum. F. R. ENNOS.

Production of paper and paper stock. L. GRAFFLIN, Assr. to BALL BROS. CO. (U.S.P. 1,692,774, 20.11.28. Appl., 18.6.25).—For paper of increased strength, straw is disintegrated by heating with a dilute solution of

sodium sulphate, and after adding a quantity of pulp suitable for ordinary paper the mixture is matted into sheets while still wet with residual sulphate liquor. F. R. ENNOS.

Newsprint paper. W. H. MILLSPAUGH (B.P. 288,212, 4.4.28. U.S., 4.4.27).—Newsprint paper of increased strength is made from ordinary stock by simultaneously forming two wet sheets of half the usual thickness, combining them by pressing, and drying. F. R. ENNOS.

Production of stencil sheets for use in duplicating. A. DE WAELE (B.P. 303,631, 6.12.27).—Yoshino paper is coated with a mixture of a cellulose derivative, particularly nitrocellulose, and a hydroxy-fatty acid, *e.g.*, ricinoleic acid. F. R. ENNOS.

De-inking process. W. LEWIS (U.S.P. 1,696,639, 25.12.28. Appl., 6.9.27).—Newspaper etc. saturated with an alkaline solution is reduced to pulp and subjected to pressure between surfaces impervious to the pulp but pervious to the ink and solution. L. A. COLES.

Mechanism for use in treatment ["filling"] of fabrics. N. ISHERWOOD (B.P. 304,433, 7.12.27).

Machine for making sheet cellulose from aqueous cellulose solutions. C. RUZICKA (B.P. 304,374, 21.10.27).

[Cutting non-fraying tape from] fabrics made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 298,207, 5.10.28. U.S., 5.10.27. Addn. to B.P. 293,858 and 297,712).

Method and machine for manufacture of pulp board. J. J. HINDE (B.P. 304,171, 14.7.27).

Treatment of roads (B.P. 303,433).—See IX.
Moulded products (B.P. 284,232).—See XIII.
Deposition of viscose and oxycellulose (B.P. 303,765).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dye sensitisers and bleaching. MUDROVČIČ.—See XXI.

PATENTS.

Treatment of fabrics consisting of or containing acetyl silk. BLEACHERS' ASSOC., LTD., W. KERSHAW, F. L. BARRETT, and R. GAUNT (B.P. 303,286, 21.1.28).—Lustre pattern effects are obtained by completely delustring cellulose acetate silk fabric and then embossing it in the usual manner at about 150°, the fabric being then preferably wetted and lightly calendered whereby the embossing marks are removed. The process may be combined with those described in B.P. 301,567—8 (B., 1929, 127). A. J. HALL.

Imparting a linen-like effect to cotton. H. EGLI, Assr. to A.-G. SEERJET (U.S.P. 1,690,572, 6.11.28. Appl., 2.1.26).—Cotton yarns and webs formed of threads the fineness of which does not exceed English yarn no. 60 are subjected at 0–5° to the action of sulphuric acid (*d* 1.51–1.54) for not less than 4 min., and after washing are treated while under tension with caustic alkali (*d* 1.12) at ordinary temperatures. F. R. ENNOS.

Plant for cooling liquids particularly for use in mercerising fabrics. S. S. HAMMERSLEY and J. A.

BOLTON (B.P. 302,004, 12.10.27).—A liquid such as a caustic mercerising bath is withdrawn from the treatment or mangle tank, passed through a strainer and pump, and cooled in a separate vessel or "evaporator," then returned to the treatment tank. The evaporator is kept cool by the evaporation of a refrigerant in coils, and the compressor for the refrigerant is preferably thermostatically controlled. The contents of the evaporator may be withdrawn by the circulating pump into a storage tank to permit of cleaning the cooling coils by flushing. B. M. VENABLES.

Dyeing machines. J. SYKES & SONS, LTD., and E. P. SYKES (B.P. 302,486, 1.12.27).

Dyeing and like machines. C. S. BEDFORD (B.P. 304,088, 2.5.28. Addn. to B.P. 290,138).

Dye becks and like apparatus for liquid treatment of textile yarns or fabrics. T. PARKINSON (B.P. 304,460, 13.1.26).

Dye sticks and the like. C. CALLEBAUT and J. DE BLICQUY (B.P. 302,857, 10.5.28).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Reaction mechanism in the sulphuric acid contact process and the action of promoters. B. NEUMANN (Z. Elektrochem., 1929, 35, 42—51; cf. B., 1928, 891; 1929, 51).—In continuation of recent work (*loc. cit.*) on the use of various metallic oxide catalysts for the sulphuric acid contact process, the view is now developed that the behaviour of the catalyst depends on the alternate reduction of the oxide and oxidation of the reduction product, with simultaneous liberation of sulphur trioxide. With iron oxide, the following series of reactions is assumed to occur: $\text{SO}_2 + 3\text{Fe}_2\text{O}_3 = 2\text{Fe}_3\text{O}_4 + \text{SO}_3$; $2\text{Fe}_3\text{O}_4 + 4\text{SO}_2 + 2\text{O}_2 = 2\text{FeSO}_4 + 2\text{Fe}_2\text{O}_3 + 2\text{SO}_3$; $2\text{FeSO}_4 = \text{Fe}_2\text{O}_3 + \text{SO}_3 + \text{SO}_2$; SO_2 (nascent) + $\frac{1}{2}\text{O}_2 = \text{SO}_3$. At temperatures above 605° the ferrous sulphate decomposes as follows: $2\text{FeSO}_4 = \frac{1}{3}\text{Fe}_2(\text{SO}_4)_3 + \frac{2}{3}\text{Fe}_2\text{O}_3 + \text{SO}_2$; $\frac{1}{3}\text{Fe}_2(\text{SO}_4)_3 = \frac{1}{3}\text{Fe}_2\text{O}_3 + \text{SO}_3$. The fact that small quantities of ferrous sulphate are found in the used contact mass when iron oxide is employed as the catalyst is cited as evidence in favour of the above view. The easy formation and decomposition of the ferrous sulphate is the most important factor of the catalysis, and represents the real "intermediate reaction" of the process. The mechanism of the reaction with vanadic acid as catalyst is similar. The intermediate compound is probably vanadyl sulphate, traces of which are found in the used contact mass. Similarly, with titanate oxide, titanate sulphate and titanate sulphate are assumed to be formed. The action of promoters in mixed catalysts is discussed, with special reference to the cases of silver and vanadic acid and of tin oxide and chromic oxide. It is considered that, with mixed catalysts, the sulphuric acid contact process consists of a continuous alternating reduction and oxidation; with an inefficient contact substance the reaction velocity of either the oxidation or the reduction is slow, and the action of a promoter is to accelerate the velocity of the slow reaction. L. L. BIRCUMSHAW.

Extraction of phosphorus pentoxide. U. ORLANDI

(Notiz. Chim.-ind., 1928, 3, 343—344; Chem. Zentr., 1928, ii, 1024).—Phosphorite, silica, and a flux (alkali sulphate etc.) are treated with oleum containing a high proportion of sulphur trioxide until reaction is complete, the temperature rising to 300°; phosphorus pentoxide and metaphosphoric acid are then sublimed in an electric furnace at 600°, whilst removal of sulphur di- and tri-oxides is effected at 1000°.

A. A. ELDRIDGE.

Removal of carbon dioxide from the atmosphere by electrolytic transport. E. MÜLLER, H. MARKERT, and F. HEINRICH (Z. Elektrochem., 1929, 35, 3—13).—The possibilities of regenerating electrolytically caustic alkali solutions which have been used for the absorption of carbon dioxide from the air are investigated, and several difficulties encountered are discussed. Electrolysis of an alkali carbonate solution, using a divided cell with a porous diaphragm, yields a solution of alkali hydroxide in the cathode compartment, and of alkali hydrogen carbonate in the anode compartment. The reactions occurring on electrolysis when (a) both anolyte and catholyte are carbonate solutions, (b) both are bicarbonate solutions, and (c) the anolyte is bicarbonate and the catholyte carbonate, are examined. In (a) bicarbonate is first formed at the anode; this then decomposes with liberation of carbon dioxide, so that eventually a point is reached where as much bicarbonate is formed as is decomposed in unit time. If at this point as much carbonate is added to the anolyte as is converted into bicarbonate, while at the same time the OH' in the catholyte is neutralised by the introduction of carbon dioxide, then the equilibrium condition represented by the following equation will be realised with a current consumption of 4 Faradays: $[\text{3CO}_2 + 2\text{H}_2\text{O}]$ (catholyte) + K_2CO_3 (anolyte) = $[\text{K}_2\text{CO}_3 + 2\text{H}_2]$ (catholyte) + $[\text{2CO}_2 + \text{O}_2]$ (anolyte). This method has been used for the removal of carbon dioxide from a continuous air stream, and details are given of a small-scale apparatus for this purpose. A current yield of over 90% may be obtained from a catholyte 10.5N with respect to potassium carbonate and 1.06N to free potassium hydroxide, and an anolyte 2.2N with respect to potassium bicarbonate and 0.2N to potassium carbonate, since tests have shown that these solutions fulfil the equilibrium conditions determined by the above equation.

L. L. BIRCUMSHAW.

See also A., Feb., 140, **Transformation of chromates into dichromates** (CARRIÈRE and CASTEL). 153, **Activity of metallised silica gels** (REYERSON and SWEARINGEN). 154, **Electrolysis of sodium sulphide solutions** (FETZER). 155, **Formation of ozone by cathode rays** (MARSHALL). 156, **Preparation of cuprous sulphate** (DRUCE and FOWLES). 159, **Synthesis of hypophosphoric acid** (MIŁOBEDZKI and WALCZYŃSKA). 162, **Titration of ammonium salts** (KAPPELMEIER). 163, **Volumetric determination of arsine** (KUBINA). 164, **Determination of lead by means of 8-hydroxyquinoline** (MARSSON and HAASE). 165, **Analysis of tungsten compounds** (SPITZIN and KASCHTANOV).

Indicator for absorption systems. MACKERT.—See I. Vanadium and molybdenum compounds

in clays. PALMER.—See VIII. Ammonia production by soil fungi. THAKUR and NORRIS.—See XVI.

PATENTS.

Manufacture of sulphuric acid [oleum etc.]. S. ROBSON, B. LAMBERT, and NAT. PROCESSES, LTD. (B.P. 303,459, 3.10.27).—Sulphurous gases are passed through electrostatic dust depositors and/or filters and then, in admixture with air or oxygen, over the catalytic material described in B.P. 301,853 (B., 1929, 94).

L. A. COLES.

Manufacture of sulphuric acid. R. VETTERLEIN (F.P. 628,923, 28.12.26. Ger., 19.7.26).—In the manufacture of sulphuric acid from sulphur dioxide and nitrosulphuric acid, one or more empty chambers or towers are interposed between the end of the reaction chambers and the absorption chambers. A chamber may also be built above the filling material in the last oxidising tower.

A. R. POWELL.

Manufacture of hyposulphurous acid and its salts and derivatives. H. KAUTSKY and A. PFANNENSTIEL (G.P. 444,063, 12.10.24).—An aqueous solution of sulphur dioxide, alkali sulphites or bisulphites, or of aldehyde or ketone derivatives thereof is treated with calcium silicide in the presence of suitable salts.

A. R. POWELL.

Recovery of hydrochloric acid from waste liquors of the fuller's earth industry. E. MAAG (G.P. 451,531, 5.8.26. Addn. to G.P. 449,993; B., 1929, 54).—The liquor in either or both stages of the evaporation is sprayed on to a layer of hot coke which is continually replenished as it becomes encrusted with separated salts. The used coke is withdrawn from the bottom of the container and is used for heating further quantities of liquor.

A. R. POWELL.

Manufacture of hydrogen peroxide. P. ASKENASY (F.P. 628,360 and 628,441, 1.2. and 3.2.27. Ger., [A] 2.2. and [B] 4.2.26).—(A) Barium peroxide is treated for 30 sec. with carbon dioxide under at least 20 atm. pressure. (B) An aqueous suspension of impure barium peroxide is treated with carbon dioxide. In both cases a 4% solution of hydrogen peroxide may be obtained and the barium carbonate may be reconverted into peroxide for use again.

A. R. POWELL.

Purification of salt and recovery of by-products. V. YNGVE (U.S.P. 1,697,336, 1.1.29. Appl., 9.4.25).—Brine is treated with excess of sodium hydroxide, and after removal of the precipitated hydroxides is concentrated, the salt so produced being washed with neutral brine; the brine mother-liquor after removal of sodium sulphate is used for treating further brine.

W. G. CAREY.

Production of sodium carbonate sulphate. A. W. GAUGER and H. H. STORCH, ASSRS. to BURNHAM CHEM. CO. (U.S.P. 1,689,526, 30.10.28. Appl., 9.1.26).—A Californian brine containing potassium, sodium, sulphate, and carbonate ions is evaporated by the sun in a deep pond, so that its temperature does not rise above 20–25° and sodium chloride only is deposited. When saturated with sodium potassium sulphate, the brine is run into a shallow pond, so that its temperature rises to 30°, and sodium carbonate sulphate is deposited.

The mother-liquor may be run off from the warmer pond at the same rate as the brine is introduced, little evaporation taking place as a crust of sodium chloride is formed.

F. G. CLARKE.

Manufacture of sodium magnesium carbonate. H. RUBINSTEIN (U.S.P. 1,684,782, 18.9.28. Appl., 23.9.26).—Finely-divided natural or artificial magnesite (1 mol.) is suspended in a solution of sodium chloride (0.2 mol.) at 60–70°, and sodium carbonate (2 mols.) is added slowly over a period of several hours, whereby a crystalline precipitate of $\text{MgCO}_3 \cdot \text{Na}_2\text{CO}_3$ is obtained.

A. R. POWELL.

Manufacture of Javelle water [sodium hypochlorite solution]. H. D. LEVINE (U.S.P. 1,694,711, 11.12.28. Appl., 7.5.25).—Sodium hydroxide solution is cooled rapidly to about 0°, and is maintained at this temperature during treatment with chlorine.

L. A. COLES.

Production of ammonium phosphates. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 303,455, 4.7.27).—Crude phosphates are dissolved in an acid which forms a readily soluble calcium salt, e.g., hydrochloric or nitric acid, and, after filtration, the solution is partially neutralised by the addition of calcium phosphate to precipitate calcium mono- or di-hydrogen phosphate, which, after separation from the liquor, is treated with ammonia, with ammonia and carbon dioxide, or with ammonium salts of acids forming insoluble calcium salts, to yield ammonium phosphates.

L. A. COLES.

Production of cyanamides of the alkaline-earth metals and magnesium. N. CARO and A. R. FRANK, ASSECS. of STICKSTOFFWERKE G.M.B.H. (B.P. 281,611, 30.9.27. Ger., 2.12.26. Addn. to B.P. 279,812; B., 1929, 53).—Alkaline-earth or magnesium oxides, or compounds yielding these on heating, are heated above 400°, preferably at 650–850°, in the presence of gas mixtures which yield hydrogen cyanide, e.g., mixtures of ammonia and carbon monoxide, at the ordinary or raised pressure. Water and catalyst poisons, e.g., sulphur compounds, are also added, and the reaction is effected in apparatus constructed of materials which do not accelerate the decomposition of the reacting gases, e.g., of ceramic material or zinc.

L. A. COLES.

Manufacture of calcium carbide and phosphorus. A. CHAGNON (F.P. 628,793, 23.4.26).—The charge is heated in a high-frequency induction furnace which is so constructed that it can be continuously replenished and the gases evolved in the reduction continuously removed.

A. R. POWELL.

Separation of lead from solutions [containing, e.g., ferrous chloride and lead chloride]. S. I. LEVY and G. W. GRAY (B.P. 304,054, 10.2.28).—Lead is recovered from solutions obtained, e.g., by treating iron pyrites residues with hydrochloric acid, by electrolysis using an iron anode and an *E.M.F.* less than that required to decompose the ferrous chloride, or by the addition of an iron-copper couple.

L. A. COLES.

Manufacture of copper sulphate [briquettes]. HUNGÁRIA MŰTRÁGYA, KÉNSAV ÉS VEGYI IPAR RÉSZVÉNYTÁRSASÁG, and L. DEUTSCH (B.P. 304,052, 10.2.28).—Copper sulphate crystals 0.25–5.0 mm. in length

are formed into briquettes by the application of low pressure in moulds in the presence of adhering mother-liquor or added copper sulphate solution, with or without the addition of binding agents, *e.g.*, sodium sulphate, and the briquettes are removed and dried.

L. A. COLES.

Decomposition of aluminous minerals with nitric acid. METALLBANK U. METALLURGISCHE GES. A.-G., Assees. of C. FRH. VON GIRSEWALD and R. KAISER (G.P. 451,117, 15.11.25).—Minerals containing alumina are heated with an excess of nitric acid, and the resulting solution is heated under pressure with an excess of the powdered mineral to obtain a neutral solution free from iron salts and from silica.

A. R. POWELL.

Dissolving and purifying materials containing alumina, applicable also to other ores. M. BUCHNER (B.P. 282,772, 23.12.27. Ger., 24.12.26).—Clay, bauxite, etc. is extracted with nitric acid in a vessel constructed of an alloy of iron, nickel, and chromium, and preferably containing tungsten or a similar metal in addition, the bauxite etc. being present in excess. The extraction is started at 80–100°, and the pressure and temperature are raised gradually until the latter reaches 150–160°.

L. A. COLES.

Manufacture of beryllium sulphate. METALLBANK U. METALLURG. GES. A.-G., Assees. of C. FRH. VON GIRSEWALD and W. SIECKE (G.P. 451,346, 21.5.26).—A mixture of finely-divided beryl and lime or magnesia equivalent to half the weight of beryllia present is sintered in a rotating furnace and the powdered product is digested with sulphuric acid.

A. R. POWELL.

Manufacture of desiccating material. H. WADE. From E. THOMAS (B.P. 303,263, 9.12.27).—The material comprises magnesium perchlorate deposited upon a carrier, *e.g.*, upon 3 pts. of barium perchlorate per 1 pt. of magnesium perchlorate, the mixture being dried at 180–300° under the ordinary or reduced pressure to yield a granular product.

L. A. COLES.

Preparation of titanium and like compounds [oxides and hydroxides]. J. BLUMENFELD (B.P. 275,672, 9.8.27. U.S., 9.8.26).—Solutions obtained by the decomposition of ilmenite etc. with sulphuric acid, containing, *e.g.*, 200 g. TiO_2 and 500 g. H_2SO_4 (free and combined) per litre, are introduced at 100–110° into about one quarter of their volume of boiling water at a uniform rate during about 10 min., the mixture being well stirred during the addition and then boiled for 2–3 hrs. to precipitate colloidal titanium hydroxide. The product may be dispersed as described in B.P. 247,296 (B., 1926, 373). Compounds of the rare-earth metals may be similarly prepared.

L. A. COLES.

Manufacture of white titanate acid [oxide]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,468, 4.10.27).—In the recovery of titanate oxide from ilmenite etc. the hydrolysis is effected in the presence of a weak reducing agent, *e.g.*, sodium sulphite or sulphurous acid, which does not convert titanium from the quadrivalent into the trivalent form, in quantity sufficient to reduce trivalent iron and other oxidising agents, including dissolved oxygen, and the product is washed with a dilute solution of the reducing

agent. A portion of the weak reducing agent may be replaced by a stronger agent, *e.g.*, iron. L. A. COLES.

Mining of boron compounds. H. BLUMENBERG, JUN. (U.S.P. 1,696,075, 18.12.28. Appl., 24.10.27).—Deposits of kernite ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 4\text{H}_2\text{O}$) are treated *in situ* with a hot solution of sodium carbonate under pressure whereby the very soluble sodium metaborate is formed. The resulting liquors are treated with carbon dioxide and allowed to crystallise to recover borax, the mother-liquor containing sodium carbonate being heated and returned to the process.

A. R. POWELL.

Decomposition of salts of complex hydrofluoric acids [fluosilicates]. A. F. MEYERHOFER (B.P. 303,760, 28.7.27).—Fluosilicates are decomposed into metal fluorides and silicon tetrafluoride by heating in thin layers under reduced pressure at a temperature below the sintering point so that the material remains as a loose powder, or sufficiently high to melt it completely. The decomposition may be promoted by the introduction of gases, *e.g.*, air, steam, oxygen, flue gases, etc.; by the addition of organic material etc. which evolves gases on heating; or by admixture of the fluosilicates with fluorspar or other silicates prior to heating. The decomposition chamber is preferably constructed of alloys containing iron, nickel, and chromium, and having a low carbon content.

L. A. COLES.

Concentration of sulphur. E. H. NUTTER and J. W. LITTLEFORD, Assrs. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,697,402, 1.1.29. Appl., 20.4.28).—Material containing sulphur is heated to cause particles of sulphur to unite and is ground to a size suitable for froth flotation; an aqueous pulp of the material is then agitated and the sulphur-bearing froth is separated.

W. G. CAREY.

Sulphur-silica composition. W. H. KOBÉ, Assr. to FLEURON, INC. (U.S.P. 1,693,714, 4.12.28. Appl., 26.11.26).—Equal parts by wt. of sulphur and silica are mixed, the silica being comminuted so that 43% passes 320-mesh and 7.2% is held on 80-mesh.

W. G. CAREY.

Preparation of "calcicated" iodine. W. L. CHANDLER (U.S.P. 1,689,775, 30.10.28. Appl., 1.5.24).—A clear or slightly cloudy solution of calcium hydroxide is agitated with excess of sublimed iodine. The solution gives an active form of iodine when acid is added.

F. G. CLARKE.

Manufacture of cyanogen chloride. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,469, 4.10.27).—The product is obtained by the action at below 8° of a (sodium) cyanide solution on chlorine hydrate, which is prepared by passing chlorine into water at 0°. The water may contain a dissolved electrolyte, *e.g.*, sodium chloride, to lower the f.p., or may be mixed with a non-reacting organic liquid, *e.g.*, carbon tetrachloride. The resulting solution may be used direct, or the cyanogen chloride may be recovered by distillation.

L. A. COLES.

Production of large crystals. P. SEIDLER (U.S.P. 1,697,543, 1.1.29. Appl., 23.11.26. Ger., 30.11.25).—Vegetable substances are added to salt solutions, which are then concentrated by heat and allowed to cool slowly,

the crystals formed being removed from the mother-liquor and dried by centrifugal action.

H. ROYAL-DAWSON.

Contact sulphuric acid process. SELDEN Co., Assees. of A. O. JAEGER (B.P. 296,048, 11.10.27. U.S., 24.8.27).—See U.S.P. 1,675,309; B., 1928, 783.

Production of phosphorus pentoxide and phosphoric acid. G. PISTOR, H. LANG, and R. SUCHY, Assrs. to I. G. FARBENIND A.-G. (U.S.P. 1,700,708, 29.1.29. Appl., 11.10.26. Ger., 22.10.25).—See B.P. 260,225; B., 1927, 601.

Recovering barium as barium carbonate from barium silicates. C. DEGUIDE (U.S.P. 1,697,722, 1.1.29. Appl., 25.3.26. Fr., 3.4.25).—See G.P. 429,169; B., 1926, 788.

Manufacture of calcium nitrate. K. BLUMRICH, Assr. to I. G. FARBENIND A.-G. (U.S.P. 1,700,116, 29.1.29. Appl., 7.11.25. Ger., 11.11.24).—See B.P. 242,990; B., 1926, 926.

Recovery of chromium [as chromate] from chromiferous waste materials [chrome leather scrap etc.]. A. TREUSCH and R. WÜRTENBERGER, Assrs. to J. MAYER & SOHN (U.S.P. 1,700,657, 29.1.29. Appl., 1.6.25. Ger., 16.6.24).—See B.P. 235,548; B., 1925, 714.

Hydrocarbons and ammonia (B.P. 302,620).—See II. **Removal of magnesium oxide from zinc sulphate electrolytes (F.P. 629,216).** **Electrolysis of ferrous chloride (B.P. 304,053).**—See XI.

VIII.—GLASS; CERAMICS.

[Heat] insulation of roofs of glass furnaces. R. D. PIKE (J. Amer. Ceram. Soc., 1929, 12, 56—61).—A description is given of a method of test as applied to a portion of the roof of a tank furnace while in operation in order to show whether or not insulation is safe. The effect of the application of insulation on the life of the roof bricks is considered.

J. A. SUGDEN.

Production of colour in glass and in gems by X-rays and radium rays. M. C. REINHARD and B. F. SCHREINER (J. Physical Chem., 1928, 32, 1886—1887).—Intense X-rays and the β - and γ -radiations from radon produce no change of colour in topaz, aquamarine, amethyst, garnet, and white and brown diamonds. The white diamond gives a blue fluorescence when exposed to the radiations from radon, but none of the other gems showed fluorescence. Natural quartz is unchanged, but fused quartz is coloured brown in an irregular manner by exposure to X-rays.

L. S. THEOBALD.

Fractional crystallisation of technically pure glasses. H. JEBSEN-MARWEDEL (Naturwiss., 1929, 17, 84).—Paragenesis of β -wollastonite and cristobalite is found to occur in technical soda-lime glass.

W. E. DOWNEY.

Vanadium and molybdenum compounds in clays. L. A. PALMER (J. Amer. Ceram. Soc., 1929, 12, 37—47).—Development of green efflorescence in certain face bricks is due to the presence of soluble alkali

vanadates. Only a small amount of molybdenum is present in the efflorescence. Eight kinds of clays examined contained insoluble vanadium and molybdenum compounds which are rendered soluble during firing. Efflorescence is prevented by firing in a reducing atmosphere or by hard firing. The effect of such treatment on shade or colour may be objectionable, and a chemical treatment of the clay is more economical. When added to the extent of 1%, calcium fluoride or sodium fluosilicate is both effective and cheap.

J. A. SUGDEN.

Plasticity and related properties of clays. D. R. IRVING and W. F. DIETRICH (J. Amer. Ceram. Soc., 1929, 12, 14—29).—Applying the Talwalkar-Parmelee method of measuring plasticity (B., 1928, 193), the effect of water content, ageing, and addition of non-plastic materials on a number of clays has been studied. The plastic torsional strength decreases and deformation below rupture increases with increase of water content. Ageing decreases the strength but increases the deformability. The addition of up to 50% of finely-ground quartz increases the strength and decreases the deformability, but beyond 50% the strength rapidly decreases, with little change in deformability. The strength increases with fineness of the non-plastic material. The plasticity test distinguishes between non-plastic clays and clays the plasticity of which has been reduced by the addition of non-plastic materials, and serves also to differentiate various types of clays. There appears to be some connexion between plastic strength and vitrification temperature. No correlation was found between the adsorption of malachite-green and methylene-blue by various clays, nor between dye absorption and plasticity, drying, or firing shrinkages.

J. A. SUGDEN.

Rate of oxidation of porcelain and ball clays. H. M. KRANER and E. H. FRITZ (J. Amer. Ceram. Soc., 1929, 12, 1—13).—Small dried test bars were heated in a laboratory tube furnace under conditions of temperature rise, atmosphere (4% oxygen-nitrogen mixture), and rate of atmospheric change which prevail in a kiln. The carbon dioxide and water evolved were absorbed by phosphorus pentoxide and soda-lime. The porcelain body contained (approx.) 25% each of ball clay, china clay, flint, and felspar. It was found that oxidation proceeds more rapidly at low temperatures than has been generally considered. Advantage may be taken of this fact to attain more economical firing. A porcelain or ball clay-flint body may be completely oxidised at 600—650° and a close-grained ball clay may be 90% oxidised at 650° and completely oxidised at 700—800°. The rate of oxidation appears to be dependent almost entirely on temperature, and the time factor is involved only in thick pieces. Oxidation seems to proceed more rapidly in a dry atmosphere. The carbon content is not necessarily a criterion of the probable rate of oxidation of a clay.

J. A. SUGDEN.

Influence of hydrogen-ion concentration and electrolytes on the turbidity, sensitivity, and settling rates of certain Pleistocene clays. H. C. GRAHAM and J. N. PEARCE (Proc. Iowa Acad. Sci., 1927, 34, 217—218).—The amount of clay which can be held in aqueous suspension depends on the stratum from which the clay was obtained, on the hydrogen-ion con-

centration, and on the concentration of the electrolyte present in the suspension. Generally the turbidity is maximal at p_H 10.5. **CHEMICAL ABSTRACTS.**

Cyanite as found in Western North Carolina. A. H. FESSLER and W. J. McCAUGHEY (J. Amer. Ceram. Soc., 1929, 12, 32—36).—A brief description is given of the deposits, formations, geology, and economic values of the mineral. J. A. SUGDEN.

Terra cotta kiln slab investigation. R. L. CLARE, G. H. BROWN, and F. B. ALLEN (J. Amer. Ceram. Soc., 1929, 12, 62—68).—The constitution of a good kiln slab was determined under actual service conditions and correlated with nature and size of grog, thermal expansion, and initial cross-breaking strength. Best results were obtained with porous and vitreous grogs with particles varying widely in size. Highly siliceous grog causes premature failure. Mechanical strength is an indication of probable life. Insufficient data is available to show the effect of a number of firings on the strength. J. A. SUGDEN.

Prevention of disintegration of blast-furnace linings. R. P. HEUER (J. Amer. Ceram. Soc., 1929, 12, 30—31; cf. Mitra and Silverman, B., 1928, 570).—Exception is taken to the statement that ferrosilicic oxide is immune to reduction and therefore cannot be catalytically active in carbon deposition. The author records the deposition of 1.1% of carbon on a sample of naturally occurring magnetite, and, further, has succeeded in producing the eutectic of the system $\text{FeO-Al}_2\text{O}_3\cdot 2\text{SiO}_2$ (at about 1100°) in blast-furnace bricks. This, it is considered, is a much safer constituent than any of the free oxides, and laboratory tests show no deposition of carbon even after prolonged exposure. J. A. SUGDEN.

See also A., Feb., 163, **Decomposition of silicates by strontium salts** (KAVINA).

PATENTS.

Enamel-coated article. J. A. ANDERSON, Assr. to CENTRAL OIL & GAS STOVE Co. (U.S.P. 1,693,130, 27.11.28. Appl., 5.12.23).—A refractory enamel for coating iron and steel articles subjected to high temperatures comprises 60.96% SiO_2 , 13.06% B_2O_3 , 12.23% Na_2O , 6.47% Al_2O_3 , 5.81% CaO , 0.86% CoO , and 0.43% NiO . This enamel is stated to resist cracking or peeling at 1000°. A. R. POWELL.

Kiln. B. H. GREENE (U.S.P. 1,694,650, 11.12.28. Appl., 30.12.27).—A kiln for firing large pottery or earthenware articles is provided with a series of refractory shelves near the side walls but spaced therefrom at their rear edges. A. R. POWELL.

[Endless conveyor for] lehrs, annealing furnaces, and the like. CANNING TOWN GLASS WORKS, LTD., and J. J. PARNABY (B.P. 304,381, 18.4.28).

Treatment of liquids (U.S.P. 1,691,458). **Flat glass bulbs for instruments** (B.P. 302,034).—See I.

IX.—BUILDING MATERIALS.

Slag wool. GUTTMANN.—See X.

PATENTS.

Cement. C. P. BACCOVICH (U.S.P. 1,696,899, 1.1.29. Appl., 16.2.24. Japan, 28.3.23).—Acidic rocks con-

taining substantial alkaline contents are crushed, dried at 200—500°, pulverised to a condition finer than commercial Portland cement, and intimately mixed with Portland cement containing silica in excess of alumina. W. G. CAREY.

Production of hydraulic cement. N. V. S. KNIBBS (B.P. 303,639, 16.12.27).—A finely-divided mixture of highly aluminous material and caustic lime is treated under pressure with steam, and the product is heated in a shaft or vertical kiln at 1000—1400°, fusion being avoided. W. G. CAREY.

Waterproofing of lime and cement mortars, wood, etc. SYND. INTERNAT. DU CIMENTOILS (F.P. 624,296, 5.3.26).—The waterproofing material comprises a mixture of rosin dissolved in linseed oil which has been boiled with litharge, resin oil, masout, and petroleum containing dissolved paraffin wax and coal tar. Linseed oil is omitted from mixtures which are to be used for treating cement and mortar. A. R. POWELL.

Manufacture of porous material. B. GRANVILLE (U.S.P. 1,691,280, 13.11.28. Appl., 26.7.26).—Porous artificial stone is made by casting a wet cement mixture containing gas-producing substances into a closed mould, applying suction to the mould so as to cause the mass to swell and fill the mould, and maintaining the suction until the mass has set. A. R. POWELL.

Porous building material. E. C. BAYER (Austr. P. 106,712, 2.9.24. Denm., 21.1.24).—Mineral substances, such as natural or artificial clay mixtures, which do not set on mixing with water are mixed with burnt clay, sand, and a froth-forming substance. A. R. POWELL.

Treatment of the surface of roads with sulphite lixivium and a hygroscopic substance. R. KARLBERG (B.P. 303,433, 3.10.27).—Sulphite lixivium, from paper pulp mills, concentrated to 50%, and calcium chloride are applied to the road in mixture or separately. W. G. CAREY.

Artificial stone. R. ULLMANN (Austr. P. 107,589, 12.1.25).—A mixture of 3 pts. of magnesia with 2 pts. of a filling material containing at least 30% of colloidal silica is bonded with magnesium chloride solution and cast into suitable moulds. A. R. POWELL.

Glazing of artificial stone surfaces. MINERAL A.-G. (Swiss P. 121,764, 9.3.26).—The surface of cement, concrete, mortar, or compressed shale articles is treated with phosphoric or boric acid or with a solution of a heavy-metal salt, then coated with a glaze of a coloured resinous material, e.g., phenol-formaldehyde or -furfuraldehyde resin. A. R. POWELL.

Impregnation of wood. SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS IND. (F.P. 592,827, 9.4.24).—The impregnating material comprises a mixture of a substance with purely antiseptic properties (e.g., creosote, cresol, or phenol), a substance of great diffusibility (e.g., petroleum, masout, shale oil, or resin), and a substance of high viscosity (e.g., coal tar or petroleum pitch). A. R. POWELL.

Impregnation of wood. SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (F.P. 598,267,

29.8.24).—Wood is impregnated with aqueous emulsions, suspensions, or solutions containing dyes or antiseptics, or mixtures of these, preferably with the addition of material with good moistening properties, *e.g.*, petroleum etc., and of material of low surface tension, *e.g.*, saponin, soap, sulphite-cellulose waste-liquor, etc.

L. A. COLES.

Impregnation of [fibrous] building materials. S. TCHAYEV (F.P. 624,776, 17.11.26).—Wood or compressed straw pulp is impregnated with solutions containing sodium dichromate, potassium ferrocyanide, sodium fluoride, nitrophenols, etc. to prevent the growth of fungi.

A. R. POWELL.

Colouring the surface of wood. M. SPARKS (U.S.P. 1,697,026, 1.1.29. Appl., 21.11.25).—Oregon pine or a similar wood is treated with a solution of ferrous sulphate and, after drying, with an ammoniacal solution of potassium dichromate.

A. R. POWELL.

Wood-preserving agent. R. JUMENTIER (F.P. 617,263, 20.10.25).—The material is made by heating together 750–900 pts. of tar, 70–120 pts. of resin, and 20–70 pts. of sulphur; 50 pts. of graphite may also be added if the wood is to be used in a damp place.

A. R. POWELL.

Regeneration of hardwood. RICHTER, GUTZWILLER & Co. (F.P. 627,227, 6.11.26).—Wood which has deteriorated by exposure to the weather is revived by treatment with a solution containing oxalic, citric, and tartaric acids, together with glycerol and alcohol.

A. R. POWELL.

Drying apparatus for wood. O. ENGELBREKTSON (B.P. 288,262, 15.3.28. Swed., 5.4.27).

Manufacture of surface coverings particularly for use on floors. Manufacture of floor coverings. ARMSTRONG CORK Co., Assees. of C. F. HUMPHREYS and J. C. MCCARTHY (B.P. 283,946—7, 26.9.27. U.S., 22.1.27).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Cottrell-Möller plant for the separation of dust from the flue gases of the Siemens-Martin furnace. S. SCHLEICHER and E. GÜBEL (Stahl u. Eisen, 1929, 49, 33–37).—The results of a series of tests on the removal of dust from flue gases using a Cottrell-Möller electrostatic plant indicate that the most efficient separation of dust is obtained with the highest possible potential and the lowest commercially practicable rate of flow of the gases through the plant. The addition of moisture has relatively little effect unless the amount present exceeds the saturation limit, when trouble is caused by condensation throughout the plant and in the flues. A purer gas is obtained by treatment at 110–120° than at higher temperatures.

A. R. POWELL.

Oxidation of iron in water by the action of oxygen and carbon dioxide. N. M. GAVRILOV, S. K. and P. K. MEL (J. Chem. Ind. Moscow, 1928, 5, 697–700).—The mechanism of corrosion of iron is considered to be as follows:—The iron, on contact with air and water, becomes covered with ferrous oxide,

which becomes oxidised to ferric oxide, which gradually becomes reduced by the iron, the metal becoming simultaneously oxidised. The action of water saturated with carbon dioxide on iron results in rapid dissolution of superficial oxides and slow dissolution of iron, with oxidation of ferrous hydrogen carbonate to ferric hydroxide. After a long period under ferrous hydrogen carbonate solution and subjection to the action of carbon dioxide, iron becomes quasi-passive; shaking detaches the film of oxides and activity is regained. When gases containing 5% of oxygen are blown on to iron covered with water, the metallic surface becomes covered with insoluble oxides and the concentration of iron in the solution is lowered. Corrosion is specially favoured if the gas contains not more than 5% of oxygen, and not less than 14–15% of carbon dioxide, the temperature being low. CHEMICAL ABSTRACTS.

Corroding action of solutions of various chlorides on cast iron and lead. B. K. PERSHKE and G. I. CHUFAROV (J. Chem. Ind. Moscow, 1928, 5, 523–528).—The corroding effect on cast iron of solutions of sodium, potassium, and magnesium chlorides decreases in that order; corrosion is maximal at about 1%, and is greatly increased by stirring or saturation with air. It is also increased by the action of light and by periodical drying, but the rate remains unchanged with time. A tar coating reduced corrosion to 20%. Lead is attacked by chloride solutions to about 11% of the extent of iron corrosion. CHEMICAL ABSTRACTS.

Corrosion of steel at high temperatures. L. LOSANA and G. REGGIANI (Notiz. Chim.-ind., 1928, 3, 346–352; Chem. Zentr., 1928, ii, 1030).—Tempering normally increases resistance to oxidation. A mixture of $10\text{CO}_2 + 4\text{CO} + 2\text{O}_2 + 48\text{N}_2 + \text{SO}_2$ (traces) + hydrocarbons (traces), saturated with water vapour at 16–20°, is more corrosive than air. Experiments were performed also with the more resistant nickel, chromium, and tungsten steels. A. A. ELDRIDGE.

Influence of tin on the quality [rolling properties] of Siemens-Martin mild steel. W. KELLER (Stahl u. Eisen, 1929, 49, 138–139).—The mechanical properties of steel with 0.06–0.8% C and 0–0.8% Sn have been systematically investigated. The metal can be rolled to thin sheets without cracking along the edges when the tin content does not exceed 0.6–0.7%, but when more than about 0.3% Sn is present the welding properties are deleteriously affected. The bending test is satisfactory up to 0.6% Sn, and with up to 0.8% Sn the metal forges well. Tin increases slightly the tensile strength and decreases the ductility of mild steel. With increasing carbon content the bad effect of tin becomes much more pronounced. A. R. POWELL.

Apparatus for the determination of silica in steel and iron by the chlorine method. P. DICKENS (Chem. Fabr., 1929, 51–52).—The apparatus comprises a gas-purification train for the chlorine, an electric tube furnace in which the metal is heated in a current of pure chlorine, and a vessel for collecting the volatilised chloride. Purification of the chlorine is effected by passing it through wash-bottles packed with glass beads moistened with concentrated sulphuric acid, then

through a tube packed with charcoal and heated at 1000° to convert any oxygen into carbon monoxide, which is without action on the metal, and finally through a drying tower, the lower part of which is filled with sulphuric acid and the upper part with calcium chloride and phosphorus pentoxide.

A. R. POWELL.

Ehn's cementation test [for steels]. R. WASMUTH and P. OBERHOFFER (Stahl u. Eisen, 1929, 49, 74—77).—Ehn's method of testing the quality of steel by examining the structure of the surface after cementation with wood charcoal and barium carbonate is shown to be unreliable except possibly in the case of mild steels. With high-carbon steels or alloy steels no difference can be discerned between "good" and "bad" metal (as determined by mechanical tests) after cementation or between steels containing varying proportions of oxygen. The effects of the method of deoxidation of the steel and of the presence of various non-metallic impurities on the progress of cementation are shown to be greater than the effect due to the actual oxygen content.

A. R. POWELL.

Tungsten steel for magnets. K. G. BRECHT, R. SCHERER, and H. HANEMANN (Stahl u. Eisen, 1929, 49, 41—42).—The most suitable heat treatment for a tungsten steel of the following composition has been investigated: 0.67% C, 0.26% Si, 0.25% Mn, 0.4% Cr, and 6.62% W. The best working properties were obtained by annealing at 650° and quenching in water, but the highest magnetic properties were obtained only after soaking for sufficient time at 820° to allow the cementite to go completely into solid solution and then quenching in water. The resulting steel had a fine hardenite structure, showing that tungsten steel is not sensitive to overheating. The fall in coercivity obtained by annealing above 820° was found to be due to a coarsening of the martensite grains and not to the presence of unchanged non-magnetic austenite. Micrographical examination showed that no change in the distribution of the two tungsten carbides present took place during the recommended heat treatment, and hence these compounds are without effect on the magnetic properties of the alloy.

A. R. POWELL.

Properties and uses of slag wool. A. GUTTMANN (Stahl u. Eisen, 1929, 49, 97—101).—For the preparation of the best quality slag wool, mobile slags with a high content of silica and a low content of calcium sulphide are most satisfactory. The physical properties of five such wools are tabulated. The heat-insulating power of slag wool depends on the fineness of its fibres, which should be such that about 93% of the apparent volume of the wool is air-space. Wool of this texture has a higher insulating power than most other heat insulators, and, except in the presence of excess moisture, does not accelerate the corrosion of iron.

A. R. POWELL.

Technical properties of metals melted in a vacuum furnace. W. ROHN (Z. Metallk., 1929, 21, 12—18).—A review of the development of vacuum melting of metals and alloys, showing the advantages to be gained by this procedure and the improvement effected in the properties of the metals. A. R. POWELL.

Technological significance of gases in metals.

E. H. SCHULZ (Z. Metallk., 1929, 21, 7—11).—A review showing the deleterious effects of gases in metal castings, with especial reference to the effect of oxygen on the properties of steels.

A. R. POWELL.

Effect of cold-working on chemical properties, especially of metals. G. TAMMANN (Z. Electrochem., 1929, 35, 21—28).—Previous work by the author and others on the subject is reviewed. In particular, the following properties are discussed: the change of potential of metals on cold-working, *e.g.*, the surfaces of copper, silver, lead, etc. become less noble when polished (Tammann and Wilson, A., 1928, 958); the relationship between the velocity of deposition of radium-*F* and the condition of the precipitating metal, the precipitation velocity being greater for the hard form of a metal than for the soft (*ibid.*, 963); the fact that mercury wets silver and its mixed crystals with gold, tin, and cadmium more quickly when they are in the hard condition than in the soft (Tammann and Dahl, A., 1925, ii, 502); the chemical changes at the slip planes and cleavage planes of crystals, with special reference to the fact that in mixed crystals of certain metals the limiting composition for resistance to chemical agents is modified in the direction of a higher proportion of the noble metal by cold-working (Tammann, A., 1924, ii, 25); the fact that the velocity of dissolution of iron in dilute acids is increased by cold-working; the increase in the dissolution velocity on the cleavage planes; the influence of cold-working on the velocity of reduction of silver bromide; the effect of plastic deformation on the velocity of adsorption of hydrogen by palladium, iron, and nickel; and the colour changes of certain mixed crystals (*e.g.*, gold-silver-copper alloys) caused by cold-rolling. The phenomenon of triboluminescence is also discussed. The effects of cold-working disappear completely on heating; with metals, when one third, and with salts (silicates and silver salts), when half, the absolute temperature of the m.p. has been reached.

L. L. BIRCHUMSHAW.

Study of annealing of metals by a new sensitive differential dilatometer. S. A. TOGINO and K. YAMAGUCHI (Bull. Inst. Phys. Chem. Res. Tokyo, 1929, 8, 34—43).—For soft metals having no transition point, the usual differential galvanometer is unsuitable by reason of its lack of sensibility and also the deformation produced by the spring. In a new form of dilatometer, the action of the spring is replaced by a component of gravity acting on a pendulum, which reduces the pressure on the test piece to the order of 0.3 g./mm.² This allows of determinations at temperatures approaching the m.p. of a metal. The instrument is extremely sensitive, as the change in length per cm. of the specimen can be magnified for reading to 24,000 times.

C. A. KING.

Studies on sherardising. M. KURODA (Bull. Inst. Phys. Chem. Res. Tokyo, 1929, 8, 12—18).—During the process of sherardising, the degree of oxidation is harmless either to the powder or to the product as the maximum depth of oxide is only a few mm. and the product has a bright silvery lustre. The process is conducted best at 370—380° for about 30 min., as at this temperature the physical properties of the metal

are not affected adversely. Slight variation in temperature has little influence on the thickness of the zinc coating.

C. A. KING.

Physico-chemical principles of soldering. L. ROSTOSKY and E. LÜDER (Z. Metallk., 1929, 21, 24—25).—The joining power of a solder may be due to any of the following three actions: (a) the solder and the object to be soldered form solid solutions with one another, e.g., brass solder and copper; (b) the solder forms inter-metallic compounds with the metal being soldered, e.g., tin-lead solder and copper; and (c) the solder dissolves the metal being soldered only in the liquid state, and, on cooling, the last-named metal crystallises out in the solder layer, e.g., tin solder and zinc. Photomicrographs of characteristic joints of these three types are given and discussed. Further, it is shown that the m.p. of a solder need not necessarily be lower than that of a metal to be soldered; thus copper may be used to solder two sheets of silver together, the soldering action being due to the formation of the readily-fusible eutectic at the junction of the silver and copper layers.

A. R. POWELL.

Preparation of synthetic bodies from metal powders. F. SAUERWALD [with J. HUNCZEK] (Z. Metallk., 1929, 21, 22).—In the preparation of articles from metal powders by high pressure, stronger articles are obtained if the pressure is applied or maintained while the article is heated than if the powder is compressed cold and the resulting body annealed at a temperature at which crystal growth commences.

A. R. POWELL.

Reciprocal displacement of metals. DAUVÉ (Ann. Chim. analyt., 1929, [ii], 11, 5—9).—An account is given of the principles and mechanism of the replacement of one metal from its salts by another metal.

A. R. POWELL.

Influence of hydrogen-ion concentration on recovery in simple flotation systems. A. M. GAUDIN (Min. and Met., 1929, 10, 19—20).—In the presence of xanthates complete recovery by flotation of chalcocite is effected within the p_H range 1—12, whereas complete recovery of pyrite is obtained within the range p_H 1—6.2 only; in more alkaline solutions no flotation of pyrite occurs at all. In the presence of isoamylamine the percentage recovery curve for chalcocite rises steeply to 100% between p_H 6 and 10, then falls abruptly; a similar curve is obtained for zinc blende. An 80% recovery of malachite from admixture with calcite is obtained in solutions of p_H 9—11 in the presence of amyl xanthate, but no sulphidising agent. Felspar is floated by sodium oleate over a very narrow p_H range with about 20% recovery, but if copper sulphate is added to the pulp recovery is complete in solutions of p_H 6.5—10. Quartz is activated by copper sulphate within the range p_H 6—11. A small amount of copper sulphate activates blende in an acid circuit, whereas a large amount activates it both in acid and alkaline circuits when xanthates are present. These results indicate that the surfaces of mineral particles adsorb hydrogen or hydroxyl ions under certain conditions, and that thereby their power of adsorbing the flotation agent is considerably modified.

A. R. POWELL.

Determination of the platinum metals in ores and concentrates. H. R. ADAM (J. Chem. Met. Min. Soc. S. Afr., 1928, 29, 106—114).—Attempts which have been made in South Africa to determine the total content of platinum metals in an ore or concentrate by collecting the metals in lead and cupelling the button with or without silver are examined. When silver is used the resulting bead is parted by heating with slightly diluted sulphuric acid until effervescence ceases; the insoluble residue is collected, washed, ignited, and weighed. The results are generally high owing to retention of silver, and, if heating with the acid is too prolonged, dissolution of part of the palladium may take place. When no silver is added the cupelled beads are heated at 1300° for 1 hr. in a small hole in a cupel placed in a small electric muffle. The beads still retain up to 10% of lead, especially if palladium is present. The use of gold instead of silver as a collector during cupellation followed by determination of the increase of weight of the gold leads to erratic results due to gold losses during cupellation and to retention of impurities by the bead.

A. R. POWELL.

Assay for platinoids in ores. J. WATSON (J. Chem. Met. Min. Soc. S. Afr., 1928, 29, 115—118; cf. preceding abstract).—Details are given for fluxing Transvaal platinum ores and for parting the silver bead obtained by cupelling the lead button produced in the pot-running; 90% sulphuric acid is recommended for the parting, and the washed residues from this operation are dried at 120—140° for weighing.

A. R. POWELL.

Age-hardening aluminium alloys. W. FRAENKEL and L. MARX (Z. Metallk., 1929, 21, 2—5).—Comparative age-hardening tests have been made on aluminium alloys containing (A) 4% Cu, (B) 4% Cu and 0.5% Mg, (C) 8% Zn, (D) 8% Zn and 0.5% Mg, and (E) 8% Zn and 0.2% Li. Maximum hardness after quenching from 500° was obtained by ageing (E) at 50°, (D) at 100°, and (B) and (C) at 150°, whereas (C) hardened slightly at 50° and became softer again at higher temperatures. The hardening effect in (E) decreased appreciably with increase of temperature, and that in (D) decreased with time at 150° after reaching a maximum in 20 hrs. The increase of hardness of all the alloys on ageing at the ordinary temperature after a short immersion (0.5—60 min.) in boiling water is shown in a series of graphs to be very irregular. As a result of these tests the authors consider that the simple precipitation theory of age-hardening does not explain all the facts, and suggest that, in some cases, the slow formation of a compound in the solid solution may be the cause of the increase of hardness observed.

A. R. POWELL.

Intercrystalline corrosion of nickel. W. KÖSTER (Z. Metallk., 1929, 21, 19—21).—When nickel is heated in an atmosphere containing sulphur dioxide the intercrystalline corrosion is shown to be due to the formation of the nickel-nickel sulphide eutectic which penetrates into the metal along the grain boundaries. The rate of penetration increases with the temperature up to 800°, and once a quantity of the sulphide is formed subsequent annealing in an atmosphere free from sulphur causes further penetration. The sulphide being hard and brittle

renders the whole of the metal brittle and leads to intercrystalline fracture when the metal is worked.

A. R. POWELL.

Electrolysis of nickel. B. BOGITCH (Compt. rend., 1929, 188, 328—329).—Nickel rods 20 mm. in diam. and containing over 99.92% Ni have been prepared from nickel alloys containing 10% of impurities (sulphur, arsenic, oxygen, phosphorus) by deposition from a solution of nickel chloride ($d\ 1.2$) at above 65° , with cathodes of thin nickel wire 0.5 mm. in diam. The impure electrolyte from the anode compartment is purified for further use by means of nickel hydroxide, which precipitates iron, arsenic, and most of the cobalt, any nickel entrained in the precipitate being used for the production of more hydroxide. The copper is found in the anode mud.

J. GRANT.

Construction and operation of Kathner [steel] normalising furnaces. C. P. MILLS (Proc. Eng. Soc. W. Pa., 1928, 44, 305—329).

Cementation of steels by special manganese alloys. J. COURNOT (Rev. Mét., 1928, 25, 669—670).—See B., 1928, 673.

See also A., Feb., 141, **Iron-zinc system** (OGAWA and MURAKAMI). **System tungsten-carbon** (BECKER). 160, **Reduction of tungsten by hydrogen** (MEIERSON). 161, **Spectroscopic analysis of alloys** (NEGRESKO). Wood's metal as cathode in electrolysis (PIETERS). 164, **Determination of lead by means of 8-hydroxyquinoline** (MARSSON and HAASE). 165, **Determination of molybdenum** (DOERNER; KASSLER). **Determination of tungsten** (SPITZIN). 166, **Determination of palladium** (OGBURN).

Blast furnace linings. HEUER.—See VIII. **Health hazards in chromium plating.** BLOOMFIELD and BLUM.—See XXIII.

PATENTS.

Preheating the charge in shaft furnaces. A. L. FEILD, Assr. to LINDE AIR PRODUCTS Co. (U.S.P. 1,695,953, 18.12.28. Appl., 25.4.24).—A current of preheated air deficient in oxygen is blown into the furnace above the smelting zone.

A. R. POWELL.

Annealing furnace. F. T. COPE, Assr. to ELECTRIC FURNACE Co. (U.S.P. 1,694,684, 11.12.28. Appl., 9.11.25).—The furnace comprises a muffle with electric resistance units adjacent to the side walls and a device whereby, when the articles are to be cooled in the furnace, two water-cooled screens may be moved up through the bottom of the furnace so as to cut off the heat from the resistors and prevent it reaching the goods.

A. R. POWELL.

[Annealing] furnace. C. H. CARPENTER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,950, 18.12.28. Appl., 17.7.26).—The furnace comprises an inclined muffle electrically heated at the top and at the lower end of the bottom and provided with a vertical discharge chute at the lower end. The hearth of the furnace consists of a metal plate with longitudinal corrugations down which the articles being treated slide, and means are provided to agitate this plate up and down to promote the sliding of the articles through the furnace.

A. R. POWELL.

Heating furnace. R. E. BISSELL, Assr. to THOMPSON PRODUCTS, INC. (U.S.P. 1,692,614, 20.11.28. Appl., 20.11.25).—An electric annealing muffle is heated by means of transverse resistances inside the top, and is provided with a series of parallel longitudinal tubes along the bottom and with means for inclining the furnace so that the articles just slide through at a pre-determined rate. The discharge chute to the quenching bath is provided with a trap-door to keep out vapours from the muffle.

A. R. POWELL.

Heat-treatment of metals. A. S. MACDONALD, Assr. to SNEAD & Co. (U.S.P. 1,695,433, 18.12.28. Appl., 11.10.24).—The metal in the form of rod or wire is heated by passing sufficient current through it to bring it to a temperature at which the desired grain-growth takes place, then quenched without switching off the current.

A. R. POWELL.

Heat-treatment of metals. K. FISCHER and M. HOLSBOER (Austral. P. 755, 27.2.26).—The metal to be heat-treated is passed continuously through a long furnace in the upper part of which gases are burnt to heat the walls of the furnace; the metal is heated by radiation from the walls or by means of a current of gas preheated by contact with the walls.

A. R. POWELL.

Retort [for heat-treatment of metals]. E. P. VAN STONE, Assr. to GEN. ALLOYS Co. (U.S.P. 1,689,808, 30.10.28. Appl., 7.4.27).—A rotating retort for heat-treating small metal articles comprises a horizontal, cylindrical body closed at one end with a flat circular disc and at the other end with a narrow tubular neck flared to engage a flange provided on the body of the retort.

A. R. POWELL.

Furnace for the reduction of iron. C. M. DAY and C. J. SCULLIN (U.S.P. 1,694,376, 11.12.28. Appl., 19.4.26).—A cupola for melting iron comprises a melting chamber above which is a combustion chamber in which the carbon monoxide is burnt and from which the waste gases pass to a recuperator above the combustion chamber. The preheated air from the recuperator passes downward through an annular space surrounding the walls of the melting chamber partly to the melting chamber and partly to the combustion chamber. Undue radiation of heat from the melting chamber is thus avoided, and the air used in the primary melting and in the secondary preheating of the charge in the combustion chamber is itself preheated.

A. R. POWELL.

Separation of gases [sulphur dioxide] or suspended matter from the discharge gas from metallurgical or other furnaces. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING (B.P. 302,386, 15.9.27).—The gases are passed into a pressure chamber partly filled with water and terminating in a long column through which a downward current of water is passed to the chamber to maintain the pressure. The soluble constituents, e.g., sulphur dioxide, are dissolved at the high pressure, and the valueless residual gas is removed still under pressure. The saturated solution is continuously pumped to regions of lower pressure, where the dissolved sulphur dioxide is removed, and the water is then returned to the circuit.

A. R. POWELL.

Manufacture of steel. H. MEYER (B.P. 281,621,

27.10.27. Ger., 6.12.26).—To reduce the tendency to blue fracture and brittleness by ageing, steel of the type used for boiler-plating, tubes, etc. is strained as by cold-rolling and then annealed at 700–950°. Hot-forging or rolling between 700° and the Ar3 point may be substituted for the cold-straining. [Stat. ref.]

C. A. KING.

Refining process [for iron]. I. G. FARBENIND. A.-G., Assecs. of A. CURS (G.P. 452,630, 2.4.26).—Ferric oxide obtained by the combustion of iron carbonyl forms an efficient refining agent for iron, rapidly removing the carbon, silicon, phosphorus, and sulphur, and yielding a product from which non-porous castings may be obtained directly.

A. R. POWELL.

Hardening iron, steel, and other ferromagnetic materials. EISEN- U. STAHLWERK HOESCH A.-G., and W. HEIDENHAIN (B.P. 282,750, 17.12.27. Ger., 28.12.26).—The article is heated in the upper part of an inclined furnace, being retained there by means of an electromagnet outside the furnace. When the temperature of the article exceeds that of the magnetic transformation point, the article commences to slide down the furnace and finally falls into a quenching bath. The inclination of the furnace and the temperature at its lower end are so arranged that the article reaches its correct hardening temperature just as it leaves the furnace.

A. R. POWELL.

Production of iron and iron alloys having a very low percentage of carbon. Production of dense iron and iron alloys directly from oxide ores. H. G. FLODIN and E. G. T. GUSTAFSSON, ASSRS. to H. G. E. CORNELIUS (U.S.P. 1,691,273—4, 13.11.28. Appl., [A, B] 22.6.25. Swed., [A] 11.5.25, [B] 26.5.25).—(A) Briquettes of finely-divided iron ore, lime and other fluxes, with or without the addition of an oxide or ore of an alloying element, *e.g.*, chromite for obtaining iron-chromium alloys, are smelted with carbonaceous material in an electric furnace to obtain iron or an iron alloy of low carbon content, and a slag with a high content of ferrous oxide and oxide of the alloying element. Addition of ferrosilicon in suitable quantities is then made to reduce to metal the oxide of the alloying element and most of the iron oxide in the slag; the resulting alloy or metal contains < 0.2% C. (B) A high-grade steel is made directly from titaniferous iron ore by smelting briquettes composed of the ore, lime, and carbon in an electric furnace and refining the iron under a basic slag in the same furnace to remove phosphorus and vanadium if present.

A. R. POWELL.

Reduction of ores to obtain ferro-alloys. A. E. WHITE. From S. G. ALLEN (B.P. 303,207, 30.8.26).—Ferromanganese and similar alloys are obtained by the blast-furnace smelting of suitable materials, using a higher ratio of ore to coke than in normal practice, and supplying oxygen to the blast and to a point or points above the mantle of the furnaces so that the temperature of the flue gases is maintained between 120° and 203°, preferably at 150°.

A. R. POWELL.

Manufacture of vanadium steel. A. L. FEILD, ASSR. to ELECTRO METALLURGICAL Co. (U.S.P. 1,697,759, 1.1.29. Appl., 12.12.25).—Vanadium oxide is added to

a bath of molten steel and maintained in contact therewith until the reducing agents normally present in the steel have caused the desired quantity of vanadium to enter the molten metal.

A. R. POWELL.

Chromium-manganese steel. M. BAEKE, Assec. of F. RAPATZ (Austr. P. 107,312, 17.10.23. Ger., 1.11.22).—A steel containing 0.4–1.0% C, 8–25% Cr, and 20–5% Mn is claimed. This retains an austenitic structure when quenched from 950–1200°.

A. R. POWELL.

Reducing or arresting rust on iron or steel surfaces. R. EBERHARD (B.P. 303,035, 26.7.27).—The metal surface is sprayed with a solution containing uric acid, urea, and formaldehyde, together with reduced acidic compounds of molybdenum, tungsten, or chromium produced by the action of powdered zinc, copper, lead, or aluminium on the uric acid solution.

A. R. POWELL.

Production of metal coatings on iron or steel or alloys thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,034, 25.6.27).—The article is provided with a thin coating of nickel, then plated with chromium, and heated at 1100–1300° in hydrogen or *in vacuo*, whereby the coatings diffuse into the surface layer rendering it highly resistant to corrosion. Similar results are obtained by plating first with chromium, then with cobalt, and heating as described.

A. R. POWELL.

Colouring of non-rusting steel rifle hammers. SIMON & Co. (G.P. 452,763, 16.4.26).—The articles are provided with a thin coating of iron by the metal-spray process, and this coating is treated by any of the known methods to obtain on it a black film of ferrosferic oxide. Such a film cannot be produced directly on non-rusting steels.

A. R. POWELL.

Manufacture of electrolytic iron free from brittleness. C. TSCHAEPPET (Swiss P. 121,383, 23.9.26).—Hollow cathodes heated by circulation of hot water through them or by means of internal electrical resistances are used for the deposition of iron from the usual electrolytes, while the bath is kept cool by promoting evaporation from its surface, by addition of cold water, or by direct water-cooling devices.

A. R. POWELL.

Rust-resisting [tinned-iron] can. N. HARRIS (U.S.P. 1,694,820, 11.12.28. Appl., 21.12.25).—Previous to tinning, the metal is treated by a suitable process, *e.g.*, pickling in a solution of ferric and antimony chlorides, to obtain a black, rust-resisting film. The ordinary hot-dipped tin coating adheres well to the treated surface, and danger of rusting due to perforation of the tin layer is avoided.

A. R. POWELL.

Copper-silicon alloys. A. L. MOND. From METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 303,684, 29.2.28).—A copper alloy contains 1–4% Si, 4–6% Fe, 1–5% Ni, and not more than 20% Zn or 1% Sn, Mn, or Al. The alloy, which may also contain 0.001–0.5% of an alkali or alkaline-earth metal is quenched from 750–950° and reheated at 350–600°.

C. A. KING.

Extracting nickel from its alloys. SOC. ANON. "LE NICKEL" (B.P. 288,605, 3.4.28. Fr., 13.4.27).—The alloy, *e.g.*, ferronickel, is first purified by known

methods, *e.g.*, preferential oxidation, to obtain a metal containing 90–95% Ni. This is cast into anodes and electrolysed in a divided cell, using a nickel chloride or sulphate electrolyte in both compartments. The anolyte is continuously removed from the cell to a vessel where it is treated with nickelic hydroxide or with nickelous hydroxide and a current of air to precipitate iron and other impurities. The filtered purified solution then passes to the cathode compartment, in which it is kept neutral by the addition from time to time of nickelous hydroxide. When the proportion of nickel in the catholyte falls below a predetermined value the solution is returned to the anode compartment and replaced by purified anolyte. A. R. POWELL.

Metallurgy of ores or materials containing tin. E. A. ASHCROFT (B.P. 302,851, 5.4.28).—Tin ores, concentrates, slags, or residues are heated with zinc or ferrous chloride and a reducing agent, such as zinc powder or sponge iron, with or without a carbonaceous material, to such a temperature that the tin is converted into stannous chloride which distils out of the mass. The tin may be recovered from the distillate by electrolysis in the fused state or in solution in water, by treating the fused stannous chloride with metallic zinc or by treating the aqueous solution with ammonia and reducing with carbon the resulting hydroxide precipitate. The residues from the distillation may be heated with carbon to reduce the iron oxides therein to sponge iron, which is then magnetically separated from the gangue for use again. When ammonia is to be used as precipitant it may be advantageously obtained by heating ammonium chloride with sponge iron so as to obtain a mixture of iron and ferrous chloride for treating further quantities of ore. The ammonium chloride is subsequently recovered by evaporating the filtrate from the stannous hydroxide. A. R. POWELL.

Purification of antimonial lead alloys and other antimonial metals. H. HARRIS (B.P. 303,328, 29.5.28).—The molten alloy is treated at temperatures between the m.p. and 550° with a mixture of 3 pts. of sodium hydroxide and 1 pt. of sodium chloride, with or without agitation by means of a current of air. Arsenic is removed first, then the tin, and, by suitable adjustment of the treatment, slags may be obtained from which these metals may be recovered separately. A. R. POWELL.

Manufacture of non-ferrous alloys [lead bronzes]. METROPOLITAN-VICKERS ELECTRICAL CO., LTD., ASSECS. OF W. J. MERTEN (B.P. 291,462, 2.6.28. U.S., 4.6.27).—A lead bronze bearing alloy containing 10–28% Pb, 2–10% Sn, and 88–62% Cu is rendered homogeneous by treating it just prior to casting with about 2% of calcium carbide, which serves to eliminate oxides and adsorbed gases and to cause the lead to remain in a highly dispersed form throughout the casting. A. R. POWELL.

Lead-base bearing metal containing zinc. J. NEURATH (Aust. P. 107,316, 16.6.26).—The alloy consists of lead with tin and antimony and up to 5% Zn, preferably 1–3% Zn, together with one or more metals of the first and second groups of the periodic system. The alloy is melted in the usual way, and is

allowed to cool slowly and free from vibration through the range 360–340° so as to cause the zinc to crystallise in the α -form, which increases the tensile and compression strength of lead much more than does the β -form.

A. R. POWELL.

Recovering vanadium [from ores]. I. M. PAUL, ASSR. TO METAL & THERMIT CORP. (U.S.P. 1,696,923, 1.1.29. Appl., 12.8.26).—The ore is heated with crystals of sodium sulphide until the latter melt in their water of crystallisation and the mixture finally dries to a solid mass. Extraction of this mass with water yields a solution containing the vanadium. A. R. POWELL.

Recovery of precious metals from ores. W. R. B. GIESEN (Austral. P. 621, 18.2.26).—The ore pulp is allowed to flow down a series of inclined plates coated with sodium amalgam, which is continually renewed from an electrolytic apparatus in which a solution of sodium chloride is electrolysed between a carbon anode and a mercury cathode. A. R. POWELL.

Coating and colouring of metals. A. PACZ (B.P. 302,943, 22.6.27).—Iron, aluminium, tin, lead, magnesium, zinc, or cadmium may be provided with a protective, coloured coating by immersion in a solution containing a soluble molybdate and one or more of the following acids or their salts: oxalic, hydrofluoric, hydrofluosilicic, silicic acid. Before treatment with this solution iron articles may be provided with a smooth coating of copper, and after treatment the coating may be further coloured by immersing the article in a solution of a chromate. A. R. POWELL.

Purification [filtering] of [light] metals. BRIT. MAXIMUM, LTD. (B.P. 300,164, 21.3.28. Fr., 7.11.27).—Light metals may be filtered in the molten state by passing them through a metal gauze covered superficially with an alloy or amalgam having m.p. below 190°. A suitable amalgam is composed of 2 pts. of tin, 1 pt. of lead, and 1 pt. of mercury; it may also contain metals the presence of which is useful in the filtered metal. The metal gauze is first pickled, then tinned, again pickled, *e.g.*, with hydrofluoric acid, and finally dipped in the amalgam. M. E. NOTTAGE.

Protective compositions for lining metal moulds. L. J. BARTON (B.P. 303,205, 31.8.27).—A mixture of a heavy oil and a refractory material, such as fireclay, lime, quartz, ganister, dolomite, or carbonaceous material, is claimed. A. R. POWELL.

Electrolytic manufacture of metal powders. H. WADE. FROM SOC. ANON. TREFILIERIES ET LAMINOIRS DU HAVRE, ANC. ETABL. L. WEILLER, SOC. CO-OPÉRATIVE DE RUGLES, ET LA CANALISATION ÉLECTRIQUE RÉUNIS (B.P. 303,984, 3.12.27).—The electrolyte is treated with finely-divided charcoal, or with colloidal carbon such as is obtained by the action of sulphuric acid on glucose. From baths so treated copper, zinc, cadmium, tin, or lead may be obtained in a form which is readily broken up to a powder which will pass 250-mesh. A. R. POWELL.

Production of magnetic dust cores. G. W. ELMEN, ASSR. TO BELL TELEPHONE LABS., INC. (U.S.P. 1,695,041, 11.12.28. Appl., 17.12.26).—A magnetic metal is atomised and the spray is collected in water,

whereby fine spherical particles of metal are obtained. These are dried, heated to oxidise the surface, and pressed into magnetic cores. A. R. POWELL.

Increasing the electrical conductivity and the flexibility of [aluminium] metals or alloys. K. HALLMANN (U.S.P. 1,695,044, 11.12.28. Appl., 21.9.22. Ger., 11.8.22).—Aluminium alloys containing 0.5–1.0% Si, up to 0.2% Fe, and 0.1–0.7% Mg are hot-rolled at 540–560° to 10–20 mm. rods, which are quenched, aged for several days at 20°, drawn down to wire of finished size, and annealed for 8–12 hrs. at 160–200°. A. R. POWELL.

Chromium-plated tools. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF H. P. HOLLNAGEL (B.P. 278,734, 7.10.27. U.S., 8.10.26).—Cutting tools such as reamers etc. are first cleaned by cathodic electrolysis in an alkaline bath, then dipped into 10% hydrochloric acid solution, washed, and plated with chromium in a bath containing 3 g. of chromium sulphate, 7 g. of chromium carbonate, and 270 g. of chromic acid per litre. A current density of 100–280 amp./ft.² is used with a combined lead and chromium anode, the work being revolved at 35 r.p.m. during plating. A. R. POWELL.

Hardness testing and hardening of metals. E. G. HERBERT (B.P. 301,762, 29.8.27).—The article to be tested is subjected to bombardment by a shower of hardened steel balls moving at a velocity just insufficient to indent surfaces possessing the desired degree of hardness; softer surfaces are then easily recognised by their roughened appearance. Alternatively, the balls are ejected on to the surface at a greater velocity such that all parts are indented, hard parts only slightly and soft parts more deeply; if the velocity of the balls is known the diameter of the depression is a measure of the hardness of the metal at that spot. The bombardment method may be used for "superhardening" the surface of metal objects by repeatedly bombarding them with balls at a gradually increasing velocity just above that required to cause indentation at every stage of the process. Apparatus for carrying out the hardness test and for hardening is described and claimed.

A. R. POWELL.

Heat-treatment of metals and alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,700,547—8, 29.1.29. Appl., [A] 21.11.25, [B] 29.5.26. U.K., [A, B] 3.10.25).—See B.P. 263,923; B., 1927, 224.

Treatment of low-carbon steel etc. A. FRY, Assr. to F. KRUPP A.-G. (U.S.P. 1,700,674, 29.1.29. Appl., 8.9.26. Ger., 7.7.26).—See B.P. 274,016; B., 1927, 912.

Wet magnetic separation of ores and other mixtures of materials on drum separators. G. ULLRICH, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,697,375, 1.1.29. Appl., 1.8.24. Ger., 13.9.23).—See B.P. 227,069; B., 1925, 177.

[Apparatus for the hot] forging of metals [with resistance heating]. L. RENAULT (B.P. 303,340, 27.7.28. Fr., 7.1.28).

Hardening of metal surfaces [gear wheels]. A. E. SHORTER, C. L. BOUCHER, and PATENT GEAR HARDENING CO., LTD. (B.P. 303,196, 29.9.27).

["Grease pot" device for] manufacture of tin plates. W. H. PHILLIPS and W. DAVIES (B.P. 303,846, 11.10.27).

Separating lead from solutions (B.P. 304,054). **Dissolution of ores** (B.P. 282,772).—See VII. **Enamel-coated article** (U.S.P. 1,693,130).—See VIII.

XI.—ELECTROTECHNICS.

Electrical conductivity of kerosene and gasoline as a function of the temperature. C. A. MOREHOUSE (Proc. Iowa Acad. Sci., 1927, 34, 271–272).—The increase in conductivity of kerosene with rise of temperature is expressed by the equation $R = 290.53 \times (1 - 0.0161t + 0.000068t^2)$. The resistance of gasoline decreases very rapidly with rise of temperature.

CHEMICAL ABSTRACTS.

See also A., Feb., 126, **Hall effect and magnetic induction in electrolytic iron** (PUGH). **Monel metal** (SCHULZE; KUSSMANN). 145, **Photo-electric cells of thallium** (MAJORANA and TODESCO). 161, **Wood's metal as cathode in electrolysis** (PIETERS). 164, **Electrodeposition of cadmium and zinc** (BRENNER). 166, **Electric arc in gases at low pressures** (NEWMAN). **Potentiometric measurement of alternating current resistance** (DENINA).

Resistivity of textiles. MURPHY and WALKER.—See V. **Removal of carbon dioxide from air.** MÜLLER and others.—See VII. **Cottrell-Möller plant for flue gases.** SCHLEICHER and GÖBEL. **Steel for magnets.** BRECHT and others. **Electrolysis of nickel.** BOGITCH.—See X. **Conductimetric study of beet juices.** STANEK and ŠANDERA; also NETUKA. **Determination of sugar.** TRYLLER.—See XVII.

PATENTS.

Electric furnace. J. C. WOODSON, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,697,486, 1.1.29. Appl., 10.2.25. Renewed, 26.10.28).—Independent resistor members comprising electric resistors interlocked with refractory members are supported by a resistor unit supporting member held in position by the bottom wall of the furnace, and are arranged in substantially parallel rows and in open, skeletal, spaced relation above the bottom wall. J. S. G. THOMAS.

[Three-phase current] electric induction furnace. M. SURJANINOV (B.P. 279,883, 27.10.27. Austr., 30.10.26).—The furnace container is wound so that between each pair of turns or groups of turns connected in the same direction to the mains there lies a third in reverse connexion with the main, so that an approximately constant magnetic field travels transversely to the turns. J. S. G. THOMAS.

Electric conductors of high negative temperature coefficient. H. ANDRE (B.P. 292,152, 17.4.28. Fr., 17.6.27. Addn. to B.P. 280,862; B., 1929, 61).—The contact pieces fixed to the conductor previously described are formed of a conducting type of carbon, e.g., retort carbon or graphite, whereby sulphuration and the segregation of sulphur from the conductor are prevented. J. S. G. THOMAS.

Magnetic pyrometer. T. SPOONER, Asst. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,697,148, 1.1.29. Appl., 14.8.26).—Windings upon a core comprising sections of different magnetic characteristics are connected to means which are responsive to temperature changes in the core. J. S. G. THOMAS.

Arc-welding electrodes. ALLOY WELDING PROCESSES, LTD., and E. H. JONES (B.P. 303,996, 13.12.27).—The electrodes are coated with a film impervious to moisture by dipping into varnish, cellulose lacquer, etc. J. S. G. THOMAS.

[Acid electrolyte for] zinc-lead electric accumulator. A. POUCHAIN (B.P. 290,665, 9.5.28. Ger., 19.5.27).—During operation of the accumulator the acidity of the electrolyte is maintained at 80–280 g. of free sulphuric acid per litre. Thus the electrolyte initially may have the composition: water 1 litre, sulphuric acid 80–150 g., zinc sulphate 450–650 g., and 15–30 g. of substances having catalytic and complex actions, *e.g.*, aluminium sulphate or boric acid. J. S. G. THOMAS.

Photosensitive cell. Photo-electric cell. S. RUBEN (U.S.P. 1,694,189 and 1,694,190, 4.12.28. Appl., [A] 6.12.27, [B] 18.10.28).—(A) A cathode, an anode, and a control device consisting of cuprous oxide are mounted in an electrical discharge tube. (B) A control device comprises a metal body coated with a photosensitive compound of the oxygen series of the sixth periodic group. J. S. G. THOMAS.

Treatment of magnetic materials. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 303,928, 14.10.27).—Nickel-iron alloys containing 60–80% Ni are heated at 600–900° for at least 2 hrs., and then cooled at a rate not exceeding 10° per min., whereby they acquire a magnetic permeability which is constant at field strengths up to 0.1 gauss. J. S. G. THOMAS.

Removal of magnesium oxide from zinc sulphate electrolytes. NORSKE ZINK KOMPANI A/S. (F.P. 629,216, 17.2.27. Nor., 18.12.26).—A quantity of the circulating liquor in the electrolytic recovery of zinc from ores is periodically removed from the circuit, neutralised with limestone, and filtered. The zinc in the filtrate is precipitated by addition of slaked lime, and magnesium salts are recovered from the filtrate. The zinc precipitate is returned to the main circuit. A. R. POWELL.

Anode for electrolysis of copper solutions. F. LAIST and F. F. FRICK (U.S.P. 1,697,771, 1.1.29. Appl., 21.7.26).—The anodes are composed of alloys of lead, antimony, and arsenic. H. ROYAL-DAWSON.

Electrolysis of ferrous chloride. S. I. LEVY and G. W. GRAY (B.P. 304,053, 10.2.28).—A concentrated solution of ferrous chloride is electrolysed at 90–100° in an electrolytic cell having a suitable cathode on which iron is deposited and a carbon or like anode surrounded by a concentrated solution of a stable chloride, *e.g.*, of calcium, sodium, magnesium. Chlorine evolved at the anode is collected, and the solution of ferrous chloride flows through the cell at such a rate that the issuing spent liquor contains about 20% by wt. of ferrous chloride. The current density employed is 10–25 amp./sq. ft. at 2.3–3.0 volts. J. S. G. THOMAS.

Manufacture [ageing] of electric incandescence lamps. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 294,242, 9.7.28. Ger., 21.7.27).

[Forming] filaments for electric incandescence lamps. FALK STADELMANN & CO., LTD., Assees. of NEUE GLÜHLAMPEN GES.M.B.H. (B.P. 297,052, 24.7.28. Ger., 13.9.27).

Electroplating of annular articles and apparatus therefor. DUNLOP RUBBER CO., LTD., and H. TREVASKIS (B.P. 304,029, 19.1.28).

Determination of combustible gas (B.P. 282,080). **High-viscosity oils** (B.P. 303,776).—See II. **Separation of lead from solutions** (B.P. 304,054).—See VII. **Heating furnace** (U.S.P. 1,692,614). **Hardening of ferromagnetic materials** (B.P. 282,750). **Metal coatings** (B.P. 303,034). **Electrolytic iron** (Swiss P. 121,383). **Extracting nickel from its alloys** (B.P. 288,605). **Precious metals from ores** (Austral. P. 621 of 1926). **Metal powders** (B.P. 303,984). **Aluminium alloys** (U.S.P. 1,695,044). **Chromium-plated tools** (B.P. 278,734).—See X. **Moulded insulators** (B.P. 284,232).—See XIII. **Introducing chemicals into rubber** (B.P. 303,564).—See XIV.

XII.—FATS; OILS; WAXES.

Absorption spectra and fluorescence of fats. H. P. KAUFMANN (Chem. Umschau, 1929, 36, 34–35).—A preliminary announcement is given of work on the examination of cacao butter and its adulterants by absorption and fluorescence spectroscopy.

E. LEWKOWITSCH.

Solution of the butyrometer problem. G. ROEDER (Milch. Forsch., 1928, 5, 258–297; Chem. Zentr., 1928, ii, 1280).—The amount of fat attacked and taken up by the sulphuric acid is given by the expression $0.0233f - 0.00362f^2$, or in presence of protein (cream) $0.01218f - 0.00148f^2$, where f is the quantity of fat observed with 21 c.c. of acid. A. A. ELDRIDGE.

Theory and practice of tung oil [gelation]. R. BÜRSTENBINDER (Chem. Umschau, 1929, 36, 35–38).—The theory of the gelatinisation and re-liquefaction of tung oil is discussed in the light of the methods employed in practice to hinder gelling. Gelling of wood oil appears to begin with an esterification (absorption of glycerol) accompanied or followed by polymerisation and condensation (elimination of glycerol); the changes of colloidal character are only secondary phenomena. Linseed oil and rosin (or their fatty acids) are considered to hinder gelling by a process of esterification or formation of mixed glycerides; the liquefying action of inert substances (*e.g.*, mineral oils) is supposed to be one of simple syneresis. E. LEWKOWITSCH.

Some Mackey tests on cottonseed oil. H. ASPEGREN (Oil & Fat Ind., 1929, 6, 19–23).—The Mackey curves (temperature developed-time) were plotted for hardened refined cottonseed oil at various stages of hydrogenation: the rate of temperature rise was less as the proportion of linoleic acid decreased, being very low when the linoleic acid content fell to 16%, and there appeared to be no tendency to

spontaneous heating when this acid had disappeared, although 83% of oleic and isooleic acids was present. When mixtures of the original and hardened (3% linoleic acid) oils were compared with hardened oils of the same linoleic acid content, the former tended to develop the steeper heating curves. Experiments on other oils confirmed the conclusion that linoleic acid was principally responsible for the heating tendency. The stearine (iodine value 88) separated from a winter oil showed a greater tendency to spontaneous heating than the demargarinated or the original oil (iodine value 109.2). Crude cottonseed oil showed no tendency to oxidise, the typical heating curves only appearing from refined oils.

E. LEWKOWITSCH.

Oil from the seeds of *Adenanthera pavonina*. A source of lignoceric acid. S. M. MUDDIDI, P. R. AYYAR, and H. E. WATSON (J. Indian Inst. Sci., 1928, 11A, 173—180).—Extraction of the kernel (49.7% of whole seed) of the seed (av. wt. 0.24 g.) of *Adenanthera pavonina* yields 28% of oil. The whole seed yields 14% of oil having $d_{15.5}^{20}$ 0.9168, n_D^{60} 1.4570, acid value 0.56, iodine value 87.9, saponif. value 181.4, unsaponifiable matter 1.4% (sitosterol 46%), acetyl value 3.4, Reichert-Polenske value 1.22, Polenske value 0.24, and viscosity (Redwood) 188 sec. The relation between iodine value and refractive index at different stages of hydrogenation of the refined oil is given. The mixed fatty acids of the oil have titre 58.4°, n_D^{60} 1.4541, mean mol. wt. 301, unsaturated acids 64% (mean mol. wt. 286), saturated acids 36% (mean mol. wt. 328), iodine value of unsaturated acids 111, Hehner value 95.5, and consist of myristic (0.4%), palmitic (9.0%), stearic (1.1%), lignoceric (25.5%), oleic (49.3%), and linoleic (14.7%) acids. Almost pure lignoceric acid, m.p. 80°, may be obtained from the mixed fatty acids by one crystallisation from alcohol.

E. H. SHARPLES.

Oxidation and polymerisation of sunflower oil. G. NEMIROVSKI (Oil & Fat Ind. Russia, 1928, No. 4, 23—26; Chem. Zentr., 1928, ii, 717—718).—When a current of air is passed through the oil at 115—120° or 130—135° the content of oxy-acids and (less rapidly) the viscosity increase with time of exposure and with rise of temperature. Catalysts (cobalt > lead > manganese salts) accelerate the reaction. Lead linoleate is more effective than the acetate. These compounds are considered to activate the true catalyst, of unknown nature, present in the oil.

A. A. ELDRIDGE.

Determination of the iodine value [of oils]. I. V. KUBELKA, J. WAGNER, and S. ZURAVLEV (Collegium, 1929, 17—30).—The iodine values of a number of different oils have been determined by the method of (a) Hanus (B., 1901, 1246), (b) Margosches and others (B., 1924, 639, 680), and (c) Rosenmund and Kuhnhehn (B., 1924, 23). All three gave concordant results for oils with an iodine value < 100, but varying results for oils with higher values. The value obtained varies with the amount of oil used in the determination. Method (b) gives the lowest results when a greater weight of oil is used, whilst method (a) is least sensitive to this variation, an excess of 100% on the prescribed weight of oil being permissible.

D. WOODROFFE.

See also A., Feb., 220, Biochemical production of fats (KULIKOV).

Action of silent discharge on oils. RABEK.—See II.

PATENTS.

Production of emulsions from fatty materials. H. E. POTTS. From V. A. T. FAURSKOV (B.P. 304,256, 12.10.27).—Fatty materials may be emulsified, and hardened fats rendered plastic, by feeding the materials on to a pair of rolls, rotating in opposite directions with a peripheral velocity not less than 30 m./min. Emulsification is effected by the mixing action in the groove between the rolls, followed by pressure as the material passes between the revolving surfaces.

E. LEWKOWITSCH.

Treatment of oil seeds and the like. E. A. ANDRÉ (B.P. 279,122, 17.10.27. Fr., 15.10.26).—Oil seeds having an oil content of 30% or more and aleurone grains (e.g., linseed, sunflower, etc.) are crushed and the oil is extracted by a neutral hydrocarbon solvent in the absence of water; the methods of dressing (methodical bolting) employed in milling are applied to the oil-free meal to obtain graded nutritive products.

E. LEWKOWITSCH.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oxidation of oil of turpentine. J. TERPOUGOV (Bull. Inst. Pin, 1928, 103—105; Chem. Zentr., 1928, ii, 1039).—A deposit from Portuguese turpentine oil consisted of zinc formate, iron salts and oxides, abietic acid, and acids of high mol. wt. The acids were oxidation products arising from the catalytic or electrolytic effect of the galvanised iron vessel, formic acid probably having been produced by degradation of soluble long-chain compounds.

A. A. ELDRIDGE.

[Oleo-casein] emulsion vehicles. H. WAGNER and J. KESSELRING (Farben-Ztg., 1929, 34, 947—954).—A comprehensive dissertation on the preparation and properties of oleo-casein water-paint vehicles. The qualitative and quantitative influence of alkalis on the consistency of casein solutions, and the specific effects of various preservatives are detailed. Casein and alkali caseinates are converted into insoluble calcium caseinate by the action of lime, but excess of lime has a peptising action and the caseinate becomes soluble, a significant factor in casein water-paint technique. The emulsification of casein solutions is discussed from the points of view of influence of concentration and acidity of the casein solution, concentration and nature of emulsoid (phase reversal being negligible in the type of material considered), and behaviour of the emulsions when treated with water, photomicrographs illustrating the different behaviour of various emulsions being included. Tung oil produces markedly different results from linseed oil. The properties of a range of commercial casein emulsions are tabulated. The general colloid-chemical relationships involved are discussed.

S. S. WOOLF.

Distribution and properties of resinous substances in various parts of the trunk of pine trees (*Pinus sylvestris*) according to seasons. I. V. FILIPOVICH and V. A. VUISOTZKI (J. Chem. Ind. Moscow,

1927, 4, 953—960).—The resinous material extracted by ether from the heartwood is yellow, and from the sapwood greenish and more stable. The former deposits crystalline precipitates of higher acid and saponif. values than the original. The difference between the saponif. value and the acid value of the resinous material is: heartwood 0—8, sapwood 6—28; the unsaponifiable matter in sapwood resinous material is the higher. Acid values of rosin vary with the season: heartwood 132—163, sapwood 52—151. The quantity of substances which can be saponified and salted out is for the heartwood rosin always higher than the unsaponifiable matter; for sapwood rosin it may be lower. The composition of the rosin depends on the position of the cutting. Heartwood rosin has a lower m.p. in summer; the sp. gr. of both heartwood and sapwood rises in midsummer and drops in autumn; the value of α is negative in winter and positive in summer. The acid and saponif. values, and the percentage of substances which can be salted out, also vary with the season.

CHEMICAL ABSTRACTS.

Cause of the low m.p. of colophony obtained by extraction. E. LJUBARSKI (Oil & Fat Ind. Russia, 1928, No. 4, 26—30; Chem. Zentr., 1928, ii, 716).—On account of chemical transformations leading to an increase in the amount of turpentine of b.p. $>180^\circ$, which remains in the residue after steam-distillation of the oil, colophony extracted from the dead tree has a lower m.p. than that drawn from the living tree. The colophony is preferably saponified, the oil of high b.p. forming an emulsion and being removed by distillation with steam.

A. A. ELDRIDGE.

Resin acids. G. ROVIN (Bull. Inst. Pin, 1928, 121—130; Chem. Zentr., 1928, ii, 1222).—The resin of *Pinus Laricio* afforded *d*-pimaric acid, m.p. 209—210°, whilst *l*-pimaric acid was not isolated; optical data indicate that the two acids are present in the proportion 74 : 26.

A. A. ELDRIDGE.

Abietic anhydride. W. NAGEL (Chem. Umschau, 1929, 36, 33—34).—The product obtained by treating abietic acid (isolated from rosin by cold extraction or by distillation) with acetic anhydride was proved conclusively to be an anhydride. The recrystallised product had m.p. 148—150°, acid value 0; the saponif. value agreed approximately with the theoretical requirements. Fonrobert and Pallauf's observations (B., 1926, 594) on the anhydride are confirmed, except that hydrolysis of the compound was found to be difficult; the anhydride can even be recrystallised from boiling alcohol.

E. LEWKOWITSCH.

See also A., Feb., 178, **High-molecular condensation products of carbamide and formaldehyde** (SCHEIBLER and others). 191, **New compounds from oil of turpentine** (WIENHAUS).

PATENTS.

[Cellulose ester] lacquer compositions. (Sir) G. C. MARKS. From CARBIDE & CARBON CHEM. CORP. (B.P. 285,049, 1.9.27).—The compositions comprise a solvent, a cellulose ester, and a polymerised substituted olefine, e.g., polymerised vinyl acetate or a polymerised mixture of 30% of vinyl chloride and 70% of vinyl acetate.

L. A. COLES.

Preparation of a softening agent for coating compositions containing nitrocellulose etc. Coating compositions. E. C. R. MARKS. From ALCOX CHEM. CORP. (B.P. 303,560 and 303,566, 6.10.27).—(A) A petroleum of about C_{20} is oxidised in the liquid phase at 135—140° with air at 150—350 lb./in.² in presence of a catalyst such as manganese oleate. The terminal carbon atom is removed as formic acid, leaving a high-molecular carboxylic acid soluble in petroleum, together with ketones and aldehydes, but further oxidation, which would result in hydroxy-acids insoluble in petroleum, is avoided. (B) The product is used as a plasticiser for coating compositions of nitrocellulose for artificial leathers, or is esterified to give a castor-oil substitute.

C. HOLLINS.

Manufacture of condensation products from formaldehyde and thiourea or a mixture of thiourea and urea. SOC. CHEM. IND. IN BASLE (B.P. 275,995, 10.8.27. Switz., 11.8.26).—Clear products are obtained by condensing not less than 1 mol. of thiourea or urea, or mixtures of these, with 1.6 mols. of formaldehyde by heating in aqueous solution at the ordinary or at raised pressure and at a temperature not much below 100°, until a sample yields a precipitate on dilution with water, when the solution is concentrated and the residue is hardened by the usual method. Alternatively, after initial condensation of 1 mol. of urea etc. with not more than 2 mols. of formaldehyde, a quantity of urea etc. is added to bring the proportion to not less than 1 mol. per 1.6 mols. of formaldehyde, after which the solution is concentrated and the residue hardened. L. A. COLES.

Manufacture of moulded products [for use as electrical insulators]. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF H. L. WATSON (B.P. 284,232, 9.1.28. U.S., 25.1.27).—Dried felt material, e.g., kraft sulphate wood-pulp sheet, is impregnated with a solution of a natural or artificial resin, e.g., Bakelite, and, after removal of the solvent, the product is moulded by heat and pressure.

L. A. COLES.

Production of new derivatives of [resinous] condensation products of aldehydes and phenols. H. BUCHERER (U.S.P. 1,697,713, 1.1.29. Appl., 18.4.21. Ger., 10.6.18).—See B.P. 148,139; B., 1922, 110 A.

Ink from hydrocarbons (B.P. 286,207).—See II. **Titanium etc. compounds** (B.P. 275,672). **Titanic oxide** (B.P. 303,468).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Freezing of raw rubber. I. A. VAN ROSSEM and J. LOTICHUUS. II. J. R. KATZ (Kautschuk, 1929, 5, 2—5, 6—9).—I. The sp. gr., hardness, and light-absorption of frozen rubber undergo a sudden marked decrease between 36° and 38°, probably due to the fusion of the crystalline constituent. The existence of a crystalline fraction in frozen rubber is supported by the much lower water-absorption of this relative to ordinary rubber. A distinct latent heat of fusion can also be detected on inducing thawing of frozen rubber by the addition of toluene in a Bunsen ice-calorimeter. The m.p. is lower for recently frozen samples than for rubber which has been in a frozen state for years; this difference is

attributed to the effect of crystal size which would become greater with time.

II. The X-ray interference phenomena with frozen rubber show this to contain crystalline material, whereas unfrozen rubber is amorphous. Immersion in a solvent, *e.g.*, benzene or carbon tetrachloride, rapidly converts frozen rubber into the completely amorphous condition, as also does repeated stretching or bending. The disappearance of the crystal-interferences on warming coincides with the later portion of the fall in the sp. gr. curve. The "freezing" of rubber is therefore regarded as representing merely crystallisation of the material. The difficulty of isolating large crystals is explained by the activity of solvents in effecting conversion of rubber from the crystalline into the amorphous state.

D. F. TWISS.

Oxidation of vulcanised rubber. A. VAN ROSSEM and P. DEKKER (*Kautschuk*, 1929, 5, 13—21).—Systematic investigation of the chemical changes in vulcanised rubber on storage shows that the proportion removable by alcoholic potassium hydroxide from the benzene-swollen and previously acetone-extracted material is a sensitive index to the degree of oxidation in its earlier stages. Equal volumes of benzene and alcoholic potash are desirable. The alcoholic potash extract contains fatty acids, arising from acetone-insoluble esters in the rubber, in addition to the oxidation products; the latter are separable by evaporating the alcoholic potash extract to small bulk, diluting with water, acidifying, extracting with ether, removing this solvent and heating the residue with alcoholic potash, when the oxidation products remain undissolved. The earlier oxidation products contain 3.5—5.5% O and are soluble in ether, but the later products containing 7—8% O are insoluble. Extraction with alcoholic potash therefore constitutes a valuable test for the quality of vulcanised rubber particularly in such articles as cannot conveniently be subjected to mechanical tests.

D. F. TWISS.

Topochemical reactions with racked rubber. F. KIRCHHOF (*Kautschuk*, 1929, 5, 9—13).—Thin strips of smoked sheet rubber, which had been "racked" to 600%, when immersed in sulphuric acid (*d* 1.8) for 4 months darkened and lost the power of contraction in warm water; a fibrous structure was still evident both from visual examination and the production of a distinct point-diagram with X-rays. Treatment of similar racked rubber with bromine water also yielded a product with superficial fibrous character, but X-rays revealed no sign of any structure, even the "amorphous ring" being lacking. Bromination apparently causes a complete structural disturbance both in the amorphous constituent and in the crystallites, whereas with sulphuric acid the action appears to be confined mainly to the amorphous phase.

D. F. TWISS.

PATENTS.

Manufacture of goods from aqueous dispersions of or containing rubber and similar resins. DUNLOP RUBBER Co., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 303,544, 5.10.27).—Pasty or creamy products formed directly, from emulsions or dispersions containing rubber or similar materials, by operations such as dipping, spreading, or electrophoresis, are substantially de-

hydrated and set by the osmotic action of a concentrated solution or melt of a salt or salts, with or without the addition of an acid.

D. F. TWISS.

Manufacture of tubes from india-rubber, gutta-percha, balata, or similar materials, or compounds thereof and apparatus therefor. Manufacture of transparent vulcanised rubber. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 303,545—6, 5.10.27).—(A) By forming deposits or products directly from a mixture of rubber latex, sulphur, and an ultra-accelerator of the zinc salt type, *e.g.*, zinc diethyl-dithiocarbamate, and vulcanising, the rubber is obtained in a transparent condition. Any additional compounding ingredients should be substantially soluble in the rubber hydrocarbon or should be removable by subsequent treatment with hot alkali solution or water. The deposits may be treated with a dehydrating and setting medium before completion of the drying and vulcanisation (*cf.* preceding abstract). (B) A flexible or rigid core of suitable composition is led through a bath of concentrated and/or compounded aqueous dispersion of rubber, gutta, or similar material; the coating is then dried, after previous treatment in a dehydrating and setting medium if desired; the formed tube is subsequently removed from the core. The coating and treatment in the dehydrating and setting medium may be repeated to produce greater thicknesses.

D. F. TWISS.

Production of sheet rubber, gutta-percha, balata, and other analogous vegetable resins and fabrics coated therewith. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 302,201, 12.8.27).—Concentrated and, if desired, compounded latex is spread on a moving backing and set by heating, the effect being sufficiently rapid, in the case of a backing of fabric, substantially to prevent penetration. Several layers may be superposed, and the products may be vulcanised during drying or subsequently, and with or without removal from the backing.

D. F. TWISS.

Manufacture of an elastic caoutchouc-like body. J. BAER (B.P. 302,399, 16.9.27. Addn. to B.P. 279,406).—Saturated, halogenated derivatives of the paraffin hydrocarbons, *e.g.*, methylene or ethylene dihalides, mixed or singly, are polymerised by the action of sulphides of the alkali or alkaline-earth metals or ammonium in the presence of alkaline, alcoholic, or other solvent.

D. F. TWISS.

Formation of articles by deposition from emulsions of rubber and viscose or oxycellulose compounds. DUNLOP RUBBER Co., LTD., and W. H. PAULL (B.P. 303,765, 7.10.27 and 18.2.28).—Articles containing rubber and viscose or oxycellulose compounds, if desired together with other ingredients such as zinc oxide, amorphous carbon, vulcanising agents and accelerators, are formed from aqueous emulsions or dispersions of these by electrophoresis and/or by dipping. Further proportions of rubber may be incorporated in the deposits so obtained, *e.g.*, on a mixing mill. Especial reference is made to boots, shoes, and tyre treads.

D. F. TWISS.

Method of introducing chemicals into rubber etc. IMPERIAL CHEMICAL INDUSTRIES, LTD., A. J.

HAILWOOD, W. J. S. NAUNTON, and A. SHEPHERDSON (B.P. 303,564, 7.10.27).—Substances which by anodic oxidation are converted into accelerators are added to a rubber latex or mixture of latex and cellulose ester emulsion for electrodeposition. A metal salt of a leuco-vat dye may be used in conjunction with the accelerator-producing compound. An example is sodium diethyl-dithiocarbamate which gives the thiuram disulphide accelerator at the anode.

C. HOLLINS.

New compounds and their application as vulcanisation accelerators for rubber. IMPERIAL CHEM. INDUSTRIES, LTD., W. J. S. NAUNTON, and J. B. PAYMAN (B.P. 303,535, 31.8.27).—*Morpholine morpholyl-dithiocarbamate*, m.p. 187°, prepared from morpholine and carbon disulphide, is oxidised to *dimorpholylthiuram disulphide*, m.p. 146–147°, from which the *monosulphide*, m.p. 125–136°, is obtained by action of sodium cyanide, or is treated with sulphur monochloride to give the “*tetrasulphide*,” or converted into the zinc or other heavy metal salts. All these compounds and their analogues from *C*-substituted morpholines are used as vulcanisation accelerators.

C. HOLLINS.

Ebonite and the treatment thereof, more particularly ebonite sheets for wireless apparatus. BRIT. HARD RUBBER CO., LTD., and P. E. WELLS (B.P. 303,814, 6.9.27).—Ebonite immune to the action of light is produced by applying to the surface of ordinary ebonite a thin coating of any synthetic resin, e.g., of the phenol-aldehyde type.

D. F. TWISS.

XV.—LEATHER; GLUE.

Smoke tannage. Effect of wood smoke on combining capacity of hide substance for chromium trioxide, vegetable tannin, and sodium hydroxide. L. S. TS'AI and E. O. WILSON (J. Amer. Leather Chem. Assoc., 1929, 24, 21–36).—Smoke tannage is effected by subjecting wet delimed pelt to the action of the smoke from green straw, the pelt is then folded, laid aside, and allowed to cool. The treatment is repeated three or four times. The product may be retanned with vegetable tannins. Smoke-tanned leather has a smell of smoke, is yellowish-brown, soft, and pliable, but has a harsh grain. It does not withstand the boiling test. Hide powder treated with wood smoke combines with less basic chromium salt, less vegetable tannin, and more sodium hydroxide, respectively, than untreated powder. These properties are attributed to the inactivation of the basic groups in collagen by the smoke tannage, with consequent activation of the acidic groups. The effect of the smoke tannage on hide powder agrees with that of formaldehyde observed by Gerngross (B., 1922, 149 A, 426 A). The tanning action of smoke is attributed chiefly to formaldehyde and certain phenols in it, which are known to possess tanning properties. D. WOODROFFE.

Determination of non-tans [in tannin analysis]. L. KEIGUELOUKIS (Collegium, 1929, 30–33).—A glass cylinder (200 c.c.), containing the equivalent of 6.25 g. of dry hide powder and ten times its weight of distilled water, is fitted with a rubber stopper, shaken for 1 hr., the necessary amount of chrome alum solution added, well shaken for several hours, and allowed to remain

overnight. A porcelain-headed filter candle is then inserted in the cylinder, the chroming liquor is drawn off, and the powder washed with distilled water and pressed (by means of the candle) until its weight is <26.25 g. Air is then drawn through the filter candle until the adhering powder is sufficiently dried to be removed with a glass rod. The cylinder and chromed powder are weighed and the latter is made up to 26.25 g. with distilled water. Tan liquor (100 c.c.) is added, the cylinder shaken for 10 min., and the non-tans are drawn off through a filter-candle and collected. A second filtration is not necessary in the case of non-tans. The method obviates the handling of the powder, and can be used for ready-chromed hide powder.

D. WOODROFFE.

See also A., Feb., 137 and 138, **Swelling of gelatin** (KESTENBAUM; KUNITZ).

PATENT.

Deposition of rubber, viscose, or oxycellulose (B.P. 303,765).—See XIV.

XVI.—AGRICULTURE.

Determination and significance of buffering power of soils poor in carbonate. K. MAIWALD (Kolloidehem. Beih., 1928, 27, 251–343).—An investigation has been made of 14 kinds of soil of glacial origin, containing 1–29.5% of raw clay and 2.6–52.7% of “schluff,” thus comprising a series ranging from light sandy soils to heavy clay soils. Robinson's method (A., 1922, i, 1228) was used to determine the colloid content of the soils, and the unusually high values 12–39% were obtained. The soils may be divided into three groups: (1) saturated, alkaline and neutral soils with low carbonate content, (2) intermediate soils showing acidity by displacement adsorption in potassium chloride solution, and (3) carbonate-free, unsaturated soils. All the soils had a content of replaceable bases (calcium, magnesium, potassium, and sodium) of 21.4–4.8 milliequivalents per 100 g. of soil. The hydrogen-ion concentration of the soils suspended in water and in *N*-potassium chloride with and without addition of acid and alkali was measured, and from the titration curves the following conclusions were reached: (a) the final value is attained only after some days (at least 72 hrs.), (b) the curves show no turning point over the region examined, (c) the curves differ completely from that obtained under similar conditions with quartz-sand in absence of buffers, thus indicating a good buffering capacity, and (d) the titration curves are more or less symmetrical on both the acid and alkaline sides. In those soils which are poor in carbonate, the buffering power is not dependent on the buffer system carbonic acid/bicarbonate, but depends on inorganic and organic complexes—mainly clay and humus compounds. In such cases the degree of buffering is determined by the following factors: (1) chemical character of the colloid complex, expressed as the absolute base-binding capacity, (2) content of adsorbed displaceable bases, (3) colloid content per g. of soil (clay and “schluff”—loam give the best and sand and sandy loam the worst buffering), (4) state of subdivision of the reactive part of the colloid and its alteration with the course of the

titration, (5) the nature and relative proportions of the adsorbed displaceable bases (this factor also influences the state of subdivision, and the tendency to change during the titration), (6) the nature of the acid or alkali added. The electrometric determination of hydrogen-ion concentration in the above systems has been improved by the following devices: a simple potentiometer as compensation instrument, ensuring the rapid reading of potential in a series of measurements; simultaneous use of a small bundle of electrodes instead of a single electrode, giving much more trustworthy values; calculation of the titration reagents in true normalities; extension of the time during which the reagent acts on the soil to at least 72 hrs. to attain an end-point. The numerical expressions of the buffering capacities have been calculated by Jensen's method of "buffering surfaces" (B., 1925, 328), and they vary on the acid side from 76.5 cm.² to 27.3 cm.² A new method of expressing buffering capacity is developed on the following lines. The hydrogen-ion concentration a of the titrating acid is determined at various normalities, and also the hydrogen-ion concentration b after addition of the soil to the acid (measured after 72 hrs.). The values of a and b are expressed as gram-ions of hydrogen per litre (not as p_H), and the expression $c = 100b/a$ gives the "remaining acid" as a percentage of the original hydrogen-ion concentration. The values of c are plotted as ordinates against those of a as abscissae. The properties of the curves so obtained with the soils examined are described. Comparison of the curves with the "buffering surfaces" of Jensen shows that soils with equal "buffering surfaces" may differ in their true buffering properties.

E. S. HEDGES.

Biochemical study of some soil fungi with special reference to ammonia production. A. K. THAKUR and R. V. NORRIS (J. Indian Inst. Sci., 1928, 11A, 141—160).—The mechanism of ammonia production by fungi has been examined particularly with reference to an amidase isolated from *Aspergillus flavus*. The reaction was found to be typically autocatalytic. Of the fifty species of fungi isolated from an Indian soil, three gave negative results when examined for their ability to fix atmospheric nitrogen.

E. A. LUNT.

Nitrogen-fixing micro-organisms of an arid soil. E. G. CARTER and J. D. GREAVES (Soil Sci., 1928, 26, 179—197).—Although azotobacter is absent from arid soils, active nitrogen fixation takes place. Twenty-seven organisms are described, all but one of which are capable of nitrogen fixation.

H. J. G. HINES.

Value of nitrification tests on soils representing extreme contrast in physical and chemical properties. W. V. HALVERSEN (Soil Sci., 1928, 26, 221—231).—Nitrification tests employing ammonium sulphate have been criticised on the grounds that the quantity of ammonium sulphate used is abnormal, and the buffering power of the soil is inadequate to counteract the acidity so produced. Data are given which show that these criticisms are invalid except for light sandy soils, and that for normal soils ammonium sulphate can safely be used in the quantities recommended.

H. J. G. HINES.

Fertiliser studies with early potatoes. G. E. V. HOUGHLAND (Soil Sci., 1928, 26, 199—215).—It is recommended that part of the nitrogen should be supplied in an organic form. High-grade potash salts are preferable to the crude forms, and the effect of fertilisers is enhanced by green manuring.

H. J. G. HINES.

Potato plants grown in mineral nutrient media. E. S. JOHNSTON (Soil Sci., 1928, 26, 173—177).—The necessity of adding boron to water cultures is demonstrated.

H. J. G. HINES.

Determination of volume-weight of different soils in the soil profile. J. S. JOFFE and L. L. LEE (Soil Sci., 1928, 26, 217—219).—A correction is made for pebbles giving a value named the active volume weight (cf. Lebedev, B., 1928, 538).

H. J. G. HINES.

Hydrometer method for making a very detailed mechanical analysis of soils. G. J. BOUYOUCOS (Soil Sci., 1928, 26, 233—238).—The method previously described (B., 1928, 683) has been extended to the construction of distribution curves.

H. J. G. HINES.

Effect of fertilisation with iodine on the growth and composition of food plants. E. MAURER, W. SCHROPP, and H. DUCRUE (Münch. Med. Woch., 1928, 75, 1246—1247; Chem. Zentr., 1928, ii, 1027—1028).—By fertilisation with potassium iodide the iodine content of the plants was increased without causing any important undesirable changes in the composition or condition of the plants. The most marked increase was observed with spinach and mangold; it was less evident with savoy, yellow beet, and lettuce, but was not observed with beans, peas, cauliflower, or kohlrabi.

A. A. ELDRIDGE.

Analysis of insecticides. Liquid insecticides non-miscible with water: hydrocarbon mixtures, carbon tetrachloride, nitrobenzene, naphthalene, methyl salicylate. M. FRANÇOIS and (Mlle.) L. SEGUIN (J. Pharm. Chim., 1929, [viii], 9, 49—58; cf. B., 1928, 382, 550, 724).—Methods for the identification and isolation of aromatic and aliphatic hydrocarbons, nitrobenzene, naphthalene, rosin, and methyl salicylate in insecticides are given. The identification of turpentine and the determination of nitrobenzene by reduction to aniline followed by bromometric titration and of carbon tetrachloride by decomposition with alcoholic potassium hydroxide and determination of the chlorine with silver nitrate are described.

E. H. SHARPLES.

Tobacco frencing, a nitrogen deficiency disease. W. D. VALLEAU and E. M. JOHNSON (Ky. Agric. Exp. Sta. Bull., 1927, 28, 179—253).—Prevention of tobacco frencing by addition to the soil of certain minimal quantities of nitrogen compounds is described; chlorotic diseases of other plants are regarded as being of similar nature.

CHEMICAL ABSTRACTS.

See also A., Feb., 222, Amino-acid synthesis in plants (LOEHWING).

Citric acid solubility of slag. KERSTEN.—See X.

PATENTS.

Mixed fertilisers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,916, 12.9.27).—Potassium

nitrate is mixed with sodium nitrate, the amount of the sodium salt being not more than 60%, preferably 20–50%, of the mixture.

W. G. CAREY.

Manure and insect-destroying composition. R. TOYER (B.P. 303,957, 8.12.27).—Soot, lime, sand, and sawdust in the proportion 2:2:1:1 are mixed with coal tar or wood-tar oil and paraffin.

W. G. CAREY.

Preparation of fertilisers. F. ROTHE and H. BRENEK, Assrs. to VER. CHEM. FABR. A.-G. (U.S.P. 1,696,975, 1.1.29. Appl., 26.1.27. Ger., 23.6.24).—See B.P. 235,860; B., 1925, 731.

XVII.—SUGARS; STARCHES; GUMS.

Effect of the drying of fresh beet cossettes on the content of nitrogenous compounds in the exhausted cossettes. O. SPENGLER and W. PAAR (Z. Ver. deut. Zucker-Ind., 1928, 796–800).—A 10 kg. batch of fresh beet slices was divided into two equal parts, one of which was dried on trays to a moisture content of 4%, and both were then extracted with hot water in a small battery of three glass vessels until the extract run off contained only 0.4% of solids. The drying of the cossettes was found to have no effect on the amount of total nitrogen or true proteins (precipitable by cupric hydroxide) present. Extraction removed 49.8% of the total nitrogen and 25.7% of the true proteins from the fresh cossettes, and 42.2% of the total nitrogen and only 7.7% of the true proteins from the dried cossettes.

J. H. LANE.

Behaviour of oxalic acid in the purification of [beet] juices. O. SPENGLER and C. BRENDL (Z. Ver. deut. Zucker-Ind., 1928, 729–746).—Calcium oxalate is a frequent constituent of evaporator incrustations in beet sugar factories. If a 12% solution of pure sucrose containing oxalic acid is treated by the methods employed in the purification of beet juices, it is found that after liming and heating at 85° as much as 0.08% of calcium oxalate may remain in solution, but this quantity is reduced to 0.001% by the first carbonatation. Larger proportions than this must remain in factory thin juices to account for the amounts sometimes found in evaporator incrustations; possibly colloidal matters interfere with the precipitation of oxalate by carbonatation. The oxalate which escapes precipitation, however, should not be deposited during evaporation if the final alkalinity of the thin juice is due to alkali carbonate, as it should be (B., 1928, 421), and not to free lime; for in the former case it should be present as soluble alkali oxalate during evaporation.

J. H. LANE.

Formation of molasses. O. SPENGLER, F. TÖDT, and C. SHEN (Z. Ver. deut. Zucker-Ind., 1928, 749–759).—The formation of molasses is believed to be due mainly to the influence of salts, colloids, in the amounts present, being unable to prevent crystallisation although they may retard it. The solubility of sucrose in water is, in general, diminished by low concentrations of mineral salts and increased by high concentrations. The former effect is attributable to the competing affinities of salts on the one hand, and sugar on the other, for water, and

therefore varies with the hydration values of different salts. The increased solubility of sucrose at higher salt concentrations is attributed by the authors to the predominance of a third affinity, that of sugar for salts, evidence for which is obtained from a study of the influence of sucrose on the electric conductivity and mol. vol. of salts in aqueous solution.

J. H. LANE.

Laboratory apparatus with conductimetric indication for the study of saturation and filtration [of beet juices]. V. STANEK and K. ŠANDERA (Z. Zuckerind. Czechoslov., 1928, 53, 129–133).—The apparatus comprises a jacketed carbonatation vessel of 3 litre capacity with stirring mechanism, pump, and motor, an electrode vessel with glow-lamp indicators for conductimetric measurements, and a bronze filter press in an attemperating bath. The effects of variations in the conditions of liming, saturation, and filtration can be studied with considerable precision with this apparatus.

J. H. LANE.

Conductimetric control of the rendement of centrifuged [beet] sugar. V. NETUKA (Z. Zuckerind. Czechoslov., 1928, 53, 134–136).—By polarisation and conductimetric ash determinations the rendement of the first centrifugal charge of raw sugar from a given massecuite can be ascertained within 10 min., in time to serve as a guide in the centrifuging of later charges of the same massecuite, thus enabling a factory to attain greater uniformity in the rendement of the raw sugar produced throughout a campaign.

J. H. LANE.

Potentiometric determination of [reducing] sugar. H. TRYLLER (Z. Spiritusind., 1929, 52, 27–28).—In the determination of reducing sugars by titration against Fehling's solution the end-point is ascertained electrically by the vanishing of *P.D.* between two thick copper wires (electrodes) one of which is immersed directly in the boiling reaction liquid and the other is enclosed in a tube containing a similar solution free from copper, closed at the bottom by a porous plug and also immersed in the reaction liquid. The free electrode and the tube containing the enclosed one pass through the stopper of the reaction flask, which also has an opening for admission of sugar solution from a burette and for escape of steam. The two electrodes are wired to a galvanometer. As the end-point of the reaction is approached the galvanometer deflection approaches zero and becomes extremely sensitive to slight changes of copper-content of the reaction liquid, so that the end-point can be easily ascertained to within 0.1 c.c. of sugar solution.

J. H. LANE.

Starch factory control. [Moisture determination.] SPROCKHOFF (Z. Spiritusind., 1929, 52, 27).—In the German starch trade the official method for determining moisture in starch is to dry 5 g. in a shallow dish for 1 hr. at 50° and then for 3 hrs. at 120°. Results accurate enough for factory control can be obtained within 1 hr. by means of an oven described, in which the weighed sample, 10 g., hangs suspended by a wire or chain passing through a hole in the top of the oven and attached to one end of a balance beam mounted above the oven, the other arm of the beam forming a pointer

which moves over a scale as the sample loses weight. The oven is kept at 90–100° for the first 15–20 min. and then at 140–150° for 25–35 min. Accuracy to within 0.2–0.4% is claimed, and the oven can be used also to determine moisture in exhausted potato pulp. J. H. LANE.

Determination of dextrose. W. D. HORNE (Planter Sugar Mfr., 1928, 81, 1).—The cyanide method is modified, the boiled alkaline copper solution being diluted while hot and an aliquot part removed for titration by means of a pipette graduated to compensate for temperature. CHEMICAL ABSTRACTS.

See also A., Feb., 174, **Detection of l  vulose in presence of other carbohydrates** (EKKERT). 175, **Triacetylstarch** (TSUZUKI). **Reaction between sugars and amino-acids** (VON EULER and BRUNUS). 205, **Determination of maltose and dextrose** (BRAUN and others). 216, **Effect of polarised light on starch hydrolysis**. 218, **Fermentation of dextrose** (PEDERSON and BREED).

Sugar-like substances in sulphite-cellulose cooking. H  GGLUND and JOHNSON.—See V.

PATENTS.

High-purity crystalline dextrose and its manufacture. E. C. R. MARKS. From CORN PRODUCTS REFINING Co. (B.P. 300,698, 19.8.27).—Crystalline dextrose is obtained from syrups of high purity (preferably above 94%) by boiling in a vacuum pan at substantially constant temperature, which depends on the form of the sugar required, viz., about 58° for β -anhydrous, about 50° for α -anhydrous, and about 45° for α -hydrated dextrose. After the syrup has been brought to the correct concentration and temperature, which depend to some extent on the purity of the syrup as well as on the form of dextrose required, formation of grain is induced by rapid boiling, usually with a seeding of the appropriate type of crystals, and when sufficient grain is present the density of the syrup is somewhat reduced and crystallisation is continued, without the formation of fresh grain, by slow boiling under a suitable vacuum. Additional syrup may be drawn in as required to feed the crystals, and, after a certain stage, syrup of lower purity than the original one may be used for this purpose, and boiling may be accelerated under a higher vacuum. Crystallisation may be completed by cooling under atmospheric pressure. The masseccuites obtained are easily purged, the crystals being well developed and in most cases single. J. H. LANE.

Preparation of betaine hydrochloride etc. [and glutamic acid from residual liquors of the beet sugar industry]. D. K. TRESSLER, ASST. to LARROWE CONSTRUCTION Co. (U.S.P. 1,685,758, 25.9.28. Appl., 13.7.25).—See B.P. 265,831; B., 1927, 423.

XVIII.—FERMENTATION INDUSTRIES.

Influence of temperature and duration of storage on the fermentative power, cell increase, and acid

production of top-fermentation yeasts. F. WINDISCH (Woch. Brau., 1929, 46, 31–36).—Three top-fermentation yeasts were investigated by methods similar to those employed in the corresponding researches on bottom yeasts (B., 1928, 31, 207, 424). The behaviour of two of these was also tested after storage under beer in addition to storage under water. As with bottom yeasts storage at low temperatures is most favourable to subsequent fermentation increase and acid production, and under water 0° is the best temperature. Under beer 4° was the optimum for the strain Happold 28A and 8° for race 468 (Hochschul-Brauerei). Generally cell increase and acid production were proportional to the attenuation. The detrimental effect of storage at relatively high temperatures (13–18°) is more marked when beer is used as the storage medium. F. E. DAY.

Substitute for beer-wort as yeast medium. J. C. WELDIN (Proc. Iowa Acad. Sci., 1927, 34, 89–90).—Yeasts produce gas abundantly in a medium containing malt extract (Difco) 15 g., potassium monohydrogen phosphate 1 g., ammonium chloride 1 g., distilled water 1000 c.c., adjusted with citric acid to p_H 5.4–5.6; they grow vigorously on a solid medium containing malt extract 15 g., potassium monohydrogen phosphate 3 g., ammonium chloride 1 g., agar 20 g. (e.g.), adjusted with citric acid to p_H 5.4–5.6. CHEMICAL ABSTRACTS.

Formation of ammonia during brewing processes. W. WINDISCH and P. KOLBACH [with K. BAUMANN] (Woch. Brau., 1929, 46, 41–45).—The total (Kjeldahl), formol, and ammonia nitrogen were determined on the cold-water extracts of two barleys and of the malts prepared from them, also on the unboiled, boiled, and boiled and fermented laboratory worts from the malts. Ammonia nitrogen was determined by adding excess of magnesium oxide and distilling *in vacuo* at 40°. The results, calculated on the original dry matter of the barleys, were (%): in the barleys 0.0053, 0.0054; in the malts 0.0063, 0.0053; in the unboiled worts 0.0083, 0.0090; in the boiled worts 0.0207, 0.0196; and in the fermented worts 0.0133, 0.0168% of ammonia nitrogen, respectively. The unboiled worts, of about 11.3% concentration, contained 1.28 and 1.36 mg./100 c.c., respectively, and in that from a third malt 1.32 mg./100 c.c. were found, which increased steadily to 1.92 mg./100 c.c. on prolonging the period of mashing at 45° (protein rest) from 0.5 hr. to 3 hrs. That this effect is enzymatic is probable from the uniform increase of the amount of ammonia nitrogen with increase of time, and since no such increase occurred in a previously boiled wort. Prolonging the protein rest also increased the amount of those substances which give rise to ammonia during boiling. In normal laboratory worts the ammonia nitrogen increased from about 1.3 mg./100 c.c. to about 3.0 mg./100 c.c. during 2 hrs.' boiling, but only by about 0.3 mg./100 c.c. during a further similar period. There was no corresponding change in the formol nitrogen during boiling. A single brewery wort examined contained more ammonia nitrogen than the laboratory worts, the amounts, calculated to 12% wort, being 2.30 and 4.24 mg./100 c.c.,

respectively, before and after boiling. The increase in ammonia nitrogen during boiling was unaffected by varying the reaction between p_H 5.01 and 6.04.

F. E. DAY.

See also A., Feb., 176—8, **Specific action of erepsin and trypsin-kinase** (ABDERHALDEN and others). 180, **Hydrolysis of polypeptides** (ABDERHALDEN and BROCKMANN). 216, **Amylase from Cholam (*Sorghum vulgare*)** (PATWARDHAN and NORRIS). **Nature of amylase** (NARAYANAMURTI and NORRIS). 217, **Vegetable oxidoreductase** (MICHLIN). **Hydrolytic action of *Ricinus* lipase** (PIUTTI and DE' CONNO). **Rate of multiplication of yeast at different temperatures** (RICHARDS). **Production of acids by *Aspergillus fumigatus*** (SCHREYER). 218, **Citric acid formation by moulds** (BERNHARDT and SCHÖN). **Fermentation of dextrose by *Serratia marcescens*** (PEDERSON and BREED). 219, **Acetic acid fermentation** (WIELAND and BERTHO). 220, **Biochemical production of fats** (KULIKOV).

Ammonia production by soil fungi. THAKUR and NORRIS.—See XVI. **Lactose-fermenting yeasts in milk.** TRÜPER.—See XIX.

XIX.—FOODS.

Lactose-fermenting yeasts in milk. E. TRÜPER (Milch. Forsch., 1928, 6, 351—402; Chem. Zentr., 1928, ii, 1276).—To a greater extent than yeasts of winter milk those of summer milk favour higher organic compounds (peptone rather than asparagine) as sources of nitrogen, producing alcohol, lactic acid, pyruvic acid, volatile acids, and a rennin-like enzyme. Lactose, galactose, dextrose, sucrose, and raffinose, but not maltose or arabinose, are attacked. Casein is a source of carbon and nitrogen; other proteins are strongly attacked, but casein only after consumption of fat, which is decomposed by a specific lipolytic enzyme. Protective treatment of the milk is discussed.

A. A. ELDRIDGE.

Influence of saprophytic and parasitic life on the acidoproteolytic power of pathogenic bacteria. C. GORINI (Lait, 1928, 8, 465—474; Chem. Zentr., 1928, ii, 948).—Different strains cause coagulation of milk by different mechanisms.

A. A. ELDRIDGE.

Inoculation of pasteurised milk. A. WOLFF (Milch. Zentr., 1928, 57, 357—364; cf. B., 1929, 70).—Further experiments are described in which pasteurised milk is treated with lactic acid bacteria which have not been heated in a previous pasteurisation process.

F. R. ENNOS.

Alkali value of cow's milk. F. E. NOTTBOHM (Milch. Forsch., 1927, 4, 336—351; Chem. Zentr., 1928, ii, 1276).—The alkali value ($Na_2O:K_2O$) of milk is 2—10; the value for colostrum is generally greater than 2. Towards the end of lactation lower values are obtained. Immediately after establishment of equilibrium or excessive values for sodium the content of lactose falls and that of chlorine rises.

A. A. ELDRIDGE.

Solubility of copper in milk. E. J. SOLOMAN and G. N. QUAM (Proc. Iowa Acad. Sci., 1927, 34, 216—217).—The degree to which copper sheet is dissolved by milk increases with rise of temperature.

CHEMICAL ABSTRACTS.

Determination of silicic acid in milk. O. KETTMANN (Milch. Forsch., 1927, 5, 73—106; Chem. Zentr., 1928, ii, 1280).—Milk contains 1.6 mg. of silica per kg. (0.021% of the ash), with fairly wide variations independent of those of other ash constituents, but dependent on the silica content of the fodder. In colostrum the silica content is above the average. Inflammation of the udder greatly reduces the silica content of the milk.

A. A. ELDRIDGE.

Diffusion of sodium chloride in cheese. O. MROZEK (Milch. Forsch., 1927, 4, 391—402; Chem. Zentr., 1928, ii, 1277—1278).—The distribution of sodium chloride and water after immersion of the cheese in salt solution and slow ripening is described.

A. A. ELDRIDGE.

Evaluation of rennin. E. MUNDINGER (Süddeut. Molkerei-Ztg., 1927, No. 19, 4 pp.; Chem. Zentr., 1928, ii, 1280).—The influence of p_H , calcium oxide and protein content, and dispersion is considered.

A. A. ELDRIDGE.

Action of rennin. E. MUNDINGER (Milch. Forsch., 1927, 4, 369—390; Chem. Zentr., 1928, ii, 1280).—“Degree of acidity” and p_H are both of importance in the preparation of rennin. Sunlight confers on rennin solutions the ability to decompose hydrogen peroxide.

A. A. ELDRIDGE.

Fluorescence and conservation of irradiated foodstuffs. Infant foods. SPOLVERINI (Riv. Ital. Actinol., 1927, 2, 1—19; Chem. Zentr., 1928, ii, 1275).—Ultra-violet irradiation of foods causes physical and chemical changes tending to greater stability, and facilitates their conservation. The foods become more or less radioactive. With certain foods irradiation is harmful.

A. A. ELDRIDGE.

Economic possibilities of rice grass (*Spartina Townsendii*). III. Composition and nutritive value. F. KNOWLES (J. Min. Agric., 1929, 35, 934—939).—Results of analyses made on *Spartina* are given with reference to the following contents: moisture, fibre, protein, ether extract, and ash, the chemical composition of which is also given. Feeding experiments indicate that poor *Spartina* is as digestible and nutritive as poor meadow hay.

E. A. LUNT.

See also A., Feb., 204, **Composition and characteristics of proteins** (SØRENSEN). **Volumetric determination of protein solutions** (TREADWELL and EPPENBERGER). 209, **Lipin content of butter** (REWALD). 219, **Proteolytic bacteria in milk** (FRAZIER and RUPP). 221, **Effect of ethylene on vitamin-B content of celery** (BABB). **Vitamin-B content of wheaten bread baked with varying amounts of yeast** (SCHEUNERT and SCHIEBLICH). 222, **Destruction of vitamin-E in a diet of natural foodstuffs** (WADDELL and others). **Effect of ethylene on bananas during**

ripening (REGEIMBAL and others). Ripening of rice seed (TADOKORO).

Butyrometer problem. ROEDER.—See XII.

PATENTS.

Preservation of eggs for the use of bakers and confectioners. PULVO, LTD., W. F. BOLTON, and C. V. B. READ (B.P. 303,530, 4.7.27).—Eggs are shelled, disintegrated to form a homogeneous mixture of yolk and white, and preheated to 59°. Salt (0.5–3%) is added and the mixture heated *in vacuo* at 54° (e.g., for 10 min.). Finally, 0.25–2% of lactic acid solution (*d* 1.2) is sprayed in. Yolks may be treated separately, in which case the whites are desiccated. Suitable apparatus is described.

B. FULLMAN.

Preparation of fresh fruit for market. E. M. BROGDEN and M. L. TROWBRIDGE, Assrs. to BROGDEN Co. (U.S.P. 1,693,575, 27.11.28. Appl., 2.12.25).—Citrus fruit is protected from blue mould and other forms of decay by immersion in a solution of soap and borax.

F. R. ENNOS.

Food product. H. F. WEISS, Assr. to WOOD CONVERSION Co. (U.S.P. 1,695,445, 18.12.28. Appl., 20.4.18).—A stock food consists of the concentrated products of hydrolysis of Western larch wood, including galactose, along with finely-divided, hydrolysed larch wood residue.

W. J. BOYD.

Drying of long-fibred substances, such as corn. TROCKNUNGS-ANLAGEN GES.M.B.H. (B.P. 298,148, 13.1.28. Ger., 4.10.27).

Manufacture of beverages comprising a mixture of milk and cocoa or a cocoa preparation. C. E. NORTH (B.P. 303,187, 27.7.27).

Devices for attaining a uniform temperature throughout the length of chilling rolls of margarine-making machines or the like. A. A. POSTNIKOFF and W. R. A. PICKERSGILL (B.P. 303,191, 20.9.27).

Crystallisation of gums, fondants, sweetmeats, etc. A. G. HULME, BAKER PERKINS, LTD., ANC. ETABL. A. SAVY, JEANJEAN & CIE. (B.P. 303,605, 7.11.27).

Sterilisation and like [heat] treatment of food and like products [put up in containers]. SUTAX, LTD., and J. W. SUTCLIFFE (B.P. 304,389, 27.10.27).

Pasteurisation of liquids (U.S.P. 1,693,034).—See I. Treatment of oil seeds (B.P. 279,122).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Testing some common antiseptics. E. S. ROSE (Amer. J. Pharm., 1929, 101, 52–55).—The antiseptic powers of a number of commercial ointments, liquids, and soaps have been measured by observation of the time taken to kill specially prepared cultures of *Staphylococcus aureus*. Of the soaps, only those which contain active germicides such as mercury salts are antiseptic. Zinc oxide, sulphur, ichthyol, compound resorcinol, and tar ointments are only feebly antiseptic. Among the

solutions used for external application, spirit of camphor, alcohol, bay rum, and aluminium chloride solution are antiseptic, whereas witch hazel, boric acid solution, and saturated solution of magnesium sulphate are not. A 2% aqueous solution of mercurchrome is not antiseptic in accordance with the author's interpretation.

E. H. SHARPLES.

Gravimetric determination of colocynthin in extractum colocynthis. P. RÖZSA (Ber. Ungar. pharm. Ges., 1928, 4, 196–207; Chem. Zentr., 1928, ii, 701).—The extract (1 g.) is washed twice with ether (5 c.c.), dissolved in warm absolute alcohol (20 c.c.), the solution boiled with animal charcoal (2 g.), filtered, and the filtrate evaporated to a syrup. The residue is extracted with chloroform, and the solvent removed; the residue is then dissolved in distilled water (20 c.c.), mixed with barium sulphate (0.5 g.), and filtered; the filtrate is evaporated to dryness and heated at 50–60° to constant weight. The fruit yields 25–30% of extract containing about 6% of colocynthin. A. A. ELDRIDGE.

Evaluation of sweet spirit of nitre. L. VAN ITALLIE, A. J. STEENHAUER, and A. HARMSMA (Pharm. Weekblad, 1929, 66, 15–22).—A critical examination of the chlorate oxidation method of determining the ethyl nitrite content of the pharmaceutical preparation. It is recommended to modify the method by addition of a known quantity of silver nitrate before adding the nitric acid, and titrating the excess of silver after precipitation of the chloride is complete. An iodometric method for determining the excess of potassium chlorate is also found to give accurate results.

S. I. LEVY.

Determination of the ethereal oil of *Folia menthae piperitæ*. HORKHEIMER (Pharm. Ztg., 1929, 74, 45).—Some precautions used in carrying out the distillation and subsequent extraction with pentane are described.

S. I. LEVY.

See also A., Feb., 135, Preparation of colloidal gold solution (KILDUFFE and HERFSOHN). 173, Selective reduction of citral, geraniol, and limonene (ESCOURROU). 192, Tricyclene group (KOMPPA). 195, Indole derivatives (KEIMATSU and others). 200, Microchemical reactions of theobromine and caffeine (WAGENAAR). Lupinine (KARRER and others). 201, Alkaloids from *Carnegie gigantea* (HEYL). Synthesis of bases of berberine type (SPÄTH and KRUTA). Strychnine and brucine (CHUSA). Micro-crystallographic identification of yohimbine (DENIGÈS). Alkaloids of Indian aconites (SHARP). 202, Heterocyclic arsenic compounds (STICKINGS; PHILLIPS). 215, Determination of morphine in biological material (BALLS and WOLFF). 221, Examination of insulin for vitamin-B (STUCKY). 222, Activation of ergosterol (BILLS and others).

PATENTS.

Manufacture of solutions of the active principle of the ovary or like glands. I. G. FARBENIND. A.-G. (B.P. 279,123, 17.10.27. Ger., 15.10.26).—Ovarian extract, obtained by saponification of the fats and cholesterol ester of ovaries, is treated with water and

extracted with ether. The residue after evaporation of the ether is dissolved in a water-soluble, nitrogen-free ether or ester of a polyhydric alcohol (diethylin, diacetin), and any cholesterol present is precipitated by addition of water.
B. FULLMAN.

Production of alkylresorcinols. A. R. L. DOHME, ASSR. to SHARP & DOHME, INC. (U.S.P. 1,697,168, 1.1.29. Appl., 16.4.25. Renewed 31.10.28).—See B.P. 250,892; B., 1927, 156.

Manufacture of double salts of carboxylic acids of aromatic sulphonic halogen-alkali-amides. F. PROSCHKO, ASSR. to ESSEFF CHEM. IND.-U. HANDELS-A.-G. (U.S.P. 1,697,139, 1.1.29. Appl., 16.1.26. Austr., 25.2.25).—See B.P. 248,339; B., 1927, 59.

Pharmaceutical products. W. SCHOELLER, A. FELDT, M. GEHRKE, and E. BORWARDT, ASSRS. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,685,341—2, 25.9.28. Appl., [A] 9.5.25, [B] 1.6.26. Ger., [A, B] 2.6.24).—See B., 1925, 692.

Purification of camphor (U.S.P. 1,693,243).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Dye sensitisers and related dyestuffs in the bleaching-out process. M. MUDROVČIČ (Z. wiss. Phot., 1928, 26, 171—192).—The influence of a large number of organic compounds on the bleaching by light of dyes of the thiazine, oxazine, axine, phthalein, and triphenyl-methane series in aqueous and alcoholic solution on paper and in gelatin and collodion has been studied.

H. F. GILLBE.

Mercuric cyanide as desensitiser. H. MEYER and R. WALTER (Z. wiss. Phot., 1928, 26, 165—170).—Mercuric cyanide may be employed as a desensitiser by addition to any ordinary organic developer, with the exception of pyrogallol. The mercury exists in the developer in the form of an alkali-mercuric cyanide complex which is not reducible by the developing agent, but exerts an oxidising influence on the colloidal silver; to this influence the desensitising action is to be ascribed.

H. F. GILLBE.

Metallic lustre tinting effects on gelatin photochemically mordanted. A. REYCHLER (Bull. Soc. chim. Belg., 1928, 37, 403—408).—A gelatin plate (thickness of gelatin, 0.0026 cm., of d 1.36), sensitised by treatment with potassium dichromate and ammonium carbonate, is exposed to light under a silver negative, which leaves a plate mordanted with chromic oxide. If this is immersed in an acid dye (eosin or acid-violet 3BN), a coloured positive is obtained which, if subsequently treated with a basic dye (malachite-green, fuschin, or crystal-violet, which by direct action on the plate would give a coloured negative), gives, after washing, a negative image of metallic lustre by reflected light, but showing only a coloured positive by transmitted light. The best results are obtained with the following pairs of dyes, eosin-malachite-green, eosin-

crystal-violet (0.05% solutions), or acid-violet (0.06%)-crystal-violet (0.04%). With dyes which are only slightly attacked by dichromate the process may be simplified and modified as follows: the plate is sensitised with the usual dichromate-carbonate mixture to which 0.075—0.125% of eosin has been added. Exposure for a short period under a negative yields (after washing) a positive slightly tinted with eosin. If this is well washed with very dilute ammonia and finally with very dilute hydrochloric acid (1 drop of *N*-acid/50 c.c.), dried, and treated with malachite-green, a positive image which gives a metallic negative by reflected light is obtained. If, on the other hand, the washing with ammonia is omitted and, at the right moment, the dried plate is treated with a neutral bath of malachite-green, it yields a metallic positive. These metallic images are formed by the interaction of the two dyes, and a similar compound (which like the image is soluble in alcohol) is obtained *in vitro* from the two dye solutions. The theory of the formation of these images is discussed.
J. W. BAKER.

Selenium toning processes. A. SEDLACZEK (Brit. J. Phot., 1928, 75, 784—785; 1929, 76, 4—6, 29—31, 44—46).—A brief review of the chemical properties of the chief selenium compounds, and a complete summary of the literature dealing with the many toning processes employing selenium and tellurium.
J. W. GLASSETT.

Determination of the ripened nucleus silver in photographic emulsions. II. KIESER (Z. Elektrochem., 1929, 35, 51—53).—The work of Weigert and Lühr (B., 1929, 73) is discussed, and it is contended that although the "primary" silver postulated by these authors probably does exist in the silver halide, it was not actually determined by the method used by them. Evidence is adduced that the substance determined was in reality silver sulphide. The author's method of determining silver in medium-free silver bromide is described (cf. A., 1928, 1339), and the presence of silver sulphide in the precipitate is ascribed to the adsorption of sodium silver thiosulphate on the silver sol, which accelerates the decomposition of the thiosulphate. It is maintained that so far no satisfactory method has been devised for the determination of small amounts of silver in silver halides.
L. L. BIRCUMSHAW.

See also A., Feb., 135, Highly sensitive negative emulsions (KIESER).

PATENTS.

Manufacture of photographic anti-halation plates or films. I. G. FARBERIND. A.-G. (B.P. 292,990, 9.3.28. Ger., 29.6.27).—A backing, which readily strips off in the developer as a coherent skin, may be prepared by coating the rear side of the support for the sensitive emulsion with a mixture made by adding to an alkaline casein solution formaldehyde and a colouring material such as acid black 2B, water-soluble nigrosin D, wool black 4B, or lamp-black.
J. W. GLASSETT.

Reproduction of images on photographic films bearing microscopic refractive elements. Soc.

CIVILE POUR L'ÉTUDE DE LA PHOTOGRAPHIE ET DE LA CINÉMATOGRAPHIE EN COULEURS (B.P. 288,290, 30.3.28. Fr., 8.4.27).—Means are described to avoid the "watering" marks obtained during the reproduction of reticulated films on similar film blanks. J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Condenser chronograph [for explosives]. H. RUMPF (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 13—17).—A method for determining detonation velocities in short lengths of explosive (1—6 cm.) is described in which the arrival of the detonation wave at a certain point of the length of explosive stops the charging of a condenser from a battery. The condenser then discharges through a resistance till the detonation wave reaches a second point in the explosive, when the discharge is stopped. The residual charge of the condenser is then measured, and from it the time taken for the wave to travel between the two points in the explosive can be found. The error in the detonation velocity does not exceed 2% by this method when the distance between the two points in the explosive is known to an accuracy of 1 mm. Experiments on instantaneous fuse gave larger values for the detonation velocity per m. when measured over 5—6 cm. than when measured over 1—2 cm. S. BINNING.

Apparatus for stability tests of smokeless powder at 135° and 120°. J. VON MEERSCHIEDT-HÜLLESSEM (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 10—13).—The apparatus consists of a covered copper bath fitted with a reflux condenser. To the underside of the cover are soldered a number of metal tubes closed at the bottom but open at the top. The test tubes containing the explosives under examination are inserted in these metal tubes, and a device is provided for removing them simultaneously for examination. The heating medium in the bath is an aqueous solution of glycerin, which has the advantage of being unflammable. Temperatures of 135° and 120° are obtained by taking solutions having d_{21}^{20} 1.24 and 1.20, respectively. S. BINNING.

Determination of the Stauchprobe of explosives. K. F. MEYER (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 6—10).—Comparison between the methods of Kast and Hess showed that the latter method had certain advantages chiefly in ease of operation. By both methods the amount of compression of the metal cylinder varied with the position of the initiator in the column of explosive under test, being greatest when the initiator was placed in the explosive as far away as possible from the cylinder. By analogy, the primer cartridge in a borehole charge should be at the mouth of the borehole. Experiments with various types of explosive lead to the generalisation that the value of the Stauchprobe in comparing explosives is limited, but when used with explosives of the same type this test gives useful results as a higher compression always implies greater brisance. No such deduction can be made when the explosives tested are of differing types. S. BINNING.

See also A., Feb., 154, **Changes in nitrocellulose when exposed to light** (COFMAN and DE VORE).

Detection of diphenylamine in Powder B. DESVERGNES.—See III.

PATENTS.

Explosive shells. O. MATTER (B.P. 303,975, 25.11.27).—The shell contains a bursting-charge of a compressed brisant explosive, *e.g.*, pentaerythritol tetranitrate, tetryl, etc., and an initiating charge of compressed lead azide or other metallic azide over a compressed brisant explosive, *e.g.*, pentaerythritol tetranitrate. In small-calibre shells the bursting-charge may be initiated by a percussion fuse containing a compressed metallic azide. S. BINNING.

Explosive priming mixtures. REMINGTON ARMS Co., INC., Assees. of J. E. BURNS (B.P. 287,517, 10.2.28. U.S., 23.3.27).—A non-corrosive priming mixture for ammunition contains normal lead styphnate, mercury fulminate, a non-corrosive oxidising ingredient (barium nitrate), and a combustible (antimony sulphide). Lead chromate or the nitrates of lead or potassium may replace the barium nitrate, and the thiocyanates of lead and copper the antimony sulphide. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Digesting sewage sludge at its optimum p_H and temperature. C. E. KEEFER and H. KRATZ, JUN. (Eng. News-Rec., 1929, 102, 103—105).—A series of samples of fresh sludge (p_H 6.4—8.4) were kept at 28° and the rate of digestion was determined by measurement of the decrease in volatile solid content and of the gas evolved. It is concluded that with p_H 6.8—7.6 digestion is complete in about 30 days, and that about 450 c.c. of methane per g. of fresh volatile solid are evolved. The p_H values were adjusted with lime or hydrochloric acid. Unadjusted sludge (p_H 5.0) was still incompletely digested after 163 days.

C. IRWIN.

Health hazards in chromium plating. J. J. BLOOMFIELD and W. BLUM (U.S. Pub. Health Rep., 1928, 43, 2330—2351).—An atmosphere containing more than 1 mg. of chromium trioxide in 10 m.³ is injurious; injury can be decreased by use of solutions of sodium thiosulphate or hydrogen sulphite, or ammonium polysulphide, and application of oils. Methods of ventilation are described.

CHEMICAL ABSTRACTS.

See also A., Feb., 165, **Determination of thallium [in rat-poisons]** (STICH).

PATENTS.

Treatment of water or sewage. G. ORNSTEIN (B.P. 300,898, 30.10.28. Ger., 19.11.27).—An arrangement is described by means of which chlorine may be dissolved in water and used directly at a predetermined rate, or the chlorine water may be led into a vessel containing broken pieces of metal and the metallic salt thus produced added to the water or sewage, or, again, the two reagents may be applied together at varying rates. C. JEPSON.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 29, 1929.

I.—GENERAL; PLANT; MACHINERY.

Surface increase in mists from the "Schlick" spray. ANON. (Chem.-Ztg., 1929, 53, 33).—The average size of particle, the volume of liquid handled per minute, the number of single particles per litre of liquid handled, and the total surface of liquid in the mist are given for pressures of 2, 4, and 6 atm. With a pressure of 8 atm. the mist has a total particle surface 1700 times the surface of the liquid fed in.

S. I. LEVY.

Removal of thin films of solution in centrifugals. H. v. WARTENBURG and H. PERTZEL [with F. SAVAAGE] (Chem. Fabr., 1929, 61—63).—The thickness of a film of liquid on a vertical flat surface, originally D_0 , is shown to diminish in a time, t , according to $D = \sqrt{hD_0^2/(2D_0^2 \cdot \rho g t + h)/3\eta}$, where h is the height and η the viscosity of the film. The formula is compared with actual determination of mother-liquor content in Glauber's salts during centrifuging, the variation being principally due to the effect of capillarity. A more exact comparison was obtained by the use of a number of brass tubes wetted with castor oil, olive oil, and paraffin oil of known viscosity. The discharged and residual oil were weighed and g in the formula was replaced by the centrifugal force. Whilst the results confirm theory to the extent that the film thickness is inversely proportional to the square root of the time of centrifuging, agreement is far from exact. For stationary films and slow-running centrifugals capillarity is important.

C. IRWIN.

Correlation of freezing points and vapour pressures of aqueous solutions by Dühring's rule. C. C. MONRAD (Ind. Eng. Chem., 1929, 21, 139—142).—Since Dühring's rule holds good for aqueous solutions if two points on the vapour-pressure curve are known, the entire curve may be calculated and the f.p. and b.p. of the solution deduced. Temperature differences between the Dühring line of the solution and that of pure water are transferred to the pressure-temperature phase diagram of water. The accuracy of Dühring's rule for low vapour pressures was checked by reference to a large number of data in literature. This method, however, is not applicable to concentrations higher than that existing at the cryohydric point. In such cases the vapour pressures of saturated solutions must be known. If the vapour pressure of the solution at any other temperature is known, a system of parallel Dühring lines may be drawn.

C. IRWIN.

Comparator for the colorimetric determination of the hydrogen-ion concentrations of coloured solutions. D. MCCANDLISH and G. HAGUES (J. Inst. Brew., 1929, 35, 66—68).—Reflected by means of two

mirrors from the same source of illumination, two beams of light of equal intensity emerge at right angles to each other after passing through the coloured solutions. To render the colour matching more delicate and accurate, the beams of light are then brought parallel and adjacent by means of a pair of right-angled prisms cemented together by Canada balsam, so that the balsam junction extends over only one half of the vertical contiguous surfaces, and there is an air gap between them over the other half.

C. RANKEN.

PATENTS.

Crushing machines. D. B. COOPER (B.P. 302,135, 22.2.28).—A bearing block for the rollers of sugar-cane mills and other purposes is described.

B. M. VENABLES.

Catalytic apparatus. A. O. JAEGER, Assr. to SELDEN CO. (U.S.P. 1,685,672, 25.9.28. Appl., 27.10.26).—The central cooling tubes are spaced closer than the peripheral elements or the velocity of the cooling gas or liquid in the cooling tubes is varied, e.g., by means of plugs, so as to secure maximum cooling in the central elements and relatively uniform temperature in the catalyst layer. With horizontal tubes, similar arrangements secure a maximum cooling effect in the upper catalyst layer, first in contact with the reaction gas. Where one or more reaction components are solid or liquid at ordinary temperatures, or where inert vapours may be present, the latent heat of vaporisation may be utilised for cooling purposes.

R. BRIGHTMAN.

Methods and apparatus for catalysing gaseous reactions. H. WADE. From SILICA GEL CORP. (B.P. 304,251 and 304,269, 12.7.27).—A finely-divided catalyst consisting of adsorbent silica gel etc., together with activating material, if desired, is sprayed into a stream of gases before their entry into a reaction chamber, and the catalyst is subsequently removed from the reaction products, passed through a regenerator, and returned to the gas stream. The process is described with reference to (A) the cracking of oil vapour using silica gel activated with iron oxide etc., and (B) the production of sulphur trioxide and sulphuric acid, in which case the catalyst is activated with platinum, copper, iron, etc.

L. A. COLES.

Gas-analysis apparatus. R. H. KRUEGER, Assr. to C. ENGELHARD, INC. (U.S.P. 1,698,887, 15.1.29. Appl. 27.8.27).—A resistance heating element is mounted in a thermal conductivity cell having walls adapted for dissipating heat, and adjustable means are provided for varying their heat-dissipating capacity.

J. S. G. THOMAS.

Apparatus for production of dispersions of solids in liquids. W. H. WHATMOUGH, Assr. to

STANDARD PRODUCTS CORP. (U.S.P. 1,698,354, 8.1.29. Appl., 16.8.28. U.K., 29.4.27).—See B.P. 293,510; B., 1928, 658.

Centrifugal separator. W. C. LAUGHLIN, Assr. to LAUGHLIN FILTER CORP. (U.S.P. 1,699,471, 15.1.29. Appl., 20.1.27).—See B.P. 283,918; B., 1928, 320.

Metal grinding balls (B.P. 277,044).—See X. Temperature-measuring device (U.S.P. 1,695,867). Gas-purifying plant (B.P. 278,710). Gas-purification electrode (G.P. 451,041).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Maceration method in microscopical examination of coal. H. BODE (Berg-Technik, 1928, 21, 205; Fuel, 1929, 8, 86—90).—In this method the coal is prepared for microscopical examination by treatment with a solution of potassium chlorate in nitric acid (Schulze's solution) and subsequently with ammonia. Lignin and the coalification products of lignin and cellulose are thereby oxidised and removed. Cellulose itself, if present, and the bituminous substances of the coal are unattacked. From the amount and appearance of the residue, *e.g.*, from the occurrence therein of fossil wood, spores, cuticle, etc., it is possible to determine the degree of coalification of the material, to distinguish vitrain, durain, and fusain in powdered coal, and to determine the nature of the plants which contributed to the formation of the coal. The method is preferable to that of the observation of etched polished surfaces in reflected light, which can lead to false conclusions. A. B. MANNING.

Spontaneous combustion of semi-coke from brown coal: its causes and prevention. P. ROSIN (Braunkohle, 1928, 27, 241—256, 282—292; Fuel, 1929, 8, 66—78).—The influence of various factors, such as moisture content, temperature of carbonisation, fineness of subdivision, the addition of catalysts, etc., on the spontaneous heating of a brown coal semi-coke has been studied. The apparatus consisted of an insulated metal cylinder capable of holding about 11 kg. of material, through which a current of oxygen could be passed, while observations were made of the temperature at several points within the material. Measurements have been made also of the adsorption of gases by the semi-coke and of the heat developed on wetting it. The material used was taken directly from the retort and cooled in the absence of air. Self-heating did not occur uniformly within the material; local centres of heating were formed showing a more rapid rise of temperature than elsewhere. The first gradual rise of temperature, due to adsorption of oxygen, was followed at about 70° by a phase of rapid oxidation resulting in combustion. The adsorption of all gases by the semi-coke was accompanied by the evolution of heat, that evolved on the adsorption of carbon dioxide being sufficient to raise the temperature of the coke to 70°. This carbon dioxide is lost on subsequent exposure of the coke to the air, the heat thereby absorbed compensating to some extent for that evolved due to oxidation. Moist semi-coke "adsorbs" more oxygen than dry coke, due probably to increased oxidation rather than to true adsorption. The heat evolved when the semi-coke is wetted may also

result in a dangerous rise of temperature. Addition of moisture promotes the initial heating of the coke, and, although it retards spontaneous combustion, wetting alone is not a sure means of preventing such combustion. Spontaneous heating of the semi-coke can be prevented or much diminished by artificial ageing, treatment with carbon dioxide, or the addition of magnesium or ammonium chloride to the coal. The coke also loses all liability to self-heating with the removal of the inorganic constituents soluble in hydrochloric acid. Of the inorganic substances the addition of which promoted self-heating, potassium carbonate had the most marked action, but no correlation could be found between the potassium content of a coke and its liability to spontaneous combustion. The addition of marcassite, iron sulphide, or calcium sulphide had no marked influence on the spontaneous heating of the semi-coke.

A. B. MANNING.

Influence of inorganic materials in lignite carbonisation. A. W. GAUGER and D. J. SALLEY (Fuel, 1929, 8, 79—85).—The effect of the addition of 5% of various inorganic constituents on the character and yields of the products from the carbonisation of a North Dakota lignite has been studied. 150—225 g. of the material were briquetted and carbonised either at 510° in an aluminium retort as used by Fischer, or at 850° in a copper retort of similar construction. The addition of the hydrated salts of aluminium, magnesium, or strontium alters the structure of the char produced, yielding a hard pseudo-coke differing materially from the soft powdered char of the untreated coal, whereas addition of the anhydrous salts produces soft, fine chars. The addition of hydrated ferric chloride increases the yield of char at the expense of the tar. The addition of sodium carbonate decreases the yields of char and tar and increases the gas yield. Addition of silica has no effect.

A. B. MANNING.

Briquetting of coal dust without the use of binding materials. W. SWIENTOSLAWSKI, B. ROGA, and M. CHORAŻY (Przemysł Chem., 1929, 13, 2—12, 40—49).—A well-defined point of maximum plasticity is found for coking but not for non-coking coals; in no case is this point attained below 400° or above 480°. The resistance to erosion or mechanical shock of briquettes made of coking coal is small compared with that of ordinary briquettes; the greatest strength is obtained if briquetting is carried out at such a temperature that the surface of fracture has a vitreous appearance (about 450°). Non-coking coals give much the stronger briquettes, the optimum temperature in this case being 405—415°, the time of heating 30—40 min., and the pressure 400—600 kg./cm.², or twice that used ordinarily. The best results are obtained when mixtures containing under 30% of coking coal are used. Inferior briquettes are yielded by coal dust the grains of which are <0.02 mm. or >3 mm. in diam. Briquettes so prepared are slightly more resistant to erosion, but not to shock, than those made with the use of binding material; their ignition point is more than 100° lower than that of ordinary briquettes; they burn with the production of less smoke; and their calorific value is higher than that of the original coal, owing to their low water content.

R. TRUSZKOWSKI.

Adsorption of pyridine vapour by coal from Upper Silesia and by its petrographic modifications. W. SWIENTOSLAWSKI and M. CHORAZY (Bull. Acad. Polonaise, 1928, A, 361—365).—Vitrain, durain, and fusain separated from the coal of Upper Silesia absorb in 26 days 70.4%, 54.5%, and 12.8%, respectively, of their dry weight of pyridine vapour. The greater is the coking power of the coals, the greater are the amounts of pyridine absorbed. W. E. DOWNEY.

Chemical composition of peat. II. Chemical composition of various peat profiles. S. A. WAKSMAN and K. R. STEVENS (Soil Sci., 1928, 26, 239—251).—Using the methods previously described (B., 1928, 880), a study was made of peats from a number of different sources. The chief interest lies in the comparison of high- and low-moor peats, which, by reason of their different floral origin, differ distinctively in the products and manner of their decomposition and serve to illustrate modifications of the general scheme of humification of organic matter. H. J. G. HINES.

Humic acids. G. STADNIKOV and P. KORSCHEV (Kolloid-Z., 1929, 47, 136—141).—The course of the adsorption of barium hydroxide by humic acid has been followed by titration with hydrochloric acid. Under the experimental conditions, marked adsorption occurred during the first 4 or 5 hrs. and thereafter slowly, not reaching a final value after 120 hrs. The amount of barium hydroxide adsorbed does not depend on the concentration of the solution, thus resembling the result obtained by Kawamura (A., 1926, 1201) using stearic acid, but differing from that obtained by this author with artificial humic acid obtained from sugar. The results indicate a value of 147 for the equivalent weight of humic acid. Experiments have also been performed on the reactions of barium humate with the chlorides and nitrates of sodium and potassium. These reactions take place slowly, reaching equilibrium after about six days. During the reaction the concentration of the soluble humate varies proportionally with the concentration of the alkali salt in the solution; the concentration of the barium salt in solution varies in a complicated way, which is expressed by a curve very similar to the ordinary adsorption curve. An explanation of these facts is attempted. E. S. HEDGES.

Methods of heating coke ovens. E. WEISE (Gas-u. Wasserfach, 1929, 72, 73—78).—The calculated temperatures obtainable by burning coke-oven gas, blast-furnace gas, and mixtures of the two with different degrees of preheating of gas and air and different excesses of air are tabulated, with and without consideration of dissociation within the gases at high temperatures. The total and useful heat contents per cub. m. of the heating gases, together with the available heat per cub. m. of the flue gases, are calculated; also the cost per cub. m. of coke-oven gas under different combustion conditions is compared with that of a standard coal giving an equal thermal efficiency when burnt under standard conditions. It is estimated that, with coke-oven gas, mixed gas I (1 vol. of coke-oven gas : 1 vol. of blast-furnace gas), mixed gas II (1 vol. of coke-oven gas : 3 vols. of blast-furnace gas), and blast-furnace gas (net cal. values 4336, 2620, 1763, and 904 kg.-cal./cub.m.,

respectively), the relative volumes of gas required to yield a given quantity of available heat are as 1 : 1.76 : 2.26 : 3.98.

W. T. K. BRAUNHOLTZ.

Automatic combustion control. T. A. PEEBLES (J. Inst. Fuel, 1929, 2, 131—140).—The difficulties of the efficient working of fuel-burning equipment, together with descriptions of automatically controlled plants using steam coal, pulverised coal, and blast-furnace gases, are given. C. B. MARSON.

Determination of the sulphur content of gases, especially of coke-oven gas. F. HEINRICH and F. PETZOLD [with E. SCHUTH] (Z. anal. Chem., 1929, 76, 120—134).—The gas is passed at the rate of 0.2 litre/min. through a solution consisting of 180 c.c. of 0.5N-sodium hydroxide and 90 c.c. of 5% hydrogen peroxide solution distributed between two absorption flasks; the initial and final volume of the gas being known, the sulphur content may be determined by titration of the solution with 0.5N-sulphuric acid. The method is applicable to gases containing sulphur in any inorganic or organic compound with the exception of thiophen. H. F. GILLBE.

Hydrocarbons in Peruvian petroleum having b.p. below 150°. W. F. SEYER and A. F. REES (Trans. Roy. Soc. Canada, 1928, [iii], 22, III, 359—360; cf. B., 1926, 653).—The oil used (d_{20}^{20} 0.8340) was from the Lobitos Oil Field, in Peru. The distillate boiling below 175° was collected and fractionated. The physical properties indicate the presence of the hydrocarbons C_6H_{14} , C_7H_{14} , C_8H_{16} , and C_9H_{18} . A. J. MEE.

Ultramicroscopy of asphalts and allied products. F. J. NELLENSTEYN and J. P. KUIPERS (Kolloid-Z., 1929, 47, 155—160).—Former ultramicroscopical investigations on solutions of asphalt and of coal tar (B., 1928, 324) have been repeated and the results confirmed. The observation of other investigators, that solutions of artificial asphalts do not show the presence of ultramicros, cannot be substantiated; the difference is merely that natural asphalts give a better ultramicroscopical effect at higher dilutions. Under the ultramicroscope a mixture of artificial asphalt with a finely-divided inorganic powder produces the same effect as natural asphalt. When mixed with soot, solutions of asphalt in nitrobenzene or aniline resemble those of coal tar, both microscopically and ultramicroscopically. E. S. HEDGES.

Characterisation and classification of gasolines. T. NOWOSIELSKI (Przemysl Chem., 1929, 13, 16—23, 66—78).—The average b.p. of gasolines is a much more instructive feature of a given sample than is its sp. gr., which gives little indication of the volatility. For practical purposes, the average b.p., the percentage distilled over at this temperature, and the b.p. range of a given gasoline suffice to characterise it. The average b.p. of a mixture of gasolines can be calculated with a mean error of 1.5° from those of the individual components, on the basis of the rule of mixtures.

R. TRUSZKOWSKI.

Fuels—alternative or supplementary to petrol—for use in internal-combustion engines for road vehicles. I. Liquid fuels. W. R. ORMANDY (J.

Inst. Fuel, 1929, 2, 199—204).—The production and economic value of such liquid fuels from high- and low-temperature distillations of coal and from lignite, oil shales, hydrogenation of lignites or bituminous coals, and treatment of water-gas by catalysts are discussed, together with the manufacture and advantages of alcohol.

C. B. MARSON.

Voltol. K. WOLF (Petroleum, 1929, 25, 95—98).—Lubricating oils are produced by subjecting mineral oils, tar, fatty oils, or mixtures of mineral and fatty oils to a glowing electrical discharge, alternating current at 5000 volts being employed. The process is carried out at about 80° and in a vacuum of 60—70 mm., using aluminium electrodes.

W. S. NORRIS.

Standardisation of oil testing. M. FRIEDEBACH (Petroleum, 1929, 25, 93—95).—The variation in flash point observed with the thermometer in different positions in the sample of oil is not greater than the experimental error (cf. Schlüter, B., 1928, 325). The thermometer is best placed towards the centre of the oil, but a precise mechanism for locating it is a needless complication. Rate of heating, location of the ignition flame so that local heating does not occur, and constancy in the length of the flame are more important factors. A simple flash-point apparatus is described.

W. S. NORRIS.

"Tar value" of lubricating oils. S. GASIOROWSKI and S. PILAT (Przemysl Chem., 1928, 12, 235—239; Chem. Zentr., 1928, ii, 613—614).

Humic acids. Pyrohymatomelanic acid. D. J. W. KREULEN (Chem. Weekblad, 1929, 26, 101—103).—See B., 1928, 555.

Paint resistant to flue gas. VAN WULLEN-SCHOLTEN. —See XIII.

PATENTS.

Vertical rotary retort for low-temperature carbonisation. G. POLYSIUS (G.P. 452,620, 25.9.24).—The retort comprises an outer drum and an inner concentric cylinder of perforated metal, the material to be carbonised occupying the annular space between the cylinders. The walls of the drums are made smooth in order to facilitate the discharge of the carbonised material.

A. B. MANNING.

Production of liquid hydrocarbons by the carbonisation and hydrogenation of carbonaceous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,343, 17.10.27).—In processes in which carbonaceous materials are heated in the presence of gases or vapours for the production of low-boiling hydrocarbons, *e.g.*, in the destructive hydrogenation of coal, tar, etc., increased yields are obtained by giving the gases in the reaction vessel a rapid oscillating movement or by re-passing them repeatedly through the chamber, so that their rate of flow therein is increased. At the same time the partial pressures of the substances under treatment are kept as low as possible.

A. B. MANNING.

Production of hydrocarbons from coal and water. A. UHLMANN (B.P. 283,177, 5.1.28. Ger., 6.1.27).—Finely-divided coal or other carbonaceous material is mixed to a pulp with water and is driven through a pipe in which it is subjected to the action of a

high-frequency field produced preferably by a pulsating continuous current. Locally produced high temperatures ("micro-carbon arcs") decompose the water and bring about hydrogenation of the coal with the production of hydrocarbons.

A. B. MANNING.

Working-up the oil-bearing residues of destructive hydrogenation of carbonaceous materials or the products obtained by extraction of substances of the nature of coal. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,404, 5.11.27).—Oil-bearing residues are mixed with finely-divided carbonaceous material, *e.g.*, coal dust, wood flour, etc., and are subjected to low-temperature carbonisation.

A. B. MANNING.

Separation of hydrocarbon oils from mixtures thereof with solid substances resulting from industrial processes in which carbonaceous materials are subjected to a heat-treatment. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,941, 20.6., 3.10., and 29.12.27).—The mixture of hydrocarbon oils and solids, *e.g.*, from low-temperature carbonisation of coal, destructive hydrogenation, or cracking operations, is stirred with an aqueous liquid containing a wetting or emulsifying agent, at raised temperature if desired, and the whole is centrifuged. With light oils, an aqueous layer is formed between the solids and the oil; heavy oils form the middle layer. Salt solutions, *e.g.*, 40—50% aqueous calcium chloride, may be used, and in some cases addition of alkali is advantageous. Alternatively, the oils may be emulsified in water, the emulsion separated from the solids, and the oils recovered by destroying the emulsion. Oxidation products of paraffin wax are suitable emulsifying agents in alkaline solution.

C. HOLLINS.

Working-up of vegetable waste. GES. F. LUPINEN-IND. M.B.H. (G.P. 452,810, 12.4.25).—Vegetable waste of high potash and phosphoric acid content is carbonised and briquetted in order to form a fuel suitable for use in a gas generator. The potassium salts and phosphates can then be recovered from the ash by extraction.

A. B. MANNING.

Agglomerated fuel. SOC. FRANÇ. DES CHARBONNAGES D'ALONG ET DONG-DANG (F.P. 629,394, 27.4.26).—Coal dust is impregnated with a solution obtained by treating vegetable tissue with alkalis or reducing agents under pressure at various temperatures, and the mixture is then briquetted and dried.

A. B. MANNING.

Compound fuel. P. H. LÉVÊQUE, Assr. to SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (U.S.P. 1,693,790, 4.12.28. Appl., 4.10.27. Ger., 11.10.26. Renewed, 12.10.28).—Anthracite is mixed with coal which has been purified by the Trent or similar oil process. The mixture is compressed, the oil acting as a binder, and then distilled.

F. G. CLARKE.

Treatment of gases arising in the distillation or carbonisation of coal and like carboniferous material. C. COOPER, D. M. HENSHAW, and W. C. HOLMES & Co. (B.P. 304,333, 15.7.27).—The crude hot gases from the carbonisation of coal and similar processes are brought into contact with salt solutions which act as a cooling medium, and are of such composition that they

will take up water from the gas but not ammonia. Their sp. gr. is sufficiently high that any insoluble constituents which are condensed will float on the surface. Solutions of ammonium salts, alone or with other salts, are suitable for the purpose. The diluted solution is reconcentrated by exposure to the atmosphere, the evaporative effect being increased by bringing the solution into direct or indirect contact with the hot gases. A. B. MANNING.

Treatment of coal-distillation gases. IMPERIAL CHEM. INDUSTRIES, LTD., K. GORDON, and J. HUGHES (B.P. 304,345, 17.10.27).—The principal constituents of coke-oven and similar gases, from which the tar, ammonia, and benzol have been removed, are separated by washing the gas under pressures of 100–250 atm. (a) with strong ammonia liquor which removes carbon dioxide, hydrogen sulphide, etc., (b) with an ammoniacal solution of a cuprous salt for the removal of carbon monoxide and unsaturated hydrocarbons (principally ethylene), and (c) with kerosene for the absorption of methane (cf. B.P. 293,138; B., 1928, 670). The liquor produced in the first washing may be used for the preparation of ammonium sulphate by interaction with calcium sulphate. The gases released from the ammoniacal cuprous solution may be washed with silver nitrate solution in order to separate the ethylene from the carbon monoxide (cf. B.P. 291,186; B., 1928, 560), or these gases may be released separately from the solution by suitable adjustment of the pressure and temperature. The methane recovered from the kerosene washing may be used for the production of acetylene or acetic acid. A. B. MANNING.

Removal of dust from low-temperature gases. KÖHLENVEREDLUNG A.-G. (G.P. 452,349, 9.5.22).—The gases, which pass from the retort through a dust separator to the condensing system, are heated before entering the separator, the amount of heat given to them corresponding with the heat subsequently lost in the separator. A. B. MANNING.

Purification of gases from organically combined sulphur. F. FISCHER (B.P. 282,634, 25.10.27. Ger., 24.12.26).—The organic sulphur compounds in gases which also contain hydrogen are converted into hydrogen sulphide by passage of the gas over finely-divided silver, or other precious metal of the first periodic group, at 200–300°. The hydrogen sulphide is subsequently removed in the usual manner. A. B. MANNING.

Removal of sulphur compounds from gases. F. NEUWIRTH (Aust. P. 107,600, 30.6.26).—The gases are passed through one or more layers of lignite. A. B. MANNING.

Water-gas producer. KÖRTING & AHRENS G.M.B.H. (G.P. 452,401, 22.4.25).—The producer is surrounded by a fire-tube boiler and is so arranged that the blow gases pass up through an annular space between the producer and the boiler and then down through the tubes of the boiler and a waste pipe to the air. A. B. MANNING.

Continuous production of water-gas. SOC. ANON. DES APPAREILS DE MANUTENTION ET FOURS STEIN (F.P. 629,397, 27.4.26).—Pulverised fuels are treated with steam in an apparatus which is heated externally to a temperature necessary for the production of water-gas. A. B. MANNING.

Treatment of coal tar. K. W. NIELSEN (Dan. P. 35,094, 24.10.25).—The warmed tar is treated with air in the presence of nitric acid or oxides of nitrogen in order to remove the free carbon. A. B. MANNING.

Separation of diphenylene oxide from coal tar. RÜTERSWERKE A.-G., Assees. of R. MÜLLER (G.P. 451,536, 6.12.25).—The fractions containing diphenylene oxide are treated with a dehydrogenating, polymerising, or condensing agent, e.g., oxygen, lead or manganese oxides, halogens, sulphur, aluminium chloride, etc., under suitable conditions of temperature and pressure, with or without the addition of catalysts, and the diphenylene oxide is then recovered from the product by distillation or crystallisation. A. B. MANNING.

Preparation of stable emulsions of coal dust in oil. A. RIEBECK'SCHE MONTANWERKE A.-G. (G.P. 453,465, 2.6.26. Addn. to G.P. 444,420; B., 1928, 81).—Instead of alkali, ammonia or organic bases may be used as stabilising agents. Only phenols or oils rich in creosote are added to oils which already contain organic bases. Pyridine and its derivatives, quinoline, aniline bases, and the crude mixtures of bases from tar can be used. W. J. BOYD.

Fractional distillation of pyroligneous acid. SOC. ANON. DES PROD. CHIM. DE CLAMECY (F.P. 628,785, 22.4.26).—Methyl alcohol, acetone, and methyl acetate are removed from the crude acid by distillation with benzene, carbon disulphide, or a halogenated hydrocarbon, and when the temperature reaches 69° and the low-boiling constituents have been removed the solvent is expelled by a current of steam. The residue is distilled, using a second column, so that water passes over at 98–100°, after which residual volatile impurities are expelled by treatment with steam at 70–97°. The lower aqueous layer separating in the receiver is withdrawn, and the upper oily layer is returned to the column. The residual concentrated acetic acid is freed from tar by extraction or distillation. L. A. COLES.

Pitch-coking process and product. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,694,800, 11.12.28. Appl., 10.11.19).—A mixture of pitch, bauxite, and coke breeze is subjected to coking treatment. L. A. COLES.

Manufacture of impregnating and adhesive compositions. J. Y. JOHNSON. From I. G. FARBEN-IND. A.-G. (B.P. 302,710, 17.9.27).—Oily or fatty pitches, e.g., stearine pitch, are heated with vegetable or animal oils, which may have been oxidised and/or polymerised, and sulphur or substances yielding it, e.g., thionyl chloride, material extracted from a tar and containing sulphur being added before, during, or after the sulphurising treatment. Pitch, asphalt, resins, fillers, etc. may also be added. L. A. COLES.

Operation of pressure stills [for cracking of hydrocarbon oils etc.]. J. E. BELL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,693,946, 4.12.28. Appl., 26.7.24).—Vapours from the still are refluxed in a zone separate from the vaporising zone in direct contact with fresh charging stock, and the reflux and unvaporised charging stock are introduced into the still stock that is to be immediately subjected to high temperature. The vapours pass through a series of condensing

chambers, maintained at progressively lower temperatures by heat-interchange, in direct contact with fresh charging stock passing to the still, and a part of the condensate is refluxed from chambers of lower temperature to those of higher temperature. H. S. GARLICK.

Cracking of oil. R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,688,830, 23.10.28. Appl., 6.12.20. Renewed, 16.7.28).—Vapours from the cracking chamber are dephlegmated in presence of fresh charging oil and water or steam. The reflux is returned to the heating coil after separation of any water.

R. BRIGHTMAN.

Conversion of hydrocarbon oils. O. C. BREWSTER, Assr. to STANDARD OIL Co. (U.S.P. 1,694,092, 4.12.28. Appl., 20.6.25).—Regulated air supply is used to maintain hydrocarbon oil, *e.g.*, crude residue, *d* 0.933—0.939, at conversion (distillation) temperature (380—405°) by internal oxidation under pressure, the feed oil being preheated similarly. Vapours from the still pass through a stripping column in countercurrent heat-exchange with the feed oil, preheated to, *e.g.*, 360—385°, the reflux and fresh oil passing back to the still, while the vapours pass to a condenser and receiver in communication with the uncondensed vapour from the feed preheater.

R. BRIGHTMAN.

Converting [cracking] hydrocarbons into lower-boiling products. F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,694,262, 4.12.28. Appl., 21.5.23).—Relatively clean oil, *e.g.*, light gas oil, is vaporised in a heating coil at 480—540° and passed into a heat-insulated chamber in contact with heavy gas oil or other relatively dirty or coke-forming stock. The liquid and vapour products are then passed by a connexion having a pressure-release valve to a vapour separator, from which the tar is drawn off and the light and heavy vapours are passed to condensers.

R. BRIGHTMAN.

Treating [cracking] hydrocarbons. A. SCHWARZ, Assr. to PETROLEUM SAND PRODUCTS CORP. (U.S.P. 1,691,085, 13.11.28. Appl., 1.3.24).—Heavy hydrocarbon oil is cracked as a vapour-oil emulsion by violent agitation, *e.g.*, 400—600 r.p.m., at cracking temperature (cf. U.S.P. 1,458,443; B., 1923, 918A).

R. BRIGHTMAN.

Fractionation of hydrocarbons. J. C. BLACK (U.S.P. 1,692,072, 20.11.28. Appl., 2.2.24).—Vapours from a continuous still are scrubbed with a "light intermediate" fraction in a condensing chamber, *a*, and passed into a chamber, *b*, from which the lighter vapours ascend through boiling-decks to the condenser, after heat-exchange with the feed to the still. Reflux from *b* is used to scrub the vapours in *a*, any surplus being stored as "light intermediate" or steam-distilled so that the vapours ascend through the boiling decks in *b*, while the reflux is stored as "heavy intermediate." The reflux from *a* is similarly treated, the light vapours ascending through the boiling decks in *b*.

R. BRIGHTMAN.

[Steam] fractionation [of hydrocarbons]. I. B. FUNK, Assr. to UNION OIL Co. OF CALIFORNIA (U.S.P. 1,691,238, 13.11.28. Appl., 30.10.26).—In order to obtain close "cuts" when using fractionating columns

of the type having perforated plates with funnels, which lead the condensed liquid to the plate immediately below, horizontal partitions are placed in the upper part of the column to condense the steam. These partitions have apertures fitted with bubble caps, and the condensed liquids are led into traps outside the column. Vapour and oil are returned to the latter through separate tubes, and the water is removed from the bottom of the traps.

F. G. CLARKE.

Treatment of liquids [petroleum oils]. L. CALDWELL, Assr. to CELITE Co. (U.S.P. 1,691,266, 13.11.28. Appl., 27.1.26).—Petroleum oil is refined by treatment with the solid obtained by chlorinating the reaction product of silica (diatomaceous earth) and excess of calcium hydroxide.

R. BRIGHTMAN.

Removal of petrolatum from oils. C. ZWEIG, Assr. to STANDARD OIL Co. (U.S.P. 1,694,090, 4.12.28. Appl., 21.6.26).—Oil containing petrolatum is diluted and agitated at a low temperature before settling to separate the wax.

H. S. GARLICK.

Production of thickened lubricating oil. R. W. HENRY and E. D. STREETER, Assrs. to GULF REFINING Co. (U.S.P. 1,691,882 and 1,691,654, 13.11.28. Appl., [A, B] 24.3.26. Renewed, [B] 3.10.28).—Basic aluminium oleate, prepared, *e.g.*, (A) by adding an aluminium salt to an emulsion of mineral oil and neutral aqueous salt solution, partly hydrolysing with alkali hydroxide, and adding an aluminium salt to reconvert neutral soap thus formed into aluminium trioleate, is used as thickener. (B) A basic aluminium salt solution, obtained by the addition of sodium hydroxide in presence of sodium citrate or other protective agent, is added to the neutral soap solution.

R. BRIGHTMAN.

Reclaiming [lubricating oil] distillation residues. R. E. POWELL, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,694,280, 4.12.28. Appl., 25.2.25).—Still residues, *e.g.*, from the distillation of lubricating stock containing naphthenic acids in presence of sodium hydroxide, are refluxed at 150—350° with 25% of gas oil and steam injection, and the residue is discharged hot on to the surface of a stream of water. After settling, the oil is separated from the aqueous naphthenate solution, continuously or in stages.

R. BRIGHTMAN.

Manufacture of motor fuel. L. KIRSCHBRAUN (U.S.P. 1,692,176, 20.11.28. Appl., 19.11.19. Renewed 12.5.28).—Fuel for internal-combustion engines is obtained by dispersing water in, *e.g.*, gasoline, with the aid of ammonia solution and phenol or naphthenic acid as emulsifier.

R. BRIGHTMAN.

Fuel and fuel ingredients. J. W. ORELUP and O. I. LEE, Assrs. to BOYCE-ITE PRODUCTS, INC. (U.S.P. 1,692,784, 20.11.28. Appl., 23.1.25).—Addition to petrol of 0.1—3.0% of higher fatty acids or their esters, amides, halogenated derivatives, or metallic salts reduces the deposition of carbon in internal-combustion engines. As example, 0.1% of mixed esters, containing 60% of ethyl laurate, is mixed with an equal volume of *o*-nitrotoluene and added to the petrol.

R. BRIGHTMAN.

Ethyl alcohol substitute and its manufacture.

R. M. ISHAM, ASST. to DOHERTY RES. Co. (U.S.P. 1,692,662, 20.11.28. Appl., 6.5.26).—A mixture with an odour resembling that of ethyl alcohol is obtained from isopropyl alcohol by addition of 3 c.c. of acetaldehyde and 6 c.c. of butyl acetate per 5 gals.

R. BRIGHTMAN.

Manufacture of producer gas from coking coals. K. KOLLER (U.S.P. 1,697,920, 8.1.29. Appl., 18.7.25. Ger., 18.7.24).—See B.P. 254,392; B., 1926, 778.

Obtaining hydrogenation gas from hydrogenating carbon and hydrocarbons from gases containing methane and hydrogen. F. BERGIUS (U.S.P. 1,699,177, 15.1.29. Appl., 30.11.25. Ger., 18.12.24).—See B.P. 244,730; B., 1927, 66.

Separation of paraffinous constituents from fluid hydrocarbons. E. A. FORSBERG, ASST. to AKTIEBOLAGET SEPARATOR-NOBEL (U.S.P. 1,699,286, 15.1.29. Appl., 28.6.27. Swed., 24.8.26).—See B.P. 276,658; B., 1929, 161.

Hydrogenation of tar oil. R. BERNHARD and W. DEMANN (U.S.P. 1,691,221, 13.11.28. Appl., 25.6.25. Ger., 30.6.24).—See B.P. 236,230; B., 1926, 973.

Coal washing apparatus. E. COPPÉE & CIE. (B.P. 288,252, 29.2.28. Belg., 5.4.27).

Gas burners. V. H. RICHARD (B.P. 304,938, 23.2.28).

Catalytic gas reactions (B.P. 304,251).—See I. Olefines and diolefines (B.P. 303,998). Sulphonic acids (B.P. 303,917). Hydrogenation of naphthalene (B.P. 304,403).—See III. Waterproofed materials (B.P. 303,935). Textile oils (B.P. 303,820).—See V. Bituminous emulsions (B.P. 301,544). Paving composition (B.P. 302,808).—See IX.

III.—ORGANIC INTERMEDIATES.

Humic acids. STADNIKOV and KORSCHEV. Sulphur content of gases. HEINRICH and others.—See II. Nitrogen in urea-nitrate mixtures. LUCAS and HIRSCHBERGER.—See XVI. Absolute alcohol. VON KEUSSLER.—See XVIII.

PATENTS.

Manufacture of olefines and diolefines [butadiene]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,998, 14.12.27).—Paraffins, olefines, or alicyclic hydrocarbons are passed in vapour form at 640–720° over lustrous carbon (B., 1923, 1130 A; 1927, 802) deposited on a non-reducible oxide such as magnesia, lime, baryta, quartz, or metals such as chromium; preferably steam is used as a diluent. *cyclo*Hexane at 650° gives 90% of olefines, chiefly butadiene and ethylene; American petroleum or synthetic benzine at 700–720° yields 80% of olefines, of which one fifth is butadiene.

C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 303,772, 7.7.27. Addn. to B.P. 256,663; B., 1926, 897).—The vapours containing the acid (acetic) anhydride are mixed with vapour of an entraining liquid (benzene, carbon tetrachloride, petrol, mixtures of these, or of ether with light petroleum, b.p. 40–70°) at above

100° but below the b.p. of the anhydride. Water is thus carried away with the entraining liquid, and the anhydride is condensed.

C. HOLLINS.

Manufacture of acetic anhydride. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 279,070, 5.10.27. Ger., 18.10.26).—A vaporous catalyst is used, preferably phosphoric acid entrained to the extent of 0.5 g. per kg. of acetic acid vapour at 600°.

C. HOLLINS.

Production of acetone. HOLZVERKOHLUNGS-IND. A.-G. (B.P. 280,184, 24.10.27. Ger., 4.11.26).—Acetylene and steam are passed at 470–500° over a heavy-metal oxide (iron oxide) mixed with a promoter which increases the oxygen tension of the oxide to about the partial pressure of the oxygen in the reacting gases, so that no substantial reduction occurs. Examples are superficially rusted iron shavings, iron shavings coated with iron oxide and manganese oxide or with iron oxide and barium carbonate.

C. HOLLINS.

Production of esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,411, 22.9.27).—Soaps, especially the fatty acid salts of tin, zinc, or magnesium, added in small proportion (0.1%) accelerate the esterification of higher fatty acids with polyhydric alcohols.

C. HOLLINS.

Manufacture of substituted thioglycollic acids. I. G. FARBENIND. A.-G. (B.P. 287,178, 16.3.28. Ger., 18.3.27. Addn. to B.P. 281,290; B., 1928, 921).—The method of the prior patent is applied to the production of trihalogeno-derivatives of benzene and toluene. 1:2:3-Trichlorobenzene, m.p. 53–54°, yields with chlorosulphonic acid at 20–70° solely the 4-sulphonyl chloride, m.p. 65–66°, from which are obtained 2:3:4-trichlorothiophenol, and 2:3:4-trichlorophenylthioglycollic acid, m.p. 149°. 2:3:4-Trichlorotoluene, m.p. 41°, similarly yields the 5-thioglycollic acid, m.p. 157–161°, and 1:2:4-trichlorobenzene, m.p. 17°, yields 2:3:5-trichlorophenylthioglycollic acid.

C. HOLLINS.

Preparation of the lactone of 14-oxy- ω -hydroxy-] tetradecane- α -carboxylic acid. SOC. ANON. M. NAEF & CIE., ASSEES. of M. NAEF & CIE. (B.P. 294,602, 26.6.28. Switz., 27.7.27).—The lactone of ω -hydroxypentadecanoic acid, m.p. 30°, b.p. 175°/11 mm., d_4^{20} 0.9383, n_D^{20} 1.4633, is obtained by heating *cyclopentadecanone* with Caro's acid. It has a musk odour with a flavour of amber. The corresponding hydroxy-acid, m.p. 84°, is oxidised by chromic acid to tridecane- $\alpha\omega$ -dicarboxylic acid, m.p. 112°.

C. HOLLINS.

[Catalyst for] catalytic preparation of oxygenated carbon compounds. E. I. DU PONT DE NEMOURS & Co., ASSEES. of W. A. LAZIER (B.P. 301,806, 13.6.27. U.S., 12.6.26. Cf. B.P. 272,555; B., 1929, 163).—A chromium mixed catalyst is prepared by heating a double chromate of a nitrogenous base and zinc, copper, cadmium, magnesium, manganese, silver, or iron. Catalysts from basic zinc ammonium chromate, zinc tetrapyridine dichromate, and manganese ammonium chromate are described.

C. HOLLINS.

Hydrogenation of open chains containing nitrogen in unsaturated union. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 304,000, 20.12.27).—Unsaturated nitrogen linkings are reduced by treating the

compound in aqueous solution with hydrogen in presence of nickel at 60–80°, an electrolyte being added which converts the reduction product (an amine) into a salt by double decomposition. Thus potassium cyanide is reduced to methylamine by hydrogen and nickel in presence of ferrous sulphate. Acetonitrile gives ethylamine; phenylacetonitrile, β -phenylethylamine; and azobenzene, cyclohexanol and cyclohexylamine.

C. HOLLINS.

Manufacture of aqueous "solutions" of organic compounds insoluble in water. I. G. FARBENIND. A.-G. (B.P. 273,757, 2.7.27. Ger., 2.7.26).—The sulphonic acids of saturated aliphatic hydrocarbons or saturated aliphatic acids, obtained as in B.P. 272,967 or 288,612 (B., 1928, 703, 865), are used for the solubilisation of water-insoluble organic compounds; e.g., 20 pts. of methylcyclohexanol give a clear "solution" in 160 pts. of water to which have been added 70 pts. of a 50% aqueous solution of sulphonated lauric acid, and 8 pts. of *n*-butyl alcohol "dissolve" in 20 pts. of the same reagent neutralised with ammonia. Sulphonated paraffin oil is also effective.

C. HOLLINS.

Production of sulphonic acids [wetting, emulsifying, and cleaning agents]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,917, 12.9.27).—Unsaturated aliphatic hydrocarbons or their halogen derivatives or mixtures containing these, e.g., petroleum, brown coal tar oils, etc., are sulphonated by treatment with sulphuric acid, oleum, or chlorosulphonic acid in acetic anhydride. The products are acid-resisting wetting, emulsifying, and cleaning agents.

C. HOLLINS.

Manufacture of substituted guanidines. SCHERING-KAHLBAUM A.-G. (B.P. 297,029, 13.8.28. Ger., 12.9.27).—Dialkylcyanamides (diethyl or dimethyl) react with amines mixed with their hydrochlorides to give tri-substituted guanidines. *N*-Phenyl-*N'*-*N'*-diethyl- (picrate, m.p. 118°), *NN*-diethyl-*N'*-isoamyl- (picrate, m.p. 120°), *NN*-dimethyl-*N'*-isoamyl- (picrate, m.p. 104°), and *NN*-dimethyl-*N'*-ethyl- (picrate, m.p. 152°) guanidines are described.

C. HOLLINS.

Manufacture of disubstituted guanidines. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 304,360, 19.10.27).—The reaction between cyanogen chloride or bromide with amines (aniline, ethylaniline, toluidines, benzylamine, methylamine, etc.) gives improved yields in presence of a salt of the amine; e.g., 5 pts. of aniline and 1 pt. of aniline hydrochloride or sulphate dispersed in carbon tetrachloride, water, etc. are treated with 1 pt. of cyanogen chloride at 0° and heated at 80–100° for 1 hr.

C. HOLLINS.

Introduction of sulphocyanide [thiocyano-] groups into organic compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,813, 5.9.27. Cf. Söderbäck, B., 1920, 189 A).—Arylamines or phenols are treated with thiocyanogen or with a thiocyanate and halogen in an organic medium which is solvent both for the amine and the thiocyanate. The solvent may be protected from the halogen by saturating it with sodium chloride or bromide. *p*-Substituted compounds yield *o*-thiocyano-derivatives, convertible into benzthiazoles. Phenol and ammonium thiocyanate in methyl alcohol, treated with bromine in the same solvent at 15–20°,

give *p*-thiocyanophenol, m.p. 58°. 4-Thiocyano- α -naphthol, m.p. 118°, 5-chloro-3-thiocyano-*o*-toluidine, m.p. 102°, 3-thiocyano-*p*-phenetidine, m.p. 85°, 1-thiocyano- β -naphthylamine, 1-thiocyano-7-methoxy- β -naphthylamine, 2-amino-1-thiocyanoanthracene, m.p. above 300°, and 2:6-diamino-1:5-dithiocyanoanthracene are also described.

C. HOLLINS.

Catalytic oxidation of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 296,071, 11.10.27, U.S., 26.8.27).—For a variety of catalytic oxidations (phthalic anhydride from naphthalene, anthraquinone from anthracene, benzaldehyde and benzoic acid from toluene, maleic acid from benzene, phenols, or furfuraldehyde, vanillic acid from eugenol, formaldehyde from methyl alcohol or methane, complete combustion of tars in coal-tar ammonia, etc.) a vanadium catalyst containing at least one multiple-component zeolite is used. An example is potassium vanadite, potassium silicate, and vanadyl sulphate, heated in burner gases at 400–500°, and, if desired, treated with dilute solutions of heavy-metal salts to effect base-exchange. Eight other examples are detailed.

C. HOLLINS.

Production of *N*-monoalkyl derivatives of aminophenols. W. TRAUBE and E. HELLRIEGEL (B.P. 280,553, 9.11.27. Ger., 15.11.26).—*p*-Aminophenol is converted by means of aqueous potassium thiocyanate into the thiocarbamido-compound, which on desulphurisation with lead acetate and alkali yields *p*-cyanamidophenol, m.p. 265°. This may be methylated with the usual agents to form *p*-cyanomethylamidophenol [*p*-hydroxyphenylmethylcyanamide], m.p. 133–134°, which is readily hydrolysed by acids, water, or alkalis to *N*-methyl-*p*-aminophenol.

C. HOLLINS.

Manufacture of *N*-[hydr]oxyethyl derivatives of 4-amino-1-[hydr]oxybenzene [*p*-aminophenol]. I. G. FARBENIND. A.-G. (B.P. 290,997, 29.12.27. Ger., 23.5.27. Addn. to B.P. 280,873; B., 1929, 38).—*p*-Aminophenol is treated in aqueous suspension with ethylene oxide in presence of calcium carbonate to give the *N*- β -hydroxyethyl and *NN*-di-(β -hydroxyethyl) derivatives.

C. HOLLINS.

Manufacture of condensation products from *m*- or *p*-cresol and aliphatic ketones. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 273,684, 15.6.27. Ger., 29.6.26. Cf. Gaebel, A., 1902, i, 767).—*m*-Cresol condenses with acetone at 30–40° in presence of hydrogen chloride to give a compound, b.p. 187°/0.8 mm. (acetyl derivative, m.p. 122–123°), said to be a polymeride of 4-isopropenyl-*m*-cresol or of the cyclic anhydride of 4- β -hydroxyisopropyl-*m*-cresol. *p*-Cresol similarly yields a compound, b.p. 192°/0.8 mm. (acetyl derivative, m.p. 151–152°).

C. HOLLINS.

Manufacture of alkylisopropenylphenols and alkylated coumarans. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 273,686, 15.6.27. Ger., 29.6.26).—The products from *m*- and *p*-cresols and acetone (B.P. 273,684, preceding) are depolymerised by distillation at 300°/100 mm., yielding 3:6- and 3:5-dimethylcoumarans, b.p. 98°/11 mm. and 102°/11 mm., respectively, together with isopropenyl-*m*- and -*p*-cresols.

C. HOLLINS.

Manufacture of alkylisopropylphenols and their hydrogenation products. SCHERING-KAHLBAUM A.-G. (B.P. 279,855, 25.10.27. Ger., 26.10.26).—The isopropenyl-*m*- and *p*-cresols and dimethylcoumarans of B.P. 273,686 (preceding) are hydrogenated in presence of nickel until 2—8 hydrogen atoms are taken up.

C. HOLLINS.

Manufacture of thymol, its isomerides or homologues, and their hydrogenation products. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 276,010, 11.8.27. Ger., 16.8.26).—The direct condensation product from acetone and *m*-cresol (B.P. 273,684, preceding) takes up 2 atoms of hydrogen in presence of nickel and the corresponding aluminium phenoxide at 180—190° and 10—13 atm. to give thymol. The product from *p*-cresol yields 3-isopropenyl-*p*-cresol. The corresponding cyclohexanols may be similarly prepared.

C. HOLLINS.

Manufacture of thymol, its isomerides and their hydrogenation products. SCHERING-KAHLBAUM A.-G. (B.P. 280,956 and 280,924, [A] 21.11.27, [B] 16.11.27. Ger., [A] 22.11.26, [B] 16.11.26. Addns. to B.P. 276,010; preceding).—(A) The ethers obtained from *m*- or *p*-cresol and acetone are applied, in place of the normal condensation products, in the process of the prior patent. (B) The normal condensation products are hydrogenated at 280° instead of the usual 180°.

C. HOLLINS.

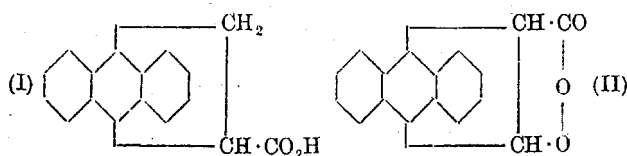
Manufacture of substituted indoles. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 303,467, 4.10.27).—Monoacyl derivatives of arylamines containing an *ortho*-methyl group are converted into indoles by heating with sodamide at 205° in a diluent such as dimethylaniline, naphthalene, etc. Aceto-*o*-toluidide gives 80—84% of 2-methylindole; 2:5- and 2:7-dimethyl- and 2-phenyl-indoles are similarly prepared.

C. HOLLINS.

Hydrogenation of naphthalene. TECHNICAL RES. WORKS, LTD., and E. J. LUSH (B.P. 304,403, 4.11.27).—Pure tetrahydronaphthalene is obtained by passing naphthalene vapour and hydrogen over a nickel catalyst at a temperature and with an excess of hydrogen such that condensation of the naphthalene on the catalyst does not occur.

B. FULLMAN.

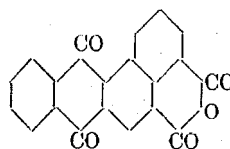
Manufacture of new carboxylic acids of the fatty-aromatic series. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 303,389, 1.7.27).— α -Unsaturated carboxylic acids or their hydrogen halide additive compounds are condensed with aromatic hydrocarbons containing more than two fused benzene rings, in presence of an acid catalyst (hydrogen halides, aluminium chloride) and a diluent if desired. Anthracene with acrylic or β -chloropropionic acid yields a compound (I), m.p. 187—188°; with maleic acid a β -lactone (II), m.p. 255°; with cinnamic acid a compound (analogous with I), m.p. 232°. Similar products are obtained



from acrylic or β -chloropropionic acid and *ms*-dichloroanthracene (m.p. 237°), 1:5-dichloroanthracene (m.p. 218°), 2:7-dimethylantracene (m.p. 205°), α -anthrol, or phenanthrene (m.p. 132°). C. HOLLINS.

Manufacture of aminoanthraquinones. H. BERTHOLD, ASSR. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,691,428, 13.11.28. Appl., 27.10.27. Ger., 2.12.25).— α -Nitroanthraquinones are reduced with hydrogenated quinolines in presence of acetic acid, pyridine, *o*-dichlorobenzene, quinaldine, or other diluent. The final mixture of quinaldine and excess unoxidised tetrahydroquinaldine may be catalytically hydrogenated and used again. 1:5-Dinitroanthraquinone affords 1:5-diaminoanthraquinone or 5-nitro-1-aminoanthraquinone; 1:5-dichloro-4-aminoanthraquinone and 8-nitro-1-aminoanthraquinone are similarly obtained. R. BRIGHTMAN.

Manufacture of benzantraquinone derivatives and substitution products. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 304,263, 18.10.27).—4-*o*-Carboxybenzoyl-1:8-naphthalic anhydride, m.p. 232°, obtained by oxidation of the acenaphthene derivative, is (unlike the latter) readily cyclised by means of sulphuric acid at 170—200° to give $\alpha\beta$ -benzantraquinone-4:5-dicarboxylic anhydride (annexed formula), m.p. above



300°; the imide, m.p. above 300°. The *N*-methylimide, m.p. 280°, and the 8:11-dichloro-anhydride are synthesised in a similar manner, the last-mentioned from 3':6'-dichloro-2'-carboxybenzoyl-1:8-naphthalic anhydride, m.p. 274°.

The condensation product of 4-*o*-carboxybenzoyl-1:8-naphthalic anhydride with *o*-phenylenediamine, viz., 4-*o*-carboxybenzoyl-1:8-naphthylenebenzimidazole, may also be cyclised to the corresponding benzantraquinone, which is a vat dye.

C. HOLLINS.

Manufacture of dibenzanthrone. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 303,538, 6.9.27).—Improved yields of dibenzanthrone are obtained in presence of aliphatic, non-sugar aldehydes or their bisulphite compounds or sulphoxylic acids, e.g., paraformaldehyde, formaldehyde-bisulphite, sodium formaldehydesulphoxylate.

C. HOLLINS.

Production of benzanthrone derivatives [isodibenzanthrones]. I. B. ANDERSON, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 303,454, 24.6. and 20.7.27).—Benzanthrone is converted smoothly into isodibenzanthrone by heating with alcoholic potassium hydroxide in a diluent (xylene, benzene, chlorobenzene, kerosene, or petrol) at 130°. Similar results are obtained with 9- and 10-methylbenzanthrone, 4:4'-dibenzanthronyl, benzanthrone from 1-chloroanthraquinone (previously separated from the product of m.p. 180—181°) or 2-chloroanthraquinone (freed from 4-chloro-benzanthrone). [Stat. ref.] C. HOLLINS.

Rectification of acetic acid. G. F. LEGENDRE, ASSR. to SOC. DES ETABL. BARBET (U.S.P. 1,698,793, 15.1.29. Appl., 12.8.25. Fr., 14.8.24).—See B.P. 238,566; B., 1925, 899.

Manufacture of alkali salts of halogenated amides of aromatic sulphonic acids. H. GÜNZLER, ASSR. to

WINTHROP CHEM. CO., INC. (U.S.P. 1,699,888, 22.1.29. Appl., 12.10.25. Ger., 18.10.24).—See B.P. 241,579; B., 1926, 565.

Halides of aromatic oxamic acids. J. HALLER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,685,698, 25.9.28. Appl., 26.11.26. Ger., 25.11.25).—See B.P. 282,891; B., 1928, 151.

Treatment of coal-distillation gases (B.P. 304,345). Diphenylene oxide from coal tar (G.P. 451,536). Pyroligneous acid (F.P. 628,785).—See II. Vat dye intermediates (B.P. 303,203).—See IV. Protecting copper apparatus against corrosion by carboxylic acids (B.P. 284,685).—See X. Esters of alcohols and fatty acids (F.P. 615,953).—See XII. Separation of mixed substances (B.P. 272,968). Alkylaminoalkylamino-derivatives of aromatic compounds (B.P. 274,058 and 302,984—5).—See XX.

IV.—DYESTUFFS.

Red Congo acid and Congo-red lakes. WEISER and RADCLIFFE.—See XIII.

PATENTS.

New basic and acid dyes of the Rhodamine type. IMPERIAL CHEM. INDUSTRIES, LTD., and L. J. ALLCHIN (B.P. 303,243, 7.11.27).—Fluorescein chloride is condensed with an unsulphonated aminophenol (e.g., *p*-aminophenol) to give basic dyes which may be converted by sulphonation into blue, acid rhodamines of good fastness properties. The basic dyes may also be synthesised from hydroxyaryl-*m*-aminophenols and phthalic anhydride.

C. HOLLINS.

Manufacture of [sulphide] dyes. SOC. CHEM. IND. IN BASLE (B.P. 290,179, 7.5.28. Switz., 7.5.27).—Chromable azo dyes are converted into their chromium derivatives, which are then heated with sodium polysulphide to give sulphide dyes. Examples are 1:2:4-aminonaphtholsulphonic acid \rightarrow α -naphthol or 5:8-dichloro- α -naphthol for greenish-blue dyes.

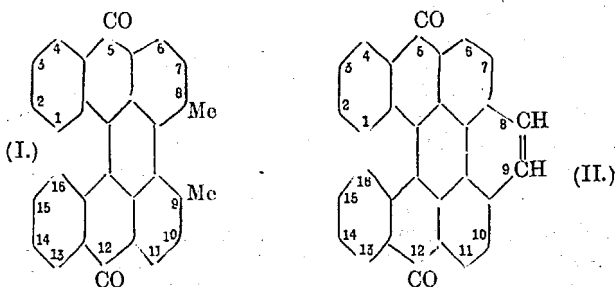
C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series], and intermediate products thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,203, 30.9.27 and 19.6.28).—Arylsulphonamido-benzanthrones, -dibenzanthronyls, or -dibenzanthronyl sulphides are obtained by the action of arylsulphonyl chloride on corresponding amines, or of arylsulphonamides on suitable halogen derivatives, preferably in presence of acid-binding agents and/or catalysts. Those containing in position 3 or 4 the arylsulphonamido-group or another group eliminated during alkaline fusion are converted by alkaline fusion into vat dyes containing free amino-groups. Thus, 3-benzenesulphonamidobenzanthrone, m.p. 273°, gives by fusion with alcoholic potassium hydroxide a reddish-blue vat dye; 3:9-di(benzenesulphonamido)-benzanthrone, m.p. 287°, gives a grey to black vat dye; 3-bromo-9-benzenesulphonamidobenzanthrone, m.p. 300°, a similar dye; 2-benzenesulphonamidobenzanthrone, a greenish-grey after hydrolysis of the benzenesulphonyl residue; di(benzenesulphonamido)-2:2'-dibenzanthronyl, grey to black; the 3:3'-isomeride, olive-grey to black;

6:6'-di(benzenesulphonamido)-3:3'-dibenzanthronyl sulphide, after hydrolysis, bluish-grey to black.

C. HOLLINS.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,184, 27.6.27).—8:9-Dimethyl-*ms*-benzodianthrone (I) is converted by alkaline condensing agents (baryta in boiling nitrobenzene, alcoholic potassium hydroxide at 170–175°, dry potassium hydroxide at 210° or in boiling aniline) into "allo-*ms*-naphthadianthrone" (II), m.p. 349–351°, an orange vat dye. 6:11-Dichloro-8:9-dimethyl-*ms*-benz-



dianthrone, prepared from 4-chloro-1-amino-2-methyl-anthraquinone, similarly yields a brown vat dye. These undergo further cyclisation by loss of hydrogen from positions 1 and 16 when stirred with manganese dioxide in sulphuric acid or heated with aluminium chloride, giving *ms*-anthradianthrone and its 5:10-dichloro-derivative, both of which are yellow vat dyes, and are obtainable by alkaline condensation of ordinary *ms*-naphthadianthrone. *allo*-*ms*-Naphthadianthrone (II) gives on chlorination with sulphuryl chloride in nitrobenzene or with chlorine in trichlorobenzene a dichloro-derivative (orange); dibromo- (scarlet) and tetrabromo- (red) compounds are also prepared. Bromination of 5:10-dichloro-*allo*-*ms*-naphthadianthrone yields a violet-red vat dye. Chloro- (orange) and dibromo- (orange) derivatives of *ms*-anthradianthrone may be obtained either by direct halogenation, or by cyclisation of halogenated *allo*-*ms*-naphthadianthrone. Dichlorodibromo-*allo*-*ms*-naphthadianthrone, obtained by chlorination and bromination, is a brilliant red vat dye.

C. HOLLINS.

Manufacture of new azo dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 301,175, 28.10.27).—Azo dyes giving yellowish-red to brown shades on wool, changed to blue, violet, or black shades on afterchroming, and reddish-brown shades when chrome-printed on cotton, are obtained by coupling diazo compounds with β -naphthol-6-carboxylic acid. Reference is made to the dyes from aniline (yellowish-red), *p*-chloro-aniline-*o*-sulphonic acid, *p*-nitro-*o*-aminophenol (brown), 2-nitro-6-aminophenol-4-sulphonic acid (violet), picramic acid, 1-amino-2-naphthol-4-sulphonic acid, 4-chloro-2-aminophenol-6-sulphonic acid, *p*-nitro-*o*-aminosalicylic acid, *o*-aminophenol-*p*-sulphonic acid, 5-sulpho-3-carboxy-2-hydroxybenzene-1-azo-4'-aminonaphthalene, 4-amino-1:2'-azonaphthalene-6:6'-disulphonic acid (dark violet), benzidine-*mm'*-disulphonic acid (reddish-brown), *o*-toluidine-*m*-disulphonic acid, *m*-dichlorobenzidinedisulphonic acid, 2-naphthylamine-1-sulphonic acid (red), *o*-toluidine-*m*-sulphonic acid, and acetyl-*p*-phenylenediaminesulphonic acid. The dyes obtained are superior

in solubility and tinctorial power to corresponding dyes obtained from 2:3-hydroxynaphthoic acid and afford yellower shades. The dyes from *p*-toluidine-*m*-sulphonic acid and aniline-*o*-sulphonic acids afford red barium and calcium lakes.

R. BRIGHTMAN.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (B.P. 275,258, 29.7.27. Ger., 29.7.26).—Diaz compounds are coupled with *N*-carbethoxyaminonaphthols, e.g., *p*-aminoacetmethylanilide \rightarrow *N*-carbethoxy-*J*-acid (level wool dye), and *m*-4-xylidine \rightarrow *N*-carbethoxy-*H*-acid (bluish-red wool dye).

C. HOLLINS.

Manufacture of azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 302,965, 22.9.27).—An amido-sulphonylanthranilic acid, $\text{NRH}\cdot\text{SO}_2\cdot\text{Ar}(\text{NH}_2)\cdot\text{CO}_2\text{H}$, is diazotised and coupled with a coupling component to give wool dyes suitable for chroming or pre-chroming and fast to washing, fulling, carbonising, and light. 5-*o*-Anisididosulphonylanthranilic acid [4-amino-3-carboxy-benzenesulphon-*o*-anisidide] is prepared by condensing 2-chloro-5-chlorosulphonylbenzoic acid with *o*-anisidine and then with ammonia under pressure. It couples with α -naphthol-5-sulphonic acid (yellowish-red on wool, becoming bordeaux-red by after-chroming), or with phenyl- γ -acid (reddish-brown; black-brown on chroming). Other examples are the 5-sulphonanilide with 1-(2'-hydroxy-3'-carboxy-5'-sulphophenyl)-3-methylpyrazolone (reddish-yellow; greener on chroming or coppering); the 5-sulphonamide with acetyl-*H*-acid, pre-coppered (wine-red), or with β -naphthylamine-7-sulphonic acid, pre-chromed (red-violet pigment for varnishes); the 5-sulphon-*p*-toluidide with β -naphthol-8-sulphonic acid, pre-coppered (brownish-orange), or α -naphthol-4-sulphonic acid, pre-coppered (copper-red), or β -naphthol-6-sulphonic acid, pre-coppered (yellowish-red-brown), or 1-*p*-sulphophenyl-3-methylpyrazolone, pre-coppered (greenish-yellow); the 5-sulphonanilide with β -naphthylamine-7-sulphonic acid (reddish-orange; bordeaux-red on chroming; pre-chromed, a violet pigment for varnishes).

C. HOLLINS.

Manufacture of arylazodiarylamines [dyes for wool and acetate silk]. I. G. FARBENIND. A.-G. (B.P. 276,372, 22.8.27. Ger., 21.8.26).—An aminoazo-compound is condensed with a halogenonitrobenzene containing reactive halogen and at least one sulphonc or carboxyl group. Examples are 4-aminoazobenzene with 1-chloro-2:6-dinitrobenzene-4-sulphonic acid (yellow on acetate silk), or 1-chloro-2-nitrobenzene-4-sulphonic acid (yellow on wool or acetate silk), or 2-chloro-3:5-dinitrobenzoic acid (reddish-yellow on wool, golden-yellow on acetate silk).

C. HOLLINS.

Manufacture of a black trisazo dye. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,424, 1.9.27).—*p*-Nitroaniline is diazotised and coupled acid with γ -acid; the product is reduced with sodium sulphide, rediazotised, and coupled alkaline with γ -acid; the disazo dye is finally diazotised and coupled with *m*-phenylenediamine. A further sulphonc group may be introduced into any of the components; e.g., 2:8-aminonaphthol-3:6-disulphonic acid may replace the second mol. of γ -acid. The resulting dyes give deep black shades on cotton or viscose silk, but leave acetate silk unstained.

C. HOLLINS.

Manufacture of nitrosoamine printing colours. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 303,942, 28.10.27).—Mixtures of nitrosoamine salts and coupling components (e.g., the Rapid Fast series of printing colours) are freed from sand, dust, iron particles, and other obnoxious impurities by dissolution in a neutral organic solvent, filtering, and removing the solvent. Suitable solvents are acetone, alcohol, methyl alcohol.

C. HOLLINS.

Benzanthrone derivatives (B.P. 304,263 and 303,454).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Tensile strength and fluidity of chemically modified cotton. D. A. CLIBBENS and B. P. RIDGE (J. Text. Inst., 1928, 19, 389—404 r).—Relations are established between the tensile strength and the fluidity in cuprammonium solution of samples of scoured, combed, Sakel yarns after treatment with hydrochloric and sulphuric acids, alkaline and neutral hypochlorite, alkaline hypobromite, and dichromate in the presence of sulphuric and oxalic acids. For a given fluidity, the same loss of strength is obtained irrespective of whether it is caused by the action of concentrated acids at the ordinary temperature or by dilute acids at the boil. Similarly, the relation between fluidity and tendering is the same whether the latter is caused by the action of hypobromite or of hypochlorite in neutral or alkaline solution, but for the same fluidity, acid attack corresponds with slightly greater loss of strength than does attack by these oxidising agents. Over the fluidity range 10—30 (corresponding roughly with 10—50% loss of strength) the curves for fluidity-% loss of strength are approximately straight lines for attack by both acids and hypochlorite solutions. Oxidation with dichromate in the presence of sulphuric or oxalic acid of concentrations insufficient to cause tendering in the absence of dichromate gives high fluidities accompanied by relatively slight loss of strength. For the yarns used, the percentage loss of strength of the single threads attacked with either acids or hypochlorite is equal to the loss of strength of the individual hairs composing the threads. When material modified by means of acids or alkaline hypochlorite is submitted to an alkaline boil, neither the strength nor fluidity of the product is materially affected, but for cotton modified by other oxidising agents both fluidity and loss of strength are increased. The most marked rise of fluidity on boiling is shown by neutral hypochlorite oxycelluloses, but the rise is slight for yarn oxidised by dichromate, although in this case the additional loss of strength is greatest. Cotton oxidised by dichromate differs from that oxidised by neutral hypochlorite in that (1) for a definite rise of fluidity it shows a much smaller loss of strength, and (2) alkali boiling produces a large additional loss of strength with a comparatively slight rise of fluidity. The fluidity of cotton attacked by dichromate indicates not the actual loss of strength, but a potential tendering which is realised only after the material is boiled with alkali. These facts vindicate the use of fluidity as a strength control, since material which would lose half its strength on laundering might be passed as satisfactory by a direct,

tensile test, whilst being rejected as unsatisfactory by the fluidity measurement. Within the limits of experimental error, the same fluidity, after boiling, corresponds with the same loss of strength whether the modification is caused by hypochlorite in neutral or alkaline solution or dichromate in the presence of sulphuric or oxalic acid, whilst the fluidity-strength relationship for yarn tendered by acid is roughly the same as that for yarn submitted to oxidising attack and subsequently boiled with alkali. Although it cannot be maintained that a given rise of fluidity always corresponds with the same loss of strength if all possible causes of the chemical modification be taken into account as well as the possibility of subsequent hot alkali treatment, any chemical processes which are accompanied by a considerable rise of fluidity of the material reduce its strength, actually or after a mild alkali treatment, by an amount which constitutes serious practical damage. B. P. RIDGE.

Identification of rayon. W. D. GRIER (Ind. Eng. Chem., 1929, 21, 168—171).—The importance of microscopical examination is emphasised. Whilst the majority of textile fibres can be identified by longitudinal observation under the microscope, it is necessary to examine transverse sections in order to identify rayon filaments, since their longitudinal appearances are similar, and in some cases the results of chemical tests are inconclusive. The appearance of sections of the more important types of rayon in ordinary and polarised light is described, and diagrams and photomicrographs are given. The technique of section cutting and the apparatus required are described. B. P. RIDGE.

Characterisation of celluloses. T. LIESER (Cellulosechem., 1929, 10, 21—27).—Numberless natural and artificial celluloses are believed to exist each with different physical and chemical properties, and all natural cellulose fibres to contain many chemically different celluloses; a chemical method, based on viscose formation, for their differentiation is described. At a certain specific concentration of sodium hydroxide, which, at a given temperature, varies with the form of cellulose, a compound of the type $2C_6H_{10}O_5 \cdot NaOH$ is formed, the formation of which is characterised by the possibility of undergoing the viscose reaction. Thus natural cotton cellulose forms this compound only with at least 16—18% sodium hydroxide solution, whilst cellulose-A (precipitated from concentrated hydrochloric acid solution) requires only a 7.5% solution. There exists a whole range of intermediate celluloses which form compounds, and, in consequence, undergo the viscose reaction, with increasingly lower concentrations of alkali. Thus, if the percentage of the sample undergoing the viscose reaction is plotted against the concentration of alkali a series of different curves are obtained with different types of celluloses. When natural cotton cellulose is treated with alkali of increasing concentration, above 2*N* the proportion undergoing the viscose reaction increases with increasing alkali concentration until it is complete at 5*N*. Lignocellulose gives the viscose reaction at a lower alkali concentration, whilst with artificial silk the proportion of viscose formed increases rapidly at concentrations of alkali above *N*. In the practical application of the method for the differentiation of celluloses and for the detection and

determination of cellulose-A (which is useless for the preparation of artificial silk), excess of alkali must be avoided, just sufficient being added to 1 g. of the cellulose to moisten all the fibres. After 1 hr. at 21°, 0.5 c.c. of carbon disulphide is added and viscose formation allowed to proceed in the thermostat. After dilution with water the insoluble residue is filtered through a hardened paper and washed until the filtrate gives no precipitate with mineral acids. The residue is then washed with 0.5*N*-hydrochloric acid, dried at 103°, and weighed. A number of typical results with various types of cellulose are given. J. W. BAKER.

Heartwood of the pine. III. Heating in sealed tubes. C. G. SCHWALBE and A. AF EKENSTAM (Cellulosechem., 1929, 10, 27—34; cf. B., 1929, 166).—The effect of drying, extraction with organic solvents and alkali, and previous impregnation with the various extracts, on the formation of cellulose has been studied in detail with standard sealed-tube heating in an attempt to elucidate the nature of the material present which inhibits the decomposition. The main results are as follows. The yields of cellulose obtained by heating spruce heart- and sap-wood, and Scottish pine sapwood with calcium bisulphite (1% CaO , 4.05% SO_2) are, respectively, 46.8, 46.0, and 48.2%, whilst under the same conditions the heartwood of Scottish pine is completely unattacked. Previous extraction of the latter with ether, benzene, or acetone before the bisulphite heating gives either no yield or only a poor-quality pulp, but pre-extraction with alcohol yields a good cellulose (cf. Hägglund, B., 1927, 294). If, however, the wood is previously air-dried (3 months) or, better, dried at 105° for 40 hrs. and then ether-extracted, bisulphite treatment then gives, respectively, 57.8 and 64.5% yields of good cellulose. The decomposition depends on the impregnability of the wood with the bisulphite, and complete drying renders the wood more permeable to the ether, allowing the more complete removal of the ether-soluble portion. When the pine heartwood is previously treated for 10 days with 0.1*N*-sodium hydroxide and then washed with cold water for 3 days, the bisulphite heating yields 66.2% of cellulose (ash 1.28%). Further washing for 10 hrs. with hot water gives a 65% yield (ash 0.979%), whilst a final washing with 0.5% acetic acid for 1 hr. and water for 2 hrs. gives a 63.4% yield (ash 0.491%). Although the substance which inhibits the bisulphite decomposition is readily removed from shavings with alkali, the use of the latter is not practicable with the larger pieces used technically. Similar pretreatment with calcium hydroxide solution yields specimens from which the calcium cannot be removed by washing and which contain about the same amount of calcium as is found after heating with calcium bisulphite. Subsequent heating with sulphurous acid (2.5—3.5% SO_2) causes no decomposition. From a study of the effect of impregnating the pine and spruce sapwoods with the various organic and alkali extracts of the pine heartwood on the bisulphite decomposition it is concluded that the substance which inhibits the bisulphite decomposition is acidic, forms a soluble calcium salt, is soluble in alcohol, benzene, acetone, and ether, and insoluble in light petroleum. The calcium hydroxide extract of the pine heartwood differs from

the spruce extracts in that it becomes turbid on acidification, and on the basis of the above properties the isolation of this acidic substance in small quantity as a viscous brown liquid is described. J. W. BAKER.

Sulphonation of lignin from pine wood. I. E. HÄGGLUND and T. JOHNSON (Biochem. Z., 1928, 202, 439–452).—Lignin isolated from pine wood by means of concentrated hydrochloric acid was treated with hot bisulphite solution, under conditions similar to those obtaining in the sulphite-cooking process. The solubility of the product was the greater the more protective the circumstances of isolation had been. Lignins as easily soluble as that in wood were obtainable. The presence of cellulose or of sugar did not affect the rate of dissolution. On sulphonation of the lignin insoluble salts of lignosulphonic acids are first formed: these are converted by dilute acids into soluble lignosulphonic acids. The rate of dissolution of lignin is directly related to the p_H of the cooking liquid. The dissolved lignosulphonic acids behave like those of waste sulphite liquors. If sulphonation and dissolution succeed each other rapidly, sulphonic acids the naphthylamine compounds of which have the same sulphur content as those of the sulphonic acids of sulphite waste liquors are obtained; but if the reactions proceed slowly, as in the case of lignin treated with hydrochloric acid, the sulphur content of the lignin compounds precipitable by naphthylamine rises. The lignosulphonic acids which go into solution contain up to 15% of loosely bound sulphurous acid. In the sulphite waste liquors sulphur dioxide is loosely combined probably with the portion of the lignosulphonic acids which cannot be salted out. This portion has a copper number which is relatively high in comparison with that of the portion which can be salted out. Both solid lignosulphonic acids and those which dissolve acquire violet-coloured fluorescence when illuminated with a quartz lamp, provided that the lignin of the acids has been prepared under protective conditions. The molecules of lignosulphonic acids contain certain groupings which condition the fluorescence, and these are exceptionally sensitive towards acids. W. MCCARTNEY.

Hydrophilic properties of sthenosised cellulosic fibres. L. MEUNIER and R. GUYOT (Compt. rend., 1929, 188, 506–508; cf. B., 1927, 276).—Sthenosis, or the strengthening of viscose with a 5% aqueous, phosphate-buffered solution of formaldehyde (p_H 1.6–1.2), is best performed by immersion for 12 hrs. followed by centrifuging, heating at 60–90° for 2–48 hrs. according to the intensity of sthenosis required, and washing to remove acid. The strength and resistance to alkalis of viscose increases, but the powers of absorption and the humidity determined both by adsorption and desorption decrease proportionally with the intensity of sthenosis. The mechanism is a partial conversion of cellulose into hydrocellulose by the acid liquor, with an increase in the number of hydroxyl groups, and, during evaporation, the catalytic formation by the more concentrated acid of methylenic derivatives. If it is assumed that the cellulose crystallites are formed from a system of parallel chains of glucose residues ($C_6H_{10}O_5$), then the changes in hydrophilic properties may be explained

by the reaction of different chains with 2 mols. of formaldehyde. J. GRANT.

Sulphite liquor. LAUBER.—See VII.

Erratum.—B., 1929, 201, col. 2, line 33, for “pine” read “spruce.”

PATENTS.

Utilisation of palm leaves. J. AYRE (Austral. P. 1298, 6.4.26).—Woolly fibres obtained by mechanical treatment of the leaves of the zamia palm etc. are purified by treatment with sulphur dioxide and combed, and the residues are incinerated, the ash being used as a fertiliser. L. A. COLES.

Saturation of fibrous material. R. T. JOHNSTON, Assr. to FLINTKOTE Co. (U.S.P. 1,698,886, 15.1.29. Appl., 20.3.23).—Material after passing through hot saturant at atmospheric pressure is exposed to a vacuum out of the saturant. J. S. G. THOMAS.

Manufacture of a material for covering or wrapping perishable goods etc. Preservation of foods and other perishable goods. A. H. KILNER (B.P. 304,168 and 304,341, 15.10.27).—(A) Cotton or other fabric is passed vertically through a bath containing viscose, whence it is withdrawn through an adjustable slit at the bottom, so that the solution is forced into the threads and meshes of the fabric. The stretched material is passed through dilute sulphuric acid to coagulate the viscose and is then washed and dried. (B) Gas-tight bags or containers for enclosing food etc. and made of the treated material are described. F. R. ENNOS.

Gas-cell fabric. W. J. KELLY, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,691,764, 13.11.28. Appl., 23.6.25).—Balloon fabric is impregnated with a solution of cellulose xanthate containing acidified sodium sulphate or other material inhibiting bubble formation, and the cellulose is regenerated on the fabric and softened, preferably under slight tension. R. BRIGHTMAN.

Protection of materials such as wool, fur, etc. from the ravages of moth. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,092, 28.9.27).—Organoderivatives of phosphorus, arsenic, antimony, bismuth, or tin, i.e., compounds in which these elements are linked directly to carbon, are used for moth-proofing purposes. Examples are tri-*p*-tolylphosphine, triphenylarsine, tribenzylarsine, tri(dimethylaminophenyl)arsine, triphenylarsine dihydroxide, triphenylstibine, tri-*o*-tolylstibine, diphenylethylstibine, tri(bromophenyl)stibine, trimethylstibine sulphate, dibromide, and dichloride, triethylstibine dichloride, triphenylstibine oxide, tri- α -naphthylstibine, bismuth triphenyl, bismuth tri-*p*-tolyl, tin tetraphenyl, tin tetrabenzyl. C. HOLLINS.

Waterproofing of textiles, paper, etc. M. E. P. FAVRESSE and J. C. PERES (F.P. 629,291, 18.2.27).—A suspension of flour in water is heated with sodium hydroxide at 120° until the starch is completely converted into dextrin. After cooling, a solution of casein and sodium carbonate or silicate is added and the articles to be waterproofed are soaked in this mixture, calendered hot, and treated with a solution of cellulose acetate in acetone and benzyl alcohol. A. R. POWELL.

Hardening and waterproofing of porous or fibrous materials. H. A. AARONSON, and CHEM. WATERPROOFING CORP. (B.P. 303,935, 20.10.27).—Textile and paper materials are impregnated or sprayed with a suitable coal-tar pitch dissolved in a non-inflammable solvent such as trichloroethylene or tetrachloroethane, and the solvent is then removed by heating.

A. J. HALL.

Preparation of fur for shrinking and felting. J. H. MARTIN (U.S.P. 1,685,168—9, 25.9.28. Appl., [A, B], 28.4.26).—Fur is treated with an aqueous solution of (A) an organic or (B) an inorganic acid containing a small amount of hydroxy-aliphatic acid, *e.g.*, glycolic or lactic acid.

R. BRIGHTMAN.

Textile oils. H. WADE. FROM STANDARD OIL CO. (B.P. 303,820, 6.7.27).—Oils suitable for lubricating textile yarns, particularly artificial silk, for knitting or weaving consist of a refined distillate mineral oil of low viscosity, oleic acid, a suitable emulsifying agent, *e.g.*, an alkali-metal soap, a sulphonated fatty oil compound, or an oil-soluble alkali-metal salt of a sulphonic acid derived from a sulphonated mineral oil, and an anti-oxidant (*e.g.*, β -naphthol or diphenylamine).

A. J. HALL.

Application of highly sulphonated oils in the textile industry. H. FLESCHE (B.P. 284,249, 4.8.27. Ger., 26.1.27).—Sulphonated oils (*e.g.*, of dihydroxystearic acid) containing at least 6% of organically combined sulphuric acid, but differing from those described in B.P. 263,117 (B., 1928, 845), are particularly suitable as additions to liquors containing high concentrations of caustic alkalis or neutral salts for the purpose of assisting their penetration of textile materials.

A. J. HALL.

Cooking of sulphite pulp. G. H. TOMLINSON (U.S.P. 1,697,032, 1.1.29. Appl., 7.12.25. Renewed 14.5.28).—The hot liquor discharged from one cooking process is employed to heat a fresh charge of chips and to wash it into the digester, where it is mixed with a charge of cooking acid heated to approximately the same temperature by means of the hot gases obtained from a previous digestion. The cooking is then completed by means of steam.

F. R. ENNOS.

Treatment of hydrated cellulose. I. G. FARBERIND. A.-G. (B.P. 278,684, 13.9.27. Ger., 5.10.26).—In order to diminish the tendency of hydrated cellulose to swell, it is immersed in or saturated with a neutral or feebly alkaline aqueous solution of a condensation product, *e.g.*, sulphur-*o*-cresol resin (cf. B.P. 186,107; B., 1922, 905 A), or the materials which can react to form such a product, with or without a catalyst. A further condensation is then brought about by addition of a substance such as formaldehyde with the formation of a product insoluble in water and dilute alkali.

F. R. ENNOS.

Treatment of fabrics made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 297,712, and 299,042, [A] 15.6.28, [B] 6.10.28. U.S., [A] 15.7.27, [B] 20.10.27. Addns. to B.P. 293,858).—Mechanical treatments are claimed.

F. R. ENNOS.

Conversion of nitrocelluloses which yield highly viscous solutions into nitrocelluloses which yield

solutions of low viscosity. I. G. FARBERIND. A.-G. (B.P. 289,387, 25.4.28. Ger., 25.4.27).—An unstable nitrocellulose is heated above 100° with water under pressure, with exclusion of acid and in the presence of an insoluble basic compound, *e.g.*, magnesia.

F. R. ENNOS.

Acylation of carbohydrates. E. B. MIDDLETON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,685,220, 25.9.28. Appl., 3.6.25).—Carbohydrates are suspended in glacial acetic acid, or other medium, containing mineral acid as catalyst, and treated with keten. Thus, cellulose in presence of 1.5% of sulphuric acid is converted into chloroform-soluble cellulose acetate.

R. BRIGHTMAN.

Manufacture of plastic compounds. G. E. ZELGER, Assr. to DU PONT-PATHÉ FILM MANUF. CORP. (U.S.P. 1,685,443—4, 25.9.28. Appl., [A] 6.4.25, [B] 30.7.26).—(A) Phosphoric esters containing both aromatic and aliphatic radicals, *e.g.*, diphenyl methyl phosphate, diphenyl ethyl phosphate, phenyl dimethyl phosphate, phenyl diethyl phosphate, or the corresponding tolyl derivatives, or (B) containing halogen-substituted aryl groups, *e.g.*, di(chlorophenyl) methyl phosphate, phenyl chlorophenyl methyl phosphate, chlorophenyl dimethyl phosphate, dichlorophenyl dimethyl phosphate, chlorophenyl diethyl phosphate, are mixed with cellulose esters or ethers to give non-inflammable plastic compounds.

R. BRIGHTMAN.

Production of high-grade knifing compositions and mixed lacquers containing them. **Production of knifing compositions.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 302,615—6, 12.9.27).—(A) The compositions contain a cellulose ester or ether, inorganic fillers of a scaly, foliaceous, or fibrous nature, *e.g.*, graphite, mica, asbestos powder, etc., and a finely-divided organic filler, *e.g.*, wood, cork, lignite, coconut fibre, etc., with or without the addition of solvents, lacquers, resins, plasticising agents, colouring materials, etc., but excluding resinous or gummy products extracted from wood meal etc. by alkaline solutions. (B) Cellulose derivatives are incorporated with more than three times their weight of kieselguhr, powdered pumice, or precipitated chalk, other substances being added if desired.

L. A. COLES.

Manufacture of waterproof straw pulpboard. E. B. WESTON and W. G. CLARK, Assrs. to TERRE HAUTE PAPER CO. (U.S.P. 1,697,702, 1.1.29. Appl., 4.11.26).—After digesting straw with steam in the presence of alkali, it is thoroughly beaten to a pulp and washed to remove alkali. A rosin size and an alum mordant are then incorporated with the pulp, and the product is formed into sheet.

F. R. ENNOS.

Cellulosic material and product obtained therefrom. H. DREYFUS and C. I. HANEY, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,697,907, 8.1.29. Appl., 19.1.27. U.K., 8.10.26).—See B.P. 288,657; B., 1928, 444.

Manufacture of artificial silk and other filaments by applying electric current. K. HAGIWARA, Assr. to T. IWASAKI (U.S.P. 1,699,615, 22.1.29. Appl., 14.3.27. Japan, 3.8.26).—See B.P. 286,086; B., 1928, 329.

Machinery for softening or loosening artificial fibrous materials. I. G. FARBERIND. A.-G. (B.P. 286,302, 24.2.28. Ger., 4.3.27).

Manufacture of [twisted] yarns or threads, and apparatus therefor. BRIT. CELANESE, LTD., W. A. DICKIE, T. H. PLATTS, and L. LATHAM (B.P. 304,806, 28.9.27).

Manufacture of [fibrous] plastic material [for bottle caps etc.]. E. C. R. MARKS. FROM LAKEWEST CORP. (B.P. 302,767, 26.10.27).

Manufacture of [compound] paper. J. R. HAPPER (B.P. 304,863, 12.11.27).

Rubber goods with textile insertions (B.P. 304,157).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Suitability of various metals and alloys for dyeing machines. J. G. GRUNDY (J. Soc. Dyers & Col., 1929, 45, 39—42).—A number of metals were tested in order to ascertain their reaction with dyestuffs and with the reagents used in dyeing, dyes from all groups of organic colouring matters being examined and the results compared against an acid-resisting enamel as standard. The majority of acid colours were sensitive to cast and wrought iron, copper, Firth stainless steel, and phosphor bronze, but not sensitive to nickel, monel, Staybrite, Dyebright, Silverite, and brass. Neolan colours were the least sensitive of those dyed from an acid dye-bath, owing, no doubt, to the fact that the colours were already metallic salts. Chrome colours, after-chromed, were very sensitive to iron, nickel, lead, and copper, but alloys of the Dyebright type were satisfactory. Direct and basic colours appear to be only affected appreciably by wrought and cast iron. Sulphur colours were considerably affected by metals containing copper owing to the action of the sodium sulphide used in the dye-bath. Vat colours were not affected by any of the metals tested, whilst the azoic colours gave poor results with only cast and wrought iron.

L. G. LAWRIE.

Colouring of rubber. NAUNTON.—See XIV.

PATENTS.

Production and colouring of textile yarns, fabrics, and the like. SILVER SPRINGS BLEACHING & DYEING Co., LTD., and A. J. HALL (B.P. 303,958, 8.11.27).—Viscose silk yarn is immersed for about 1 hr. at ordinary temperature in a 3—6% solution of caustic soda, then freed from alkali by washing and souring, dried, and used together with untreated viscose silk in the manufacture of woven or knitted materials which are afterwards dyed with direct dyes; two-tone coloured materials are thereby obtained since the alkali-treated silk has a greater affinity than untreated silk for many direct dyes. Caustic potash may be used instead of caustic soda, but a less marked increase of affinity of the silk for direct dyes is obtained (cf. B.P. 295,488; B., 1927, 745).

A. J. HALL.

Increasing the affinity of animal fibres for dyes. E. DU BOIS, Assr. to CHEM. WORKS FORMERLY SANDOZ

(U.S.P. 1,698,226, 8.1.29. Appl., 23.12.25. Ger., 6.1.25).—See B.P. 245,759; B., 1927, 475.

Apparatus for washing, bleaching, and dyeing artificial silk in hanks or spinning cakes. F. KÜTTNER A.-G. (B.P. 288,990, 16.4.28. Ger., 16.4.27).

Collapsible spindle for dyeing and analogous treatment of wound yarns. J. and T. BRANDWOOD (B.P. 304,800, 28.7.27).

Wetting, etc. agents (B.P. 303,917).—See III. Printing colours (B.P. 303,942).—See IV. Colouring of rubber (B.P. 304,376 and G.P. 452,340).—See XIV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Viscosity relationships in the system sulphuric acid-nitric acid-water. F. H. RHODES and H. B. HODGE, JUN. (Ind. Eng. Chem., 1929, 21, 142—150).—A large number of viscosity determinations were made on "mixed acid" at 0—75° with a view of identifying compounds which might be present. Capillary viscosimeters calibrated with sucrose solution were used. The binary system sulphuric acid-water shows in the viscosity curve maxima at 85% and 100% of acid and a minimum at 95% of acid, these points being less marked at the higher temperatures. In the system nitric acid-water less definite maxima occur at about 65% of nitric acid. Other variations in the curves are probably due to polymerising or depolymerising effects. In the system nitric acid-sulphuric acid maxima occur at 88% and 100% of sulphuric acid, with a minimum at 97% sulphuric acid. This is held to indicate the existence of a compound $10\text{SO}_3 \cdot \text{N}_2\text{O}_5 \cdot 9\text{H}_2\text{O}$. Diagrams of the viscosities of the ternary system are given. They show the superimposed effects obtained with the various binary systems, and do not indicate the presence of any new compounds.

C. IRWIN.

Manufacture of "converted" saltpetre. F. CHEMNITZ (Chem.-Ztg., 1929, 53, 85—86).—Sodium nitrate of 96—98% and potassium chloride of 80% purity, the latter in slight excess, are mixed with a mother-liquor from a previous raw saltpetre crystallisation. The chloride should contain not more than 0.6% of perchlorate nor more than 0.5% of magnesium salts, which must be precipitated by addition of the equivalent amount of sodium carbonate. The reaction is controlled by determinations of the sp. gr. of the solution at different stages. A purified product containing only 0.3—0.5% of sodium chloride and 0.1% of sodium nitrate is obtained.

F. R. ENNOS.

Preparation of sulphite liquor. H. LAUBER (Papier-Fabr., 1929, 27, 65—69).—The use of pyrites in the place of sulphur for the preparation of sulphite liquor is discussed. Sulphur losses in the roasting of the ores are dependent on the crystalline form and fineness of grinding of the material and on the presence of impurities (e.g., copper and zinc sulphides) which affect the fusion temperature. The efficiency of various types of pyrites burners is discussed, together with the technique of purification and absorption of the gases.

A. G. POLLARD.

Determination of potassium cyanate. J. LÉBOUCQ (Ann. Falsif., 1928, 21, 595—600).—The method depends on the formation of *s*-dicarbonamidohydrazine (solubility in water 0.15 g./litre) by the action of semicarbazide hydrochloride on the cyanate. The product is weighed and the proportion of cyanate calculated. The method can be applied in the presence of cyanide, less than 10% of cyanate being detectable if care be taken to operate in an acid medium after fixing the hydrocyanic acid with dextrose. It may also be applied to the determination of cyanate formed by the action of copper sulphate on an alkaline cyanide. D. G. HEWER.

[Determination of free alkali in hypohalogenite solutions.] P. FUCHS (Z. anal. Chem., 1929, 76, 150).—Rupp and Lewy's method (B., 1928, 365) is not new.

H. F. GILLBE.

Determination of nitrogen in cyanamide. S. REYNAERT (Natuurwetensch. Tijds., 1929, 11, 27—28).—It is unnecessary to continue heating in the Kjeldahl method until the mixture is decolorised; accurate results are obtained with 6 hrs.' heating only. Addition of potassium sulphate is also unnecessary. S. I. LEVY.

Action of water on calcium silicates. THORVALDSON and VIGFUSSEN.—See IX. **Radium and luminous paints.** PARSONS.—See XIII. **Agricultural lime.** REYNAERT. **Nitrogen in urea-nitrate mixtures.** LUCAS and HIRSCHBERGER.—See XVI. **Determination of potassium iodide.** WEICHHERZ.—See XX. **Adsorption of carbon dioxide by ferric hydroxide.** TILLMANS and others.—See XXIII.

PATENTS.

Speeding of chemical reactions. H. B. KIPPER (U.S.P. 1,697,390, 1.1.29. Appl., 14.5.26).—The manufacture of hydrochloric acid and sodium sulphate from sodium chloride and sulphuric acid is hastened by grinding and beating the material in a heated vessel having rapidly moving metal parts, caking of the material on the vessel being prevented by means of slowly moving metal parts. W. G. CAREY.

Manufacture of hydrochloric acid. E. R. WOLCOTT, Assr. to TEXAS Co. (U.S.P. 1,691,452, 13.11.28. Appl., 1.10.22).—Sodium chloride and clay are intimately mixed with a small amount of binder, *e.g.*, asphalt, pitch, acid sludge, and distilled in steam below 820°.

R. BRIGHTMAN.

Decolorisation of tartaric acid solutions. URBAIN CORP. (F.P. 622,649, 2.2.26).—The solution is treated with active carbon with or without an alkali or a colloidal precipitating agent, *e.g.*, an alkali alginate, and precipitation of the impurities is effected by subsequent addition of an electrolyte, *e.g.*, a solution of a copper, tin, or alkaline-earth salt. A. R. POWELL.

Production of alkali carbonates from alkali sulphates by the wet process. W. H. FRIEDRICH (G.P. 453,217, 24.2.26).—About 5% of an alkali hydroxide, calculated on the weight of the sulphate, is added as catalyst in the process described in G.P. 431,509 (B., 1926, 915).

L. A. COLES.

Production of alkali cyanides. H. B. KIPPER (U.S.P. 1,697,529, 1.1.29. Appl., 1.3.26).—The materials

are continuously and rapidly stirred and beaten in a closed vessel to maintain them in a finely-divided and disseminated condition while they are subjected to the action of highly heated reactive gases so that alkali cyanides are formed. The inner walls of the vessel are kept free by continuous and slow scraping.

W. G. CAREY.

Manufacture of potassium manganate. SOC. CHIM. DES USINES DU RHÔNE (B.P. 292,991, 19.3.28. Fr., 29.6.27).—Pure or dilute oxygen is blown through a mechanically stirred suspension of an oxide of manganese, or of compounds capable of liberating the oxides, in an excess of aqueous melted potash, containing 60—85% of potassium hydroxide, maintained at 160—200°.

W. G. CAREY.

Decolorisation of alkaline-earth thiocyanate solutions. R. R. FULTON, Assr. to KOPPERS Co. (U.S.P. 1,691,345, 13.11.28. Appl., 31.12.26).—Derivatives of soluble fatty acids are added to the solutions, particularly in presence of cellulose. *E.g.*, calcium thiocyanate solution is decolorised by being heated for 1 hr. at 120—130° with 0.4—2% of formaldehyde, or 3—5% of sodium formate.

R. BRIGHTMAN.

Rendering calcium cyanamide free from dust. J. BRESLAUER and E. DE LUSERNA, Assrs. to SOC. D'ETUDES CHIM. POUR L'IND. (U.S.P. 1,691,044, 13.11.28. Appl., 11.1.27. Switz., 22.1.26).—Calcium cyanamide is incorporated with a fusible potassium hydrogen salt, *e.g.*, potassium hydrogen sulphate, at 200°.

R. BRIGHTMAN.

Drying of superphosphate. VER. F. CHEM. & MET. PROD. (F.P. 626,190, 13.12.26).—The material falls in a finely-divided condition from the decomposition chamber into a countercurrent of hot gases, preferably a mixture of air and combustion gases.

L. A. COLES.

Production of basic copper carbonate from impure solutions of copper salts. A. M. BICKFORD & SONS, LTD., Assees. of J. HENDRY and P. A. BERRY (Austral. P. 2775, 13.7.26).—Bases other than copper are removed by selective precipitation before the basic copper carbonate is precipitated.

L. A. COLES.

Apparatus for the production of silicon or titanium chloride or bromide. I. G. FARBERIND. A.-G., Assees. of M. SCHLIEMANN (G.P. 453,367, 6.8.26).—Apparatus of the usual type is constructed of metal and provided with efficient cooling arrangements.

L. A. COLES.

Production of titanium hydroxide. SILESIA VER. CHEM. FABR., Assees. of G. ALASCHESKI (G.P. 453,120, 25.11.25).—Solutions containing titanium salts are atomised and treated with a countercurrent of superheated steam and/or hot gases.

L. A. COLES.

Simultaneous production of phosphorus or phosphoric acid and binding agents having latent hydraulic properties. I. G. FARBERIND. A.-G. (B.P. 297,416, 18.8.28. Ger., 21.9.27).—The process of B.P. 285,055 (B., 1928, 710) is modified by forming the charge of a mixture of crude calcium and aluminium phosphates in such proportions that a slag is produced similar in composition to that of the blast-furnace slag cement previously described.

W. G. CAREY.

Sodium sulphide pellets. R. ROOS, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,699,377, 15.1.28. Appl., 13.11.24. Ger., 18.1.24).—See G.P. 426,052; B., 1926, 583.

Hydration of phosphoric anhydride. E. URBAIN, Assr. to URBAIN CORP. (U.S.P. 1,698,484, 29.1.29. Appl., 21.4.27. Fr., 21.4.26).—See B.P. 269,908; B., 1927, 748.

Catalytic gas reactions (B.P. 304,269).—See I. **Vegetable waste products** (G.P. 452,810).—See II. **Catalysts** (B.P. 301,806).—See III. **Moth-proofing materials** (B.P. 303,092).—See V. **Recovery of metal compounds from ammoniacal liquors** (B.P. 301,342).—See X. **Determination of carbon dioxide** (U.S.P. 1,695,031).—See XI.

VIII.—GLASS; CERAMICS.

Relations between physical characteristics of glasses and their working properties on machines. G. GEHLHOFF (J. Soc. Glass Tech., 1928, 12, 145—157 p).

—Data are collected to show the influence of viscosity, thermal expansion, conduction and radiation, devitrification, annealing rate, and resistance to thermal and mechanical shock on the capability of working various types of glass by machine. A list of temperatures is given at which typical lead and soda-potash glasses are sufficiently hot just to adhere to various mould-forming materials.

A. COUSEN.

Refractory materials, with special regard to the glass industry. W. MIEHR (J. Soc. Glass Tech., 1928, 12, 158—176 p).—The selection and improvement of natural and artificial raw materials now used for making refractories for the glass industry, methods of producing these refractories, and the chemical and physical properties of the finished products are dealt with. Finally, an account of some experiences in the use of new kinds of refractory products in the works is appended.

A. COUSEN.

Characteristics of refractory clays for the glass industry. G. GEHLHOFF, H. KALSING, K. LITZOW, and M. THOMAS (J. Soc. Glass Tech., 1928, 12, 280).—A correction (cf. B., 1928, 928).

A. COUSEN.

Properties of glasses containing zinc oxide. S. ENGLISH, W. E. S. TURNER, and F. WINKS (J. Soc. Glass Tech., 1928, 12, 287—296).—Glasses of the molecular type $6\text{SiO}_2 \cdot (2-x)\text{Na}_2\text{O} \cdot x\text{ZnO}$ become increasingly quick-setting as the zinc oxide content increases. The values of d and n_D increase linearly from 2.4520 and 1.49975, respectively, for the first member of the series— SiO_2 74.09%, Na_2O 24.07%, ZnO 1.29%—to 2.5499 and 1.50829 for the last member— SiO_2 71.94%, Na_2O 14.70%, ZnO 11.39%. The average value of total dispersion is 0.00873 and of v is 57.8. For substitutions of sodium oxide by zinc oxide up to 6% there is no distinct increase in the annealing temperature, and up to 10% the effect is only small. Several change points occur in the rate of expansion of the samples between 0° and the lower critical temperature. Over the range 0—100° the thermal expansion decreases linearly with increase of zinc oxide content.

A. COUSEN.

The glass-melting furnace as a heat machine. D. AUFHÄUSER (J. Soc. Glass Tech., 1928, 12, 297—306).—The furnace is considered from the point of view of a heat machine, heat evolution and exchange being velocity problems, whilst the intensity factors are temperature difference and area of exposed surface.

A. COUSEN.

Ultra-violet light-transmitting glasses. D. STARKIE and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 306—320).—The transmission of eight commercial glasses of this type of 2 mm. thickness over the range 7000—2000 Å. has been determined. "Corex" glass transmitted practically as much light as quartz, having a transmission in the therapeutic region (2950—3150 Å.) of 89%, whilst the transmission of the remaining glasses were: "Sanalux" 73%, "Sun Ray" 62%, "Holvi" 61%, "Vita" 54%, "Helio" 52%, "Uviol" 46%, "Quartz-Lite" 4% (ordinary window glass 1%). Corex had the lowest iron oxide content, namely 0.0129%, that of the remaining samples ranging from 0.021% to 0.054%. After a 12 weeks' exposure to sunlight, Corex became stained with a white film, but, on being cleaned, showed no change in transmission. The other glasses tested were more durable on exposure, but showed the following decreases in transmission: Vita 13%, Helio 11%, and Sanalux 7%. When artificially aged under the carbon arc the greatest change occurred in the first 2 hrs., the fall in transmission coinciding with a decrease in the proportion of ferrous oxide to total iron oxide in the glass.

A. COUSEN.

Influence of ferric oxide content on the light-transmission of soda-lime-silica glass, with special reference to the ultra-violet. D. STARKIE and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 324—332).—Soda-lime glasses of the approximate composition SiO_2 75%, CaO 10%, Na_2O 15%, and containing iron oxide in amounts varying from 0.005% to 1.0%, were studied over the range 7000—2000 Å. The intensity of transmission on either side of the yellowish-green region decreased as the iron oxide content increased. By extrapolation, the limiting transmission for the glass free from iron was given as 2230 Å. All the glasses were melted in an oxidising atmosphere, and the ratio of ferrous oxide to total iron oxide averaged 6.9% as against 30% for commercial glasses transmitting ultra-violet light.

A. COUSEN.

Change in optical transparency of certain ultra-violet transmitting glasses after exposure to X-rays and ultra-violet light. G. M. SHRUM, C. G. PATTEN, and H. D. SMITH (Trans. Roy. Soc. Canada, 1928, [iii], 22, III, 433—436).—Commercial ultra-violet glasses were examined and showed the effect of solarisation when exposed to ultra-violet light, and both solarisation and coloration when exposed to X-rays. If the glasses were kept at 100° for some time the coloration faded, and the effect of solarisation was reduced to zero when the glasses were gradually heated to 300°. A thermo-luminescence was observed for all samples, and it was found that the recovery from the solarisation effect coincided with the disappearance of the thermo-luminescence. It is possible that the solarisation is a photochemical oxidation.

A. J. MEE.

Calculation of glass constants on the basis of recent investigation. E. ZSCHIMMER (J. Soc. Glass Tech., 1928, 12, 333—366).—A continuation of the systematic survey of recent determinations of the values of physical properties of glass (cf. B., 1928, 404), the properties dealt with being viscosity, annealing constants, density, thermal expansion, refractive index, and dispersion. A. COUSEN.

Action of fluorine in enamel smelts. H. G. WILCOX and F. C. WESTENDICK (J. Amer. Ceram. Soc., 1929, 12, 83—86).—By chemical analysis of the raw and smelted enamels it was shown that only part of the fluorine is lost during smelting. Longer smelting duration causes an increase and higher alumina content a decrease in fluorine lost. It is incorrect to assume that the fired and raw weights of either cryolite or fluorspar are the same. J. A. SUGDEN.

Some leadless borosilicate glazes containing nickel oxide. H. H. HOLSCHER (J. Amer. Ceram. Soc., 1929, 12, 111—117).—The literature pertaining to the use of nickel oxide is reviewed. The results of many trials, where comparison is possible, agree with previous investigators. It is concluded that nickel oxide has not proved useful in glossy, non-opaque glazes, and, in any case, is not recommended for use where a dark colour is desired, as all such colours are turbid. With suitable contents of lime (above 0.35%), barium oxide (not above 0.2%), and magnesia (barium oxide + magnesia not over 0.3%), nickel oxide can be made to produce attractive light tan, semi-matt, opaque glazes. J. A. SUGDEN.

Service of refractory blocks in a small experimental glass tank. W. L. PENDERGAST and H. INSLEY (J. Amer. Ceram. Soc., 1929, 12, 123—141, and Bur. Stand. J. Res., 1929, 2, 452—465).—Trials have been made in a small, laboratory, continuous glass tank 1 ft. × 1 ft. × 4 ft. long. The sides of the tank were built up of six blocks (6 in. thick) under test. The batch used was one higher in soda than the average soda-lime bottle glass, and therefore more corrosive. Each run lasted about 30 days. Many types of fireclay and special refractories were tested. There appears to be little connexion between either chemical composition or porosity and resistance to glass attack. It is pointed out that homogeneity of a refractory must have a considerable effect on its resistance to attack, as shown by the many cases of pitting. Such corrosion may be brought about by the presence of coarse pores, difference in composition between grog and bond, or lack of adhesion between the particles in the refractory. In order to predict the resistance of a refractory to glass attack, means of measuring these properties will have to be devised. J. A. SUGDEN.

New apparatus for measuring the thermal expansion of refractory materials at 1600°. K. ENDELL (Feuerfest, 1929, 5, 3—4).—Prismatic or cylindrical test-pieces, 60—100 mm. long, plane at each end, are heated in an electric muffle furnace. Four heating elements ("silite" rods) are placed symmetrically about the test-piece, which rests on a refractory support. Two telescopes, with parallel optical axes the distance between which can be varied and accurately

measured, are focussed one on each end of the test-piece. The length (or distance between marks) of the test-piece before the test is accurately measured with callipers. The telescopes are fitted with adjustable ocular micrometers with glass scale. At a distance of 1 m. from the object, each division of the scale corresponds to 0.1 mm. Changes in length can thus be read off directly without moving the apparatus, and inaccuracy due to parallax is avoided. F. SALT.

Determination of the sp. gr. of refractory materials. E. KÜHN (Feuerfest, 1929, 5, 5).—A "volumoscope" is described by means of which more than 30 sp. gr. determinations can be made in 1 hr. The apparatus is filled with 200 g. of the material, a vacuum is produced, and by a simple operation of the hand the true sp. gr. can be read off. The material being tested comes into contact with air only. F. SALT.

Smooth-surface tile. V. S. SCHORY (J. Amer. Ceram. Soc., 1929, 12, 75—78).—Successful attempts to produce a white floor tile with a surface which is unglazed or non-vitrified but which is smooth enough not to hold the dirt are described. After trials with many bodies it was found that an oxidising fire was essential to the production of the desired surface. A consequent reduction in the amount of buff firing material had to be made in order to retain the ivory-white colour. The composition is given of a reliable body maturing at cone 7. The type of felspar used was found to affect the extent of the development of the smooth surface, but no explanation of this is given. J. A. SUGDEN.

Pyrophyllite, a new ceramic raw material. G. R. SHELTON (J. Amer. Ceram. Soc., 1929, 12, 79—82).—The physical and chemical properties of the mineral are given and a brief description of the deposits and their origin and distribution. Tests are being carried out on the possible substitution of the mineral for some of the usual ingredients of white-ware bodies. It has been found that when replacing ball clay an increase in the amount of pyrophyllite causes an increase in whiteness, density, and shrinkage, a marked decrease in plasticity, and a decrease in adsorption. Owing to its talc-like properties it is expected that pyrophyllite will reduce the abrasion of press dies. The use of the mineral as a refractory (cone 27) and in glazes is being examined. J. A. SUGDEN.

Scratch-hardness tests of ceramic materials. L. NAVIAS (J. Amer. Ceram. Soc., 1929, 12, 69—74).—Preliminary experiments are described. Three sets of standards were prepared: (1) Mohs' scale of minerals, (2) a set of steel samples, and (3) a set of Carboloy (tungsten carbide alloy) samples. The last two series were arranged according to the Brinell and Rockwell hardness tests. The three sets of standards were compared with one another according to their scratch-hardness. Scratch tests were then carried out on stoneware, porcelains, glazes, glasses, and enamels. With one or two exceptions the order of hardness was the same with each of the three series of standards. J. A. SUGDEN.

Absorption of two earthenware bodies and their resistance to crazing in the steam test. E. P.

WRIGHT (J. Amer. Ceram. Soc., 1929, 12, 118—122).—An ivory and a white earthenware body matured to various absorptions were, when glazed, autoclaved to steam pressures of 100 and 150 lb./in.² The results show that bisque firing treatment as expressed by the absorption is of primary importance in resistance to crazing. Steam treatment at 100 lb. pressure will cause crazing above an absorption of 7—8% for the ivory body and above 8.5% for the white body. Treatment at 150 lb. pressure lowered the critical resisting absorption to 6% in each case. With both types of body there is a narrow range within which suitable glaze-firing conditions may increase the resistance to crazing. The composition of bodies and glaze is not given.

J. A. SUGDEN.

New method of direct firing glazed ceramic bodies. F. A. WHITAKER (J. Amer. Ceram. Soc., 1929, 12, 87—95).—The Carboradiant principle of oil firing has been applied to a rectangular sanitary-ware kiln. Improvement in control, increase in first-quality ware, and economy of fuel resulted. J. A. SUGDEN.

PATENTS.

Manufacture of reinforced glass. SOC. D'ETUDE DES VERRES ET GLACES DE SÛRETÉ (B.P. 293,053, 30.6.28. Fr., 1.7.27).—A sheet of transparent strengthening material, such as celluloid, is interposed between two sheets of glass the inner surfaces of which have been smoothed by grinding or by means of a concentrated varnish. The indices of refraction and the dispersion are the same for the two materials.

W. J. BOYD.

Manufacture of unsplinterable glass. J. H. ROBERTSON (B.P. 303,332, 25.6.28).—Two sheets of glass coated successively on one side with a solution prepared by treating casein with aqueous phenol solution, pouring off excess solvent, and dissolving the residue in strong ammonia, and with a solution of celluloid and phenol in, *e.g.*, amyl acetate, the coatings being dried after each application, are united to an interposed sheet of celluloid by gentle squeezing in a bath of alcohol, followed by heating at 55° for 20 min. in a press; the composite sheet is subsequently heated at 55—60° for several hours.

L. A. COLES.

White base for enamels and glazed. DEUTS. GASLÜHLICHT AUER GES.M.B.H. (F.P. 625,260, 29.11.26. Ger., 10.12.25).—White coatings are obtained by using bases comprising mixtures of titanium oxide with cerium oxide, or of either of these with white oxides of multivalent elements, preferably those of the 3rd, 4th, and 5th groups of the periodic system, *e.g.*, alumina, silica, stannic oxide, etc.

L. A. COLES.

Production of cold glazes. KERAMENT- & KUNST-STEINWERKE C. H. JERSCHKE A.-G. (F.P. 625,203, 27.11.26).—The sand in the usual mixtures is replaced by material capable of absorbing water, preferably slowly quenched slag in grains of 1—2 mm. diam., in quantity not less than 25% of the mixture. In the production of coloured glazes, a thin layer of the coloured material may be applied over a colourless foundation layer.

L. A. COLES.

Ceramic-glazing process. W. O. PROUTY, Assr. to AMER. ENCAUSTIC TILING CO., LTD. (U.S.P. 1,693,252,

27.11.28. Appl., 6.5.25).—Ceramic articles having a variegated colour are obtained by firing the bisque after the successive application of a glaze mixture of suitable colour, a glaze mixture of a different colour and containing insoluble material that is eliminated during the firing, and a final glaze mixture.

L. A. COLES.

Manufacture of chromite-containing refractory material. J. I. BROWN (B.P. 304,492, 10.2.28).—The usual clay, magnesia, or lime binder of the chromite material is replaced by a hydraulic cement (40% Al_2O_3). The addition of 5—10% of such fused cement enables moulded blocks or bricks to be laid without previous burning. For rammed materials 10—20% of cement is added.

J. A. SUGDEN.

Mould for high-temperature casting of refractory bodies. K. E. PEILER, Assr. to HARTFORD-EMPIRE CO. (U.S.P. 1,696,688, 25.12.28. Appl., 28.9.26).—A mixture of 80—85 pts. of fused silica, part of which is amorphous, ground to 20-mesh, 20—15 pts. of powdered lime, and a suitable quantity of water is dry-pressed and hardened by heat in the presence of steam.

L. A. COLES.

Manufacture of waterproof polishing and abrasive paper etc. C. KLINGSPOR (B.P. 302,430, 5.10.27).—The binding material comprises animal glue previously soaked in water mixed at a temperature not above 75° with a liquid phenol-formaldehyde condensation product, with or without the addition of alcohol, glycerin, linseed oil, etc. After applying the abrasive powder, the binder is hardened by heating gradually to a temperature not exceeding 150°.

L. A. COLES.

Manufacture of splinterless glass. G. B. RILEY (B.P. 304,630, 21.10.27).

Refractory materials (B.P. 302,087).—See X. **Dry-coating of lamp bulbs** (U.S.P. 1,698,845).—See XI.

IX.—BUILDING MATERIALS.

Setting of cement. III. H. GESSNER (Kolloid-Z., 1929, 47, 160—175; cf. B., 1929, 56).—Measurements of the hydration of cement show that definite amounts of water are taken up at different aqueous vapour pressures, the amount increasing with the time. The vapour-pressure isotherms resulting from the water-content curves show a definite break at a vapour pressure of 10.4 mm. in systems 3 days old; the break is shown less clearly by older systems. The conclusion is reached that in the initial stages of setting a definite hydrate is formed in the sheaths surrounding the clinker particles; during the subsequent course all the water bound by capillary forces becomes bound chemically. The velocity of diffusion of water in set cement is practically nil. Comparison of measurements of swelling with those of water content shows that the total system cement + water undergoes a contraction of about 7%.

E. S. HEDGES.

Action of water on tricalcium silicate and β -dicalcium silicate. T. THORVALDSON and V. A. VIGRUSSEN (Trans. Roy. Soc. Canada, 1928, [iii], 22, III, 423—431).—The hydrolysis of fairly pure samples of tricalcium silicate and β -dicalcium silicate was studied in an attempt to discover if products of definite chemical

composition are formed. The rates of hydrolysis of these two compounds were compared; that of tricalcium silicate is much more rapid than that of β -dicalcium silicate. It is possible that the hydrolysis of the latter takes place in two steps. The hydrolysis curves for the two silicates were determined from the point of complete hydrolysis to a point where the solution was slightly supersaturated by the lime liberated. The product of hydrolysis highest in lime which can exist in equilibrium with a solution of calcium hydroxide below saturation has a lime : silica ratio of approx. 3 : 2. An inflexion in the hydrolysis curves occurs at a point where the lime : silica ratio is approx. 1 : 1; this indicates the existence of a hydrated monocalcium silicate. The products of hydrolysis of the two silicates are probably identical.

A. J. MEE.

PATENTS.

Production of fused cement in rotating kilns. G. POLYSUS (F.P. 625,600, 2.12.26. Ger., 23.12.25).—The hot gases leaving the fusion compartments are divided into two unequal portions, the greater being used for drying and calcining the charge, and the smaller for preheating the heating gases or the air for combustion. The calcining and drying compartments project into the fusion chamber to prevent stoppage of the opening for withdrawing the smaller supply of gases.

L. A. COLES.

Cements, paints, etc. [containing alkali silicates]. A. G. RODWELL, W. ROPER, and A. J. C. HART (B.P. 304,355, 19.10.27).—The products comprise sodium silicate and sodium nitrate solutions, together with mineral pigments.

L. A. COLES.

Production of porous building materials. G. RICHTER (B.P. 305,415, 25.4.28).—To the concrete mixture is added a metal powder, *e.g.*, aluminium, which has been activated so as to decompose water readily, together with a non-hydrolysing aggregate, *e.g.*, flaky ashes.

M. E. NOTTAGE.

Production of artificial stone. E. A. P. RENOUF (F.P. 629,450, 4.5.26).—The product comprises a hard nucleus (silica) and a softer matrix bound together by chemical reaction; *e.g.*, a mixture of alumina with pieces of quartz (5 mm. diam.) which have been coated with milk of lime and dried is heated at 1150–1200°.

L. A. COLES.

Bituminous emulsions and their use in the coating of substances with bitumen. COLAS PRODUCTS, LTD., W. S. WHITING, and A. G. TERRY (B.P. 301,544, 2.9.27).—Mineral aggregates are coated with bitumen emulsions in the presence of a strongly electrolytic alkali salt, excluding ammonium salts, *e.g.*, 0.5–2.0% of sodium chloride calculated on the weight of the emulsion, which may be added to the mineral aggregate or to a mixture of it with the emulsion. A filler capable of absorbing water, *e.g.*, up to 4% of soft limestone, may be added to the aggregate.

L. A. COLES.

Bituminous composition for paving. G. COBB (B.P. 302,808, 28.12.27).—A single-layer paving composition contains 3½–5% of bitumen and crushed rock aggregate.

L. A. COLES.

Imitating marble and other surfaces. E. C. R. MARKS. From OXFORD VARNISH CORP. (B.P. 304,509, 12.3.28).

Manufacture of road-surfacing materials or compounds. J. Y. JOHNSON. From AMIESITE ASPHALT CO. OF AMERICA (B.P. 304,226, 14.10.27).

Manufacture of cement. T. RIGBY (U.S.P. 1,699,451, 15.1.29. Appl., 20.7.25. U.K., 28.7.24).—See B.P. 243,410; B., 1926, 129.

Manufacture of concrete. M. LÉVY, Assr. to "PRODOR" FABR. DE PROD. ORGANIQUES SOC. ANON. (U.S.P. 1,699,705, 22.1.29. Appl., 2.11.22. Switz., 16.11.21).—See B.P. 201,650; B., 1923, 977 A.

Treatment of wood and like porous material. M. SMITH (U.S.P. 1,699,635, 22.1.29. Appl., 29.12.25. U.K., 17.8.25).—See B.P. 265,643; B., 1927, 328.

Waterproofed materials (B.P. 303,935).—See V. **Binding agents having hydraulic properties** (B.P. 297,416).—See VII. **Cement with metallic lustre** (B.P. 303,938).—See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Coalescence of non-metallic inclusions in steel. C. H. HERTY, JUN. (Proc. Eng. Soc. W. Penn., 1928, 44, 259–264).—Since the larger a non-metallic particle is, the faster it will rise to the surface of the steel bath, it is desirable that inclusions formed on deoxidation should coalesce as rapidly as possible in order to be eliminated from the steel before it is cast. If the inclusion has a m.p. higher than that of the bath, no coalescence can be expected, although the particles may coagulate in the steel to form clouds of particles; and, in general, the lower the m.p. of the impurity the greater is the rate of coalescence. Surface tension also affects coalescence to a minor degree. Manganese and double silicates have a m.p. which allows of ready coalescence, but additions of aluminium to killed or rimmed steel generally form inclusions having a higher m.p. than that of the steel, and particles of alumina can be observed in the metal. It is possible that the use of double deoxidisers, *e.g.*, magnesium-aluminium alloys, might produce cleaner steel by reason of the formation of more fusible compounds.

C. A. KING.

Troostite. A. E. CAMERON and I. F. MORRISON (Trans. Roy. Soc. Canada, 1928, [iii], 22, III, 289–291).—A series of heat-treatments on a medium-carbon (0.5% C) and a high-carbon (0.8% C) steel were carried out. The specimens were subjected to drastic quenching operations (+1000°) followed by tempering and drawing operations at 100°, 200°, 300°, 400°, and 500°. The same area in each steel was microscopically examined after each drawing operation and the photographs are reproduced. It appears that globular troostite is not a decomposition product of martensite, but is a primary decomposition product of austenite. The troostite-like structures formed by tempering operations are in reality free-grained sorbite, and may be developed from either primary troostite or martensite. Martensite tends to sorbitise as soon as the slightest reheating is

applied. The carbon content determines the granulation of troostite. In the 0.5% carbon steel troostite remains unchanged until tempered at 300°, whilst with 0.8% carbon steel the globular troostite has granulated appreciably at 200°.

A. J. MEE.

Determination of molybdenum in steel in presence of tungsten and vanadium. I. KASSLER (Z. anal. Chem., 1929, 76, 113—120).—After dissolution of the steel in hydrochloric acid and oxidation with nitric acid, the solution is evaporated at 80—90° (but not boiled) in order to obtain the tungstic acid free from molybdic acid. Separation is effected not by filtration and washing, but by removal and filtration of an aliquot part of the diluted solution. If much molybdenum is present a small quantity may be retained by the tungstic acid, in which it may be determined colorimetrically.

H. F. GILLBE.

Experimental demonstration of the refining of metals by oxidation. J. SILBERSTEIN (Ind. Eng. Chem., 1929, 21, 172).—In the refining of iron or copper by oxidation, losses of metal occur as oxide if the concentration of impurities is small. The effect is capable of demonstration on the laboratory scale with lead containing 0.2—0.5% Na. On melting and stirring, a white film of sodium oxide appears. If the film is removed as it is formed the colour becomes bluish, lead as well as sodium being oxidised. Some time is necessary before the sodium is completely oxidised.

C. IRWIN.

Reduction of the alkaline-earth sulphates in metallurgical operations. G. CHARPY and L. JACQUÉ (Compt. rend., 1929, 188, 473—475).—The authors' experiments indicate that sulphates of calcium and barium and probably of strontium are equally effective for the sulphidation of castings, any differences previously observed being due to the physical rather than the chemical properties of the melt. The sulphur equivalents of the amounts used were 1—4% of the iron present. Reduction of the sulphate to the sulphide which may be due to carbon and/or metallic iron is perceptible at 600° and rapid at 900°.

J. GRANT.

Determination of tin in rubber-coated [tinned copper] wires. A. LEJEUNE (Bull. Soc. chim. Belg., 1929, 38, 25—30).—The rubber is removed from 5 m. of the wire by rubbing with a cloth soaked in benzene, the wire is dried and cut into small pieces, and 3—4 g. of these are dissolved in 20 c.c. of nitric acid (*d* 1.36). The solution is evaporated to dryness, the residue extracted with 200 c.c. of boiling 1% nitric acid, and the insoluble metastannic acid is collected, washed with 1% nitric acid, ignited, and weighed. The copper retained by the precipitate is too small to affect the results, which are usually reported in g./m.² of surface.

A. R. POWELL.

Metals and alloys for dyeing machines. GRUNDY.—See VI. Zinc foil for Leclanché cells. DROTSCHMANN.—See XI. Protective layer in iron pipes. TILLMANS and others.—See XXIII.

PATENTS.

Bonding of fine ores, burnt pyrites, etc. with liquid slags. M. PASCHKE and E. SCHIEGRIES (G.P. 453,469, 2.5.26).—The ore mixed with coke is introduced into molten slag from a blast furnace making

ferromanganese to obtain a product containing 35—40% Fe and 5—10% Mn.

A. R. POWELL.

Working of iron ores, slags, and residues containing lead, zinc, and silver. SOC. MIN. ET MÉTALLURG. DE PENARROYA (F.P. 620,994, 30.12.25).—The material is smelted in a blast furnace the hearth of which consists of a double layer of refractory firebricks. Between these layers and between the upper layer and the side walls outlets are provided for the removal of lead and silver. The furnace is operated with a hot blast so that the upper part of the shaft reaches a temperature of at least 450°, whereby the zinc is volatilised and may be recovered from the fume in settling chambers or by a bag plant.

A. R. POWELL.

Detinning of tinned iron scrap. REYMERSHOLMS GAMLA INDUSTRI AKTIEBOLAG, Assces. of A. G. SUNDBERG (Swed. P. 59,566, 9.7.24).—The scrap is treated with a solution of copper salts containing alkali chlorides such as is obtained by the atmospheric oxidation of copper ores moistened with chloride solutions. The tin dissolves in the solution with the separation of an equivalent amount of copper.

A. R. POWELL.

Utilisation of scrap metal. T. GOLDSCHMIDT A.-G. (B.P. 282,378, 5.12.27. Ger., 20.12.26).—The metal is formed into bundles which are heated by means of electrical eddy currents. The molten metal may be then worked up into cast iron or iron alloys by the adjustment of the carbon content and/or the addition of other substances.

M. E. NOTTAGE.

Uniting metalically [welding] the seams of aluminium-plated sheet-iron. F. JORDAN (B.P. 304,170, 15.10.27).—The seams are pressed together and heated suddenly by electrical resistance, so that the aluminium coating at the joint is converted into oxide and the iron base melts and forms a tight weld.

A. R. POWELL.

Alloy steels. TERNI (SOC. PER L'INDUSTRIA E L'ELETTRICITA) (F.P. 625,055, 24.11.26. Italy, 30.10.26).—Steels for use in the manufacture of turbines contain 0.05—1.5% C, 7—25% Cr, 0.05—20% Ni, 0.1—3% Si, and 0.1—2% Mn.

A. R. POWELL.

Heat-treatment of [alloy] steel. F. KRUPP A.-G. (F.P. 626,672, 23.12.26. Ger., 1.4.26).—Nickel, chromium, silicon, vanadium, or tungsten steels are quenched from above 900°, to obtain a martensitic structure, and annealed at 600—650° to render them suitable for use in vessels handling gases and vapours at high temperatures and pressures.

A. R. POWELL.

Manufacture of resistance-surface or abrasive-resisting alloys and castings, with special reference to a cast-metal grinding ball. L. J. BARTON (B.P. 277,004, 31.8.27).—The alloy contains 98.22—95.54% Fe, 0.8—1.3% C, 0.3—1.0% Si, 0.6—1.8% Mn, less than 0.18% (S + P), and none or 0.5—3.0% Cr. It is made by melting steel scrap under a calcium silicate slag, adding ferromanganese until the slag becomes yellowish-green in colour to eliminate gases, deoxidising with ferrosilicon, and adding small amounts of aluminium as the metal is being poured into moulds.

A. R. POWELL.

Production of metals or alloys together with refractory materials. T. R. HAGLUND (B.P. 302,087, 18.2.28).—Iron ores relatively rich in silica are smelted in an electric furnace to produce iron or ferrochromium, using magnesite and bauxite as fluxes in such proportions that the slag contains less than 15% SiO_2 , more than 15% MgO , more than 20% Al_2O_3 , and up to 35% Cr_2O_3 . The slag is cast into suitable shapes in moulds or is granulated, ground, and mixed with binding materials for the manufacture of refractory bricks.

A. R. POWELL.

Hardening of high-speed steel. W. STAUFFER (B.P. 289,082, 27.3.28. Switz., 23.4.27).—During heating, the articles to be hardened are packed in at least one non-caking carbide (*e.g.*, silicon carbide) in a finely-divided form, such carbide having no appreciable carbonising action even at the temperature required (1380°) for hardening the steel. Graphite powder may be added to the carbide.

M. E. NOTTAGE.

Hardening metal articles by nitrogenisation. P. F. M. AUBERT, A. J. P. and H. A. M. DUVAL (AUBERT & DUVAL FRÈRES) (B.P. 290,214, 5.3.28. Ger., 10.5.27; cf. B., 1923, 273 A).—The parts of the articles not to be treated are coated with a metal or metal alloy (*e.g.*, of tin) and then covered with a layer of water-glass and aluminium powder, after which either the whole articles or the tinned parts are dipped in a bath of sodium or potassium nitrate heated to about 400°.

M. E. NOTTAGE.

Flotation process. C. SHAPLEY (U.S.P. 1,689,693, 30.10.28. Appl., 13.10.27).—Non-metallic minerals such as fluorspar, rutile, or corundum may be floated by immersing the ore in a 1% soap solution for a short period, draining off the excess solution, and agitating the ore in the usual flotation machines, using water free from any material which will decompose the soap.

A. R. POWELL.

Oil flotation process [for ores]. A. W. HAHN (U.S.P. 1,690,225, 6.11.28. Appl., 5.10.26).—Soluble lower salts or oxides of metals present in the ore pulp are oxidised by treatment with bleaching powder in presence of an alkali prior to adding a flotation agent.

R. BRIGHTMAN.

Roasting of sulphide minerals. J. B. READ and M. F. COOLBAUGH (U.S.P. 1,694,794, 11.12.28. Appl., 25.5.26).—Mineral sulphides and oxidising gases are passed in the same direction through a roasting furnace so that sulphates are formed and, as the charge approaches the end of the furnace, a predetermined weight of the original sulphide is added to convert a desired proportion of the sulphate into oxide. L. A. COLES.

Recovery of metals and metal compounds which are soluble in ammoniacal liquors. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 301,342, 24.8. and 7.11.27).—Oxidised copper ores are roasted at 600° in a current of chlorine or, after admixture with magnesium or ammonium chlorides, in a current of air. The product is leached with water, then with ammoniacal ammonium chloride solution to extract copper, silver, zinc, nickel, and cobalt. The filtered solution is heated with copper turnings to remove silver, then distilled to recover the ammonia and precipitate the carbonates or hydroxides

of the other metals. The precipitate is converted into metal and the constituents are separated by electrolysis.

A. R. POWELL.

Protection of copper apparatus against corrosion by carboxylic acids. SOC. CHIM. DES USINES DU RHÔNE (B.P. 284,685, 15.9.27. Ger., 4.2.27).—The ingress of oxygen into the apparatus during use is prevented by adding reducing agents, *e.g.*, sulphurous acid or its salts, covering the surface of the liquid with an inert oil, or passing a stream of inert gas through the apparatus.

A. R. POWELL.

[Refining of] nickel and nickel alloys. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 304,371, 20.10.27).—The molten metal or alloy is treated with 0.25–0.5% of vanadium, niobium, or tantalum, preferably in the form of a 30% alloy with nickel, to remove the sulphur, then with 0.1% of magnesium to remove gases and oxides immediately before casting.

A. R. POWELL.

Production of aluminium-silicon alloys. A. PHILLIPS, E. BARON, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (B.P. 305,311, 24.11.27).—Alloys containing 5–15% Si may be prepared in the "modified" state by adding about 4% of anhydrous powdered carbonate or bicarbonate of an alkali metal to the molten alloy, heating to 1000°, and stirring.

M. E. NOTTAGE.

Treating impure lead and lead alloys. Treating mixtures containing alkali salts of certain metals [slags from lead refining]. Treating metal particles [lead alloys]. G. E. DALBEY and T. P. HANFORD, ASSRS. to STANLEY CHEM. CO. (U.S.P. 1,693,639–1,693,642, 4.12.28. Appl., [A, c, d] 14.1.27, [B] 4.5.26).—(A) Antimonial lead containing tin is melted under a flux containing sodium antimonate, from which the antimony is displaced by the tin in the alloy. (B) Tin, arsenic, and antimony are removed from lead by melting it with litharge and adding gradually a concentrated solution of sodium hydroxide so that the material on the surface of the metal remains in a powdery condition. (C) Alkaline slags obtained in the above treatments are leached with a small quantity of water to remove most of the unchanged sodium hydroxide, then with more water to dissolve sodium stannate, zincate, and arsenate. The residue is heated with metallic tin and more water to decompose plumbates and plumbites, the resulting stannate solution being added to that obtained by leaching, and the insoluble sodium pyroantimonate is separated from the metallic lead by elutriation. The solution of sodium salts is treated with sodium sulphide to remove zinc and lead and then with carbon dioxide to precipitate stannic acid and leave a final solution of sodium carbonate and arsenate. (D) Finely-divided lead alloys are refined by digestion with concentrated sodium hydroxide solution in the presence of an oxidising agent to remove tin, arsenic, and antimony.

A. R. POWELL.

Activation of refractory metal filaments. J. W. MARDEN, ASSR. to WESTINGHOUSE LAMP CO. (U.S.P. 1,698,850, 15.1.29. Appl., 31.3.23).—A refractory metal filament to which a mixture of potassium thorium fluoride and powdered aluminium is applied and retained

by a suitable binding material is heated *in vacuo* so that the fluoride reacts with the aluminium and thorium is deposited on the filament. J. S. G. THOMAS.

Removal of vapours from annealing boxes in bright-annealing processes. T. STASSINET (G.P. 450,186, 13.12.25).—To prevent condensation of moisture or hydrocarbon vapours in the outlet tubes of annealing boxes through which a reducing gas or vapour is passed, these tubes are provided with an electric heating coil packed in insulating material surrounding the tube.

A. R. POWELL.

Refining or purifying metals and alloys. F. W. CORSALLI (B.P. 303,094, 23.6.27. Addn. to B.P. 297,759; B., 1928, 899).—Liquid metal in a collecting vessel is submitted to a swinging movement while being heated. Such a vessel may consist of a forehearth with a stepped wall over which the metal enters, the hearth being capable of a rocking motion; or the container may be bath-shaped, pivoted on a central knife-edge, and operated by eccentric discs or crank rods. C. A. KING.

Electrodeposition of [iron-nickel] alloys. STAND-ARD TELEPHONES & CABLES LTD. FROM WESTERN ELECTRIC CO., INC. (B.P. 304,354, 19.10.27).—To obtain electrolytically an alloy of 79% Ni and 21% Fe an electrolyte containing 212 g. of nickel sulphate crystals, 22 g. of ferrous sulphate crystals, 18 g. of nickel chloride crystals, 2.5 g. of ferrous chloride crystals, 25 g. of boric acid, and 180 g. of sodium sulphate crystals per litre is used with iron and nickel anodes, the surface areas of which are in the ratio of 15 : 85. The bath is operated at 50° with a current density at the anode of 4 amp./dm.²

A. R. POWELL.

Electrolytic production of metals, especially magnesium. A. JESSUP (F.P. 626,497, 1.4.26).—The electrolysis is carried out in a cell divided into two compartments by a vertical wall terminating a short distance above the bottom of the cell, which is covered with a molten alloy of higher sp. gr. than the electrolyte. The electrolyte may be the same in both compartments or may consist of a single fused salt in one compartment and a mixture in the other. The cathode consists of a molten metal which floats on the surface of the electrolyte, and the anode of a carbon or graphite plate; the alloy serves as the cathode in one compartment and the anode in the other. A. R. POWELL.

Chromium plating [bath]. SOC. NANTAISE ELECTRO-CHIM. ET MÉTALLURGIQUE (F.P. 625,313, 11.3.26).—The electrolyte contains more than 50% of chromium trioxide together with small quantities of chromium salts. The throwing power of this electrolyte is claimed to exceed considerably that of the solutions generally used. A. R. POWELL.

Coating metallic or non-metallic bodies with (A) rhodium, iridium, and ruthenium, (B) with osmium. S. G. S. DICKER. FROM N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 304,396 and 304,580, 31.10.27).—The body to be coated, *e.g.*, a wire of tungsten, molybdenum, or nickel, is passed continuously between two electrical contacts in a chamber filled with a carbonyl chloride derivative of (A) rhodium, iridium, or ruthenium, or (B) osmium. As it passes between the contacts the

wire is heated by resistance to 500–600°, whereby a bright coating of the required metal is deposited on its surface. A. R. POWELL.

Uniting metalically the seams of aluminium-plated sheet iron. F. JORDAN (U.S.P. 1,699,434, 15.1.29. Appl., 12.11.27. Ger., 15.5.26).—See B.P. 304,170; B., 1929, 249.

Brass composition and its production. O. JUNKER (U.S.P. 1,699,665, 22.1.29. Appl., 23.7.25. Ger., 8.5.24).—See B.P. 243,998; B., 1926, 133.

[Spraying nozzle for introducing water into] cupola furnaces. A. J. STEPHENS. FROM VULCAN-FEUERUNG A.-G. (B.P. 304,887, 16.12.27).

Electric annealing furnace (B.P. 292,139).—See XI.

XI.—ELECTROTECHNICS.

Zinc foil for the Leclanché cell. C. DROTSCHMANN (Chem.-Ztg., 1929, 53, 29–31, 50–52, 66–68).—The best conditions for, and effect of, amalgamation are discussed. With dry-cell mixtures containing 0.2–0.8% of mercurous chloride, the rate of amalgamation depends on this proportion, on the temperature, and on the time allowed, but is sufficiently rapid at 75° (3–10 min.). Amalgamated zinc electrodes in such mixtures, with no positive electrode, showed no corrosion after remaining sealed for 12 months. When the positive element is present, a dotting over of the zinc surface occurs rapidly and reaches a maximum, with amalgamated electrodes, in 48 hrs. Etching or washing of the zinc surface before amalgamation does not influence this change, which has no apparent effect on keeping the cell. Strips of copper, iron, lead, and tin soldered to the zinc foil caused no observable corrosion in 12 months in dry cells containing mercurous chloride. The presence of lead in the zinc foil is also not harmful, but foil containing arsenic suffered rapid corrosion. The presence of arsenic in the positive electrode mixture was found to affect the cell very badly. Hammering or deforming the surface of the zinc foil causes corrosion, but moderate local heating, such as is necessary for soldering, does not itself increase corrosion. The blackening of the zinc, observable in all cells partially or completely discharged, could not be explained by analysis of the black material, which contained only 1% Pb and 1% Hg. Chemical examination of the metal yields no information as to its utility for dry cells, and tests based on the rates of temperature rise when pieces of the same weight and surface are treated with dilute sulphuric acids in Dewar flasks are suggested. S. I. LEVY.

Effect of gases on the resistance of granular carbon contacts. P. S. OLMSTEAD (J. Physical Chem., 1929, 33, 69–80).—The resistance of granular carbon contacts, outgassed at 200° to a pressure of 10⁻⁵ mm., has been measured by a special technique which is described, together with the effect of adsorbed nitrogen on the resistance. The contact resistance between carbon granules increases with the gas pressure over the range 10⁻⁵ to 760 mm. The effect is reversible and varies exponentially with the pressure of the gas over this range. At constant pressure the increase in resistance diminishes with a rise in temperature. It

is concluded that the resistance of a granular carbon contact is partly determined by the amount of gas adsorbed at the surfaces of contact. At constant pressure, also, the contact resistance decreases with an increase in voltage, but at constant pressure and constant voltage the resistance decreases with a rise in temperature. These facts indicate that at least a part of the effect of applied voltage in reducing the resistance is due to the raised temperature of the contact. The possibility of an electrostatic effect, however, is not excluded. The effect of gas held in the pores of the carbon on contact resistance is also described and discussed. The falling resistance-voltage curve shows a sudden break at a higher voltage which depends on the kind of carbon used. This voltage is considered to mark the temperature at which further decomposition of the granules begins. At this point there is a linear relation between the resistance and the percentage of hydrogen found by analysis in the carbon.

L. S. THEOBALD.

Manufacture of magnesia insulating materials.

F. E. M. BUSCHMANN (Chem.-Ztg., 1929, 53, 31–32).—The preparation of solid insulating blocks from basic magnesium carbonate and asbestos is described.

S. I. LEVY.

Comparator for determination of p_H of coloured solutions. McCANDLISH and HAGUES.—See I. Voltol. WOLF.—See II. Ultra-violet light-transmitting glasses. STARKIE and TURNER.—See VIII.

PATENTS.

Electric furnace for annealing metals. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE GES.M.B.H. (B.P. 292,139, 7.5.28. Ger., 15.6.27).—Electric resistance heating elements, if desired mounted on frames, are arranged between the articles to be annealed, and similar heating elements are arranged on the inner walls of the furnace. J. S. G. THOMAS.

Magnesium primary cell. R. T. WOOD, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,696,873, 25.12.28. Appl., 5.8.25).—A magnesium electrode is placed in a neutral electrolyte containing a strong, soluble, oxidising agent which reduces corrosion of the magnesium on open circuit. J. S. G. THOMAS.

Mercury rectifier. C. KRÄMER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,694,328, 4.12.28. Appl., 24.4.26. Ger., 9.5.25).—An anode and a cathode are mounted in an evacuated vessel containing and interconnected with a receptacle for supporting hygroscopic material and having a porous wall so that water vapour may be admitted to, and mercury excluded from, the receptacle. J. S. G. THOMAS.

Oscillation generator. W. H. BURTIS (U.S.P. 1,692,074, 20.11.28. Appl., 21.4.27).—A piezo-electric crystal is coated with a silicate binder to retain a powdered metal, which forms an electrode.

F. G. CLARKE.

Dry-coating of lamp bulbs. D. S. GUSTIN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,698,845, 15.1.29. Appl., 10.12.27).—An electric discharge is passed between electrodes which are respectively arranged near the external and internal surfaces of a hollow vessel con-

taining the coating-material, the vessel being rotated during passage of the discharge. J. S. G. THOMAS.

Vacuum device and method of cleaning up residual gases therein. D. MACRAE, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,699,112, 15.1.29. Appl., 9.1.23).—A galvanised iron electrode arranged in a bulb exhausted to a moderate degree is heated to cause evaporation of the zinc thereon and so effect a clean-up of the residual gases in the bulb.

J. S. G. THOMAS.

Electrode [for water-resistance steam generator]. H. W. MATHESON and J. C. SMITH (U.S.P. 1,693,794, 4.12.28. Appl., 23.8.24).—A hollow, inverted, frusto-conical electrode forms an upwardly-enlarging steam conduit within a tubular discharge zone from which steam is directed into the conduit. J. S. G. THOMAS.

Electric gas-purifying plant. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE G.M.B.H. (B.P. 278,710, 4.10.27. Ger., 5.10.26).—The rectifier for producing high-tension continuous current consists of metallic bodies, e.g., copper plates, coated on one side with a metallic oxide, e.g., copper oxide, with interposed metal foil, such as lead foil, mounted alternately in series and in intimate contact with each other. [Stat. ref.] J. S. G. THOMAS.

Precipitation electrode for electrostatic gas-purifying apparatus. ELEKTRISCHE GASREINIGUNGS GES.M.B.H. (G.P. 451,041, 5.1.23).—The electrodes consist of metal rods shaped like the blades of a turbine and are so placed in the gas conduit that the gas stream impinges on them in a horizontal direction and after flowing over their entire surface leaves them in the same direction. A. R. POWELL.

Temperature-measuring device. F. S. STICKNEY, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,867, 18.12.28. Appl., 6.4.27).—The cold junction of a thermocouple is connected to a Wheatstone bridge having side resistances respectively of high and low temperature coefficients of resistance. An electrical indicator is placed in the thermocouple circuit, and temperature changes in the cold junction are made sluggish by a heat-storage device. J. S. G. THOMAS.

Electrodes for indication and determination of the chemical composition of liquids. H. S. HATFIELD (B.P. 302,490, 5.12.27).—In apparatus for the determination of electrode potentials, two electrodes of relatively large area are placed on either side of a removably-mounted, thin, porous plate forming one wall of a chamber containing one liquid and making external contact with another liquid. The electrodes may be connected to actuate a low-resistance galvanometer etc. J. S. G. THOMAS.

Determining gaseous carbonic acid. H. SCHMICK, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,695,031, 11.12.28. Appl., 20.7.26. Ger., 1.9.25).—A quartz filter and a gas containing carbon dioxide are arranged between a source and a receiver of radiation, and means are provided for determining the action of heat rays at the receiver. J. S. G. THOMAS.

Dielectric substance and its manufacture. A. MOUILLEFARINE (U.S.P. 1,696,501, 25.12.28. Appl.,

18.2.27. Fr., 18.2.26).—Wood is dyed to resemble ebonite by immersion for 24 hrs. in a liquid composed of Basic Black, acetic acid, and sodium chloride, heated to 100° for the first 4 hrs., and then cooled. The wood is further heat-treated and impregnated at 150–300° with an insulating mass of bituminous shale. It is then dried and varnished. J. S. G. THOMAS.

Mica plate [insulating] compositions. MICA INSULATOR Co., Assees. of J. M. COFFEY (B.P. 288,260, 13.3.28. U.S., 5.4.27).—Mica splittings are bound together by a reaction product of glycerol and phenol or its homologues. Thus the binder may be prepared by heating 10 pts. of phenol with 7 pts. of glycerin with a small amount of sulphuric acid at 160–190°, the reaction being complete when about 3½ pts. of water have been distilled. The acidity of the resultant rubbery mass is neutralised with sodium carbonate.

J. S. G. THOMAS.

Leading-in conductor. S. RUBEN (U.S.P. 1,692,998, 27.11.28. Appl., 8.9.27).—A layer of cuprous oxide is integrally formed with a metal body. J. S. G. THOMAS.

Primary battery. W. A. F. BLEECK (U.S.P. 1,699,580, 22.1.29. Appl., 4.11.25. Austral., 23.2.25).—See B.P. 248,011; B., 1927, 820.

Pasting of electric accumulator plates. C. A. VANDERVELL & Co., LTD., R. C. PATERSON, and P. J. BEDDOUS (B.P. 305,302, 17.11.27).

Electric discharge tube. S. G. S. DICKER. From N. V. PHILIPS' GLOELAMPENFABR. (B.P. 304,423, 23.11.27).

Cathodes for gas-discharge vessels. SIEMENS & HALSKE A.-G. (B.P. 279,497, 21.10.17. Ger., 23.10.26).

Insulation [tape] material for electric apparatus. H. D. SYMONS (B.P. 302,397, 17.9.27).

Hydrocarbons from coal and water (B.P. 283,177).—See II. **Iron-nickel alloys** (B.P. 304,354). **Electrodeposition of magnesium** (F.P. 626,497). **Chromium plating** (F.P. 625,313).—See X.

XII.—FATS; OILS; WAXES.

Determination of water and fats in materials rich in fats. N. D. PRIANISCHNIKOV and S. M. TELNOV (Z. anal. Chem., 1929, 76, 161–166).—The method, especially suitable for the evaluation of oil-containing seeds etc., combines the volumetric determination of water by distillation with benzene and the removal of fats by benzene in one operation. The apparatus consists of a flask (100 c.c. capacity) with a bent neck the upper portion of which is vertical and enlarged to hold a glass extraction thimble containing about 5 g. of material. Solvent vapours, after passing the thimble, enter the usual arrangement of reflux condenser and graduated tube for the volumetric determination of water. The loss in weight of material is equal to the fat and water content. To obtain satisfactory separation of water the solvent (b.p. 95–120°) should contain 4–6% of isobutyl alcohol, when the observed volume of water should be multiplied by 0.945, or 4–6% of *n*-amyl alcohol, when the correction factor is 0.992. J. S. CARTER.

Examination and identification of fats and fat mixtures. II. Clouding point and setting curves. B. LUSTIG and G. BOTSTIBER (Biochem. Z., 1929, 204, 46–61; cf. B., 1929, 178).—Every species of fat examined was shown to have its own clouding point and its own setting curve with definite maximum and minimum. By an examination of these curves fats may be identified and adulterations detected.

J. H. BIRKINSHAW.

Fatty acids and component glycerides of some New Zealand butters. T. P. HILDITCH and (Miss) E. E. JONES (Analyst, 1929, 54, 75–95).—The composition of the mixed acids in 3 samples of New Zealand butter was determined by the following method. Steam-volatile acids were removed by prolonged distillation in steam, recovered from the aqueous distillate by extraction with ether, and fractionally distilled. The non-volatile acids were separated into solid and liquid acids by the lead salt method and converted into the methyl esters, which were quantitatively fractionated by distillation under reduced pressure. The approximate percentage composition of the acids was: butyric 3, hexoic 2, octoic 1, deoic 2, lauric 4, myristic 11, palmitic 28, stearic 9, oleic 33–34, linoleic 4–5%. There was consistent evidence of the presence of a small percentage of acids less saturated than oleic acid. The butyric acid content as determined from the Kirschner value is about 20% in excess of the amount actually present. The percentage of fully saturated glycerides was determined by the permanganate method (B., 1928, 791), and the acids thus combined were fractionated *via* the methyl esters. The amount of saturated glycerides was about 30%; the fatty acids contained therein were the same as those in the whole fat and in proportions not greatly dissimilar. There was, however, a tendency for the lower acids to associate with the unsaturated acids and a slight compensating concentration of higher acids in the fully-saturated glycerides. These glycerides are probably of the complex, mixed type. The remainder (70%) of the butter fat consisted of mixed glycerides of saturated and unsaturated acids, the molecular proportions being 104 mols. of saturated acids to 100 mols. of unsaturated acids. The approximate composition of the butter fats might be thus formulated: mixed fully-saturated glycerides 30%, mixed mono-oleo-disaturated glycerides 36% and mixed dioleo-monosaturated glycerides 34%, all the saturated acids being comparatively evenly distributed throughout the whole fat. J. S. CARTER.

Deterioration of soap-nicotine preparations. II. C. C. McDONNELL and J. J. T. GRAHAM (Ind. Eng. Chem., 1929, 21, 70–73; cf. McDonnell and Nealon, B., 1924, 803).—Commercial soap-nicotine preparations decrease in nicotine content on storage, the loss in the case of hard soaps being greater than with soft soaps. Excess of alkali or fat has no appreciable influence. When air is not excluded the loss of nicotine from soap preparations made from drying oils is due mainly to oxidation; in the case of those made from non-drying oils it is caused by volatilisation of the nicotine. Both hard and soft soaps, whether made from drying or non-drying oils, when packed so that they were completely protected

from the air, suffered no loss of nicotine during 2 years' storage. F. R. ENNOS.

Reactions of soya-bean oil. A. RICHARD (Ann. Falsif., 1928, 21, 579—582).—As little as 10% of soya-bean oil may be detected in admixture with arachis or olive oil by emulsifying 10 c.c. of the sample with 1 c.c. of nitric acid, and leaving the tube on a boiling water-bath for 15 min. Pure olive or arachis oil solidifies, as under these conditions the oleic acid appears to be transformed into its isomeride, and in 24 hrs. solidification is practically complete. Soya-bean oil shows no trace of solidification, and the viscous mass, instead of being yellow, is reddish-brown. After determining the presence of olive or arachis oil the proportion may be found from the iodine value.

D. G. HEWER.

Coffee berry oil. L. VON NOËL (Pharm. Zentr., 1929, 70, 69—77; cf. Meyer and Eckert, B., 1911, 139).—By modifying the ordinary methods of separating the unsaponifiable constituents, the fatty acids of the oil were obtained white and without rotation; they consist of 29% of palmitic, 3% daturic, 14% of carnaubic, 2% of oleic, and 50% of linoleic acids, but decoic acid was not found. The oil is very rich in unsaponifiable constituents, which have a very high specific rotation; such rotation, however, is not due to the presence of sterols. The fatty acids are very little affected by roasting the coffee. S. I. LEVY.

PATENTS.

Manufacture of esters of aliphatic, aromatic, and alicyclic alcohols and fatty acids by the action of alcohols on oils or fats or on glyceryl esters of fatty acids. SOC. ANON. ASSOC. PARISIENNE POUR L'IND. CHIM., Assees. of E. DESPARMET, R. WEIL, and F. SCHMITT (F.P. 615,953, 1.10.25).—The oil, or fat, or fatty acid is heated with the alcohol in the presence of a catalyst, such as a mineral acid or a salt, e.g., trisodium phosphate. E.g., 500 kg. of copra, 1000 kg. of butyl alcohol, and 10 kg. of hydrochloric acid (*d* 1.16) are heated under reflux for 10 hrs., a further 10 kg. of hydrochloric acid are added, and the heating is repeated. Finally a third portion of acid is added and the same treatment repeated. The excess of butyl alcohol is distilled off, and the residual liquid, which separates into two layers, is transferred to a separating funnel. The lower layer contains the glycerol and the upper the butyl ester. The latter is removed, washed, neutralised, and purified by distillation *in vacuo*. A. R. POWELL.

Production of fatty bodies soluble in water. R. VIDAL (B.P. 289,001, 22.12.27. Fr., 19.4.27. Addn. to B.P. 280,193 and 285,473; B., 1929, 137).—Ricinoleic acid is used instead of oleic acid etc. as described previously, and the alkali hypochlorites or hypobromites are added repeatedly in small quantities. L. A. COLES.

Production of soap from sulphurised rosin. L. DE MOLTKE-HUFFELDT (F.P. 629,214, 17.2.27).—Rosin (500 g.) and sulphur (125 g.) are heated together for $\frac{1}{2}$ hr. at 155—160°. The mass is finely powdered, 195 c.c. of sodium carbonate solution (*d* 1.32) diluted with 700 c.c. of water are added, and saponification is effected at 100°. A transparent, yellow, gelatinous soap

is obtained. Before treatment with sulphur the rosin may be dissolved in a medium such as turpentine or rosin oil. W. J. BOYD.

Manufacture of linoleum. G. E. HEYL, and HYCOLITE LIQUID WALLPAPER MANUF. CO., LTD. (B.P. 303,292, 7.2.28).—A mixture of 60—50% of finely disintegrated, dry, oil-resistant paper or fibrous pulp (e.g., wood pulp) and 40—50% of a mineral lubricant such as china clay or plaster of Paris is substituted for part of the cork usually employed. The paper or pulp may be rendered non-absorbent of oil by soaking in varnish or a drying oil and drying prior to final disintegration. W. J. BOYD.

Treatment of tall oil. OEL- U. FETT-CHEMIE GES.M.B.H. (B.P. 281,637, 21.11.27. Ger., 4.12.26).—Tall oil is separated into fatty acid and resinic acid by distillation in a high vacuum (below 8 mm. of mercury), the process being interrupted when resinic acid begins to distil over. E. LEWKOWITSCH.

Impregnating compositions (B.P. 302,710). **Lubricating oils** (U.S.P. 1,691,882 and 1,691,654).—See II. **Esters** (B.P. 302,411).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Paint resistant to flue gas. W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1929, 34, 1061—1063).—A preliminary account is given of investigations on the durability of paints designed for exposure to hot flue gases. Indications are obtained that, contrary to expectation, the introduction of tung oil does not improve the durability of such paints. Adhesion to red lead priming paint is, in general, very poor, no advantage being shown over tests carried out directly on bare metal. The relative efficiency of various pigments, e.g., white lead, lamp black, graphite, is discussed. Types of failure are illustrated. S. S. WOOLF.

Radium, with special reference to luminous paint. A. T. PARSONS (J. Oil and Colour Chem. Assoc., 1929, 12, 3—25).—A general account of the chemical and radioactive properties and the manipulation of radium is given, and its applications to medical practice and chemical investigations are indicated. Radioactive luminous paints consist of a phosphorescent body, e.g., suitably prepared zinc sulphide etc., the luminescence of which is excited by radioactive material, e.g., radium, mesothorium, etc. The manufacture of luminous zinc sulphide is described. Work on the permanence of luminous paint and the recovery of radium therefrom is summarised. S. S. WOOLF.

Physical chemistry of colour lake formation. **IV. Red Congo acid and Congo-red lakes.** H. B. WEISER and R. S. RADCLIFFE (J. Physical Chem., 1928, 32, 1875—1885; cf. B., 1928, 131).—Aqueous solutions of Congo-red contain a red, colloidal anion, which is invisible in the ultramicroscope, but does not pass through a membrane permeable to ions in solution. Replacement of the sodium by hydrogen gives a blue colloidal acid according to the scheme $2\text{Na}^+ + \text{R}''$ (red) $+ 2\text{H}^+ + 2\text{Cl}^- \rightleftharpoons \text{H}_2\text{R}$ (blue) $+ 2\text{Na}^+ + 2\text{Cl}^-$, and since the reaction is partially reversible, Congo-red is an unsuitable indicator in the presence of salts. The blue acid

is slightly soluble in water yielding the red anion, the solubility increasing with a rise in temperature. In the blue sol the equilibria nH_2R (blue) $\rightleftharpoons nH_2R$ (solution) $+ 2nH + nR''$ (red) exist. Mordants such as alumina adsorb the blue colloidal acid forming lakes. The red lake is an adsorption complex of the hydrous oxide and red acid or alkali salt, and not a metallic salt (cf. Bayliss, A., 1911, ii, 886). The existence of the red Congo-acid in the solid state is still open to question.

L. S. THEOBALD.

Determination of turpentine vapour in the air. W. D. BOGATSKI and W. A. BIBER (Z. anal. Chem., 1929, 76, 103—108).—The rose colour produced by turpentine in a hydrogen chloride solution containing 1% of vanillin requires the presence of at least 0.7 mg. of turpentine; on heating to boiling, however, the colour changes to a dull greenish-blue, visible with 0.5 mg. of turpentine. If a hydrogen chloride solution of d 1.19 be employed, spontaneous heating occurs on addition of an alcoholic solution of turpentine, and the colour changes from rose to a bright greenish-blue, which reaches its maximum intensity after about 30 min. Under these conditions 0.14 mg. of turpentine may be detected, whilst if the mixture be heated the colour change is visible with 0.02 mg. Benzene, benzine, acetaldehyde, carbon monoxide, xylene, and phenol give negative reactions, aniline produces a yellowish colour, whereas pyrogallol and resorcinol produce only the rose colour. A positive reaction is given by methyl ethyl ketone, camphor, pulegone, pinene, and dipentene.

H. F. GILLBE.

Method of observing the drying times [of varnish films]. H. WOLFF and W. TOELDT (Farben-Ztg., 1929, 34, 1060—1061).—The spreading of a drop of a solution of a red dye in boiled linseed oil on a drying varnish film reaches a minimum at the "dust-dry" stage, spreading occurring in the film prior to this stage and on the surface subsequently. By plotting "spreading area" against time, the progressive drying of films may be recorded. A permanent record may be obtained by "printing off" the test plates on photographic paper. The "sand method" is recommended for the observation of drying after the "dust-dry" stage. S. S. WOOLF.

Light-coloured condensation resin. H. A. GARDNER, C. A. KNAUSS, and A. W. VAN HEUCKEROTH (Ind. Eng. Chem., 1929, 21, 57).—Equimolecular proportions of phthalic anhydride, triethylene glycol, and tartaric acid on heating at 185—200° for 3 hrs. yield a pale amber resin in the form of a viscous solution, soluble in acetone, alcohol, and chloroform, insoluble in toluol, and compatible with nitrocellulose solutions. If heating is continued for 15—18 hrs. a dark amber plastic mass is produced which is incompatible with nitrocellulose solutions, but compatible with cellulose acetate solutions. These substances are apparently true liquid resins. Exposure tests on lacquers containing equal parts of the dark resin and cellulose acetate gave fairly satisfactory results.

F. R. ENNOS.

PATENTS.

Paints and cements having a metallic lustre. C. NITTINGER (B.P. 303,938, 24.10.27).—A paint or cement consisting of ground coal slag, zinc powder

prepared by grinding electrodeposited zinc, and a suitable vehicle is claimed.

S. S. WOOLF.

Red lead paint. C. D. FAHSEL (U.S.P. 1,697,388, 1.1.29. Appl., 10.11.21. Renewed, 5.9.28).—Red lead is mixed with boiled linseed oil, a light oil varnish, and a paraffin oil.

S. S. WOOLF.

Paints, particularly for the surface-covering of thin rubber sheet material. P. SCHIDROWITZ and D. J. BURKE (B.P. 304,334, 16.7.27).—An improved paint suitable for application to thin flexible material, such as artificial leather, is produced by dissolving a gum resin, e.g., wax-free shellac or a dammar resin, in a low-viscosity solution of a cellulose ester in a high-boiling solvent, e.g., ethyl lactate or benzoate, and intimately mixing this solution with a fine or colloidal suspension of the pigment in a non-drying oil or liquid plasticiser such as tricresyl phosphate. The paste is then thinned with a diluent comprising a liquid hydrocarbon, e.g., toluene or xylene, and an alcohol, e.g., amyl alcohol. Such paint avoids, largely or entirely, the common tendency to cracking and peeling.

D. F. TWISS.

Paint and varnish remover containing an alkali-metal benzoate as thickening agent. B. N. LOUGOVOY, ASSR. to CHADELOID CHEM. CO. (U.S.P. 1,691,771, 13.11.28. Appl., 25.2.26).—An alcoholic solution of sodium benzoate is mixed with acetone or other ketone. Benzene, toluene, or other hydrocarbons, but not chlorinated hydrocarbons, and, e.g., ceresin or paraffin wax, may also be added.

R. BRIGHTMAN.

Pigment for heat- and acid-resistant paints. H. HEMPEL and E. MURNSEER (F.P. 628,543, 4.2.27).—Iron or steel is dissolved in a mixture of hydrochloric and nitric acids, excess of acid is removed, and the paste formed after 8—10 days is dried slowly, ground, and dispersed in water. The water is evaporated and the treatment is repeated. The mass on being dried and calcined yields a brown-red product.

S. S. WOOLF.

Lacquers or enamels. Insulating enamels, varnishes, or like coatings. BRIT. THOMSON-HOUSTON CO., LTD., H. W. H. WARREN, and A. T. WARD (B.P. 303,915 and 303,936, [A] 5.9.27, [B] 22.10.27).—(A) The adhesion of cellulose lacquers to smooth surfaces is improved by the incorporation of about 5% of a "glyptal" resin (in the partly condensed or soluble form) which has preferably been previously plasticised with oleic acid. (B) The flexibility of the usual insulating varnish system is improved by the use of a primer containing a "glyptal" resin which has been plasticised with oleic acid, tricresyl phosphate, etc., and which is cured by heat treatment *in situ*. One or two coats of the primer are applied and stoved for 1 min. at about 310°, subsequent coatings of oil varnish being stoved for 1 min. at 350°. A suitable primer consists of "glyptal" plasticised with 20% of oleic acid, furfuraldehyde, glycol diacetate, butyl phthalate, and coal-tar naphtha.

S. S. WOOLF.

Manufacture of varnish. E. E. WARE, ASSR. to ACME WHITE LEAD & COLOR WORKS (U.S.P. 1,697,213, 1.1.29. Appl., 23.6.24).—A liquid varnish-making ingredient passing continuously through a series of

containers is subjected to predetermined temperature and pressure, and a resin ingredient is introduced at such a rate that uniformity in the proportion of ingredients and in their heat treatment is maintained.

S. S. WOOLF.

Plastic resinous material and its manufacture.

H. M. WEBER, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,692,524, 20.11.28. Appl., 12.1.24).—Formalin is ground with 0.25—1.0 mol. of magnesium oxide and the mixture is heated, *e.g.*, at 70°, with 1 mol. of *m*- and *p*-cresol or of cresylic acid containing not more than about 10% of *o*-cresol. The product is dried, incorporated with filler, and moulded at 160°. If the cresylic acid contains larger amounts of *o*-cresol, 1% of aluminium palmitate may be added to prevent sticking during moulding.

R. BRIGHTMAN.

Resinous composition containing sulphur and its manufacture.

C. ELLIS (U.S.P. 1,690,160, 6.11.28. Appl., 19.12.22).—A phenol is heated to above 160° with at least 1.6 pts. of sulphur in presence of an alkaline catalyst; *e.g.*, potassium carbonate with phenol gives a resin fusible at 130—140°.

R. BRIGHTMAN.

New synthetic resins from aromatic hydrocarbons. P. KRISHNAMURTHY (B.P. 298,939, 6.2.28. India, 17.10.27).—Aromatic hydrocarbons with side-chains (*e.g.*, toluene) are chlorinated in the latter, and the purified product is treated in a solvent (carbon disulphide) with anhydrous zirconium tetrachloride or ferric chloride.

B. FULLMAN.

Manufacture of condensation products of urea with formaldehyde.

H. BARTHÉLEMY, Assr. to SOC. IND. DES MATIÈRES PLASTIQUES (U.S.P. 1,691,427, 13.11.28. Appl., 16.9.26. Fr., 24.9.25).—The syrupy solution of the initial condensation product of urea and formaldehyde is treated with an aliphatic anhydride, *e.g.*, acetic anhydride, preferably in presence of an alcohol.

R. BRIGHTMAN.

Purification of phenol-formaldehyde resins.

F. SEEBACH, Assr. to BAKELITE GES.M.B.H. (U.S.P. 1,697,885, 8.1.29. Appl., 12.2.26. Ger., 6.3.25).—See B.P. 248,726; B., 1927, 532.

Azo dyes (B.P. 302,965).—See IV. **Nitrocellulose of low viscosity** (B.P. 289,387). **Knifing compositions and lacquers** (B.P. 302,615—6).—See V. **Paints** (B.P. 304,355).—See IX. **Soap from sulphurised rosin** (F.P. 629,214).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Diffusion of water through rubber. E. E. SCHUMACHER and L. FERGUSON (Ind. Eng. Chem., 1929, 21, 158—162).—Measurements by manometric and gravimetric methods indicate that the rate of diffusion of water through a rubber membrane is inversely proportional to the square of the thickness, and decreases greatly with increase in hardness of the rubber. Saturation with water increases the permeability, probably due in part to the increased vapour tension of water within the rubber and in part to the decreased hardness. The increase in permeability with temperature is also possibly associated with decreased hardness. No simple relation exists between the rate of diffusion and minor

alterations in composition of the rubber. A simple formula is mathematically derived applicable to the calculation of the rate of diffusion of water at various humidities through rubber samples of various dimensions and composition.

D. F. TWISS.

Colouring of rubber.

W. J. S. NAUNTON (J. Soc. Dyers & Col., 1929, 45, 31—35; cf. B., 1928, 720).—Rubber can be coloured in the uncured state as latex or sheet or in the cured state as cold-cured, soft heat-cured, or hard rubber (ebonite). Rubber colours can be classified as (a) soluble in rubber, (b) slightly soluble, and (c) insoluble in rubber; *e.g.*, inorganic or organic pigments, or lakes. Latex can be coloured with almost any dyestuff which carries a negative charge, and hence does not cause coagulation. Vat dyes can be mixed with the latex and caused to oxidise in an insoluble form by agitation with air. Crêpe or uncured sheet rubber is best coloured by boiling it in an aqueous solution of a basic dye. Cold-cured rubber can also be dyed in a similar manner to crêpe, but usually a pigment is worked into the dry rubber mix. The successful colouring of soft rubber depends on the use of accelerators, as almost all organic dyestuffs are suitable if the rubber is cured in the presence of a super-accelerator. Ebonite, which possesses a natural brownish-black colour, can be coloured by means of a balanced mixture of white pigment and colour.

L. G. LAWRIE.

Practical method for obtaining dry air for humidity control in a rubber laboratory. F. S. CONOVER (Ind. Eng. Chem., 1929, 21, 162—164).—It is believed that zero humidity is more convenient and more satisfactory as a standard condition for the storage of rubber samples, before mixing, vulcanisation, and testing, respectively, than the relative degree of humidity recommended by the Physical Testing Committee of the American Chemical Society (cf. B., 1929, 104). A description is given of storage cabinets ventilated by a current of air dried by passage through a tower of silica gel.

D. F. TWISS.

PATENTS.

Factice. [Rubber substitute.] SOC. ANON. LE TANK (F.P. 629,401, 28.4.26).—Before treatment with sulphur chloride the oils are mixed with talc, calcined magnesia, or a mixture of magnesite with zinc oxide or manganese dioxide. Oils which react feebly with sulphur chloride are treated with magnesium linoleate, aluminium chloride, and zinc oxide. If mineral oils are used, a preliminary chlorination is effected by means of chlorine or hydrogen chloride in the presence of a catalyst. In this way uniformly vulcanised oils are obtained which can be used for the production of resilient tyres.

D. F. TWISS.

Preparation of aqueous emulsions or dispersions of rubber, gutta-percha, balata, or similar materials. J. M. A. TOUCHON (F.P. 628,093, 25.1.27).—A concentrated solution of rubber in a volatile solvent is mixed in a colloid mill with water in which emulsifying or dispersing agents such as soap, saponin, casein, dextrin, glue, or naphthenates may be present. The rubber solution may be replaced by finely-divided rubber to which dispersing agents may be added.

D. F. TWISS.

Production of rubber and other goods with textile insertions attached thereto or embedded therein. ANODE RUBBER Co. (ENGLAND), LTD. From ANODE RUBBER Co., LTD. (B.P. 304,157, 10.8.27).—Fibrous materials and fabrics to be impregnated or coated with organic materials, such as rubber, by agglomeration from dispersions, *e.g.*, by electrophoretic migration, are wetted with the dispersions or similar dispersions prior to the process of impregnation or coating. Such preliminary treatment ensures a high degree of adhesion. D. F. TWISS.

Vulcanisation of plates or articles of rubber. PHIL. PENIN GUMMI-WAAREN-FABR. A.-G., ASSEES. of E. WLCECK (G.P. 452,466, 7.5.25).—Rubber mixtures containing pigments or other ingredients of an inorganic or organic character are vulcanised by successive immersion, repeated, if necessary, in a 4–10% aqueous solution of sulphur dioxide and in saturated aqueous hydrogen sulphide. D. F. TWISS.

Colouring of rubber. IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, W. J. S. NAUNTON, and A. SHEPHERDSON (B.P. 304,376, 21.10.27).—A pigment comprising a vat dye, such as duranthrene-blue, or dimethoxydibenzanthrone, and a substratum, such as clay or light magnesium carbonate, substantially insoluble in water, is prepared by mixing this substratum with the dye in the presence of water (and of a protective colloid, if desired) or of a medium (*e.g.*, benzene) possessing a definite small solvent action for the dye; as a further alternative the dye may be added to the substratum in the form of an alkaline solution of the leuco-compound and then oxidised. The pigment preparations so obtained are then incorporated into rubber, increased tinctorial value being obtained. D. F. TWISS.

Colouring of rubber with lipid-soluble dyes. CONTINENTAL-CAOUTCHOUC U. GUTTA-PERCHA Co. (G.P. 452,340, 21.3.25).—Lipid-soluble dyes or colour bases are dissolved in a molten mixture of rosin and free fatty acid or esters of such acids; the solidified mixture is introduced into rubber in the customary manner and generally dissolves completely, thereby enabling the production of transparent rubber articles. D. F. TWISS.

Moulding of ebonite composition. H. GRAY, Assr. to B. F. GOODRICH Co. (U.S.P. 1,691,347, 13.11.28, Appl., 14.6.23).—A composition comprising 100 pts. of rubber, 40 pts. of sulphur, and 2 pts. of organic accelerator is mixed with 35–65 pts. (per 100 pts. of composition) of clay, asbestos, whiting, gas black, or other inert pigment, and vulcanised, *e.g.*, first in a mould, for 2 min. at 175°, and finally with open heat, at 145°, giving ebonite of high softening point. R. BRIGHTMAN.

Controlling the vulcanisation of rubber and similar materials. S. M. CADWELL, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,698,712–5, 15.1.29. Appl., [A–C] 1.11.27, [D] 25.6.23. Renewed [D] 12.7.28).—See B.P. 218,247; B., 1925, 772.

Separation of mixed substances (B.P. 272,968).—See XX.

XV.—LEATHER; GLUE.

Tanning value of "Takaout" galls from *Tamarix articulata*. F. and M. H. DE BALSAC and A. DEFORGE (J. Soc. Leather Trades' Chem., 1928, 12, 559–564).—Samples of "Takaout" galls, derived from *Tamarix articulata*, Wahl, and grown in various parts of Morocco and Algeria, contained water 11.7–13.1, tannin 41.6–56.3, soluble non-tans 11.4–17.1, insoluble matter 21.5–28.2%; the ratio tans : non-tans was 2.4–4.9. They contain a pyrogallol tannin which penetrates rapidly yielding a soft, full, creamy-white leather. Very little tannin is present in other parts of the tree. The galls are used by the native tanners. D. WOODROFFE.

Effect of heat on wetted, vegetable-tanned leathers. I. W. J. CHATERS (J. Soc. Leather Trades' Chem., 1928, 12, 544–558).—Strips of the leather were suspended in water in a specially devised apparatus, and the shrinkage of the leather was observed as the water was heated. Temperature-shrinkage curves when plotted showed a pronounced shrinkage at 70°, due to gelatinisation of the leather substance. The shrunk leather dried out hard and brittle. The shrinkage was least along the lines of tightness of the leather and most in a direction at right angles to them. The middle layer of a piece of sole leather was scarcely affected, and the flesh part shrank less than the grain. The shrinkage temperature was unaffected by dyeing the leather, but was lowered by stripping it with borax. D. WOODROFFE.

Determination of fat in leather. D. WOODROFFE (J. Soc. Leather Trades' Chem., 1928, 12, 569–572; cf. B., 1926, 926).—Water is not retained by oils if they are heated at 105° for 3 hrs., but this may involve slight decomposition. A greater residue is obtained by evaporating off the solvent after extracting air-dry leather with light petroleum and drying the residue in a water-oven than by drying it at 105° or by extracting leather dried at 105° and drying the extract in either a water-oven or at 105°. The higher figure is attributed to the extraction of moisture from the leather and its retention by the fat extracted if the latter is heated in a water-oven only. The leather should be dried at 105° prior to the fat extraction to avoid this. D. WOODROFFE.

Significance of mechanical wood-joint tests for the selection of woodworking glues. T. R. TRUAX, F. L. BROWNE, and D. BROUSE (Ind. Eng. Chem., 1929, 21, 74–79).—Wood-joint tests are not considered so suitable for grading animal glues for woodworking as viscosity and jelly-strength tests. Different gluing conditions are necessary with different kinds of glue, and no deductions can safely be drawn from tests made under any one set. When a glue is applied under the most suitable conditions, the failure of the joint depends on the strength of the wood rather than on the grade of glue. Wood-joint tests are used to some extent for casein and vegetable glues, as a more refined technique for evaluating them has not yet been developed and as these glues yield stout joints under a comparatively wide range of conditions. F. R. ENNOS.

Nature of adhesion between glue and wood. F. L. BROWNE and D. BROUSE (Ind. Eng. Chem., 1929,

21, 80—84).—The mechanical adhesion theory of the strength of glued wood joints is criticised, and experiments are described which suggest that specific adhesion is essential for satisfactory gluing of wood.

F. R. ENNOS.

Determination of tannins in drugs. LINDE and TEUFER.—See XX.

PATENTS.

Manufacture of synthetic tanning agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,454, 9.1. and 9.7.28).—Hydroxymethyl derivatives obtained by the action of formaldehyde on phenols or their carboxylic acids are condensed with resorcinol below 50° in presence of a condensing agent to yield tanning agents; one or several hydroxymethyl groups may be present in the starting material. In the examples phenol, salicylic acid, and *p*-cresotic acid are used.

C. HOLLINS.

Adhesives for use in shoe-making. BRIT. UNITED SHOE MACHINERY CO., LTD., F. RICKS, and R. A. LINEHAM (B.P. 304,823, 27.10.27).

XVI.—AGRICULTURE.

Scottish soil types with special reference to North-East Scotland. G. NEWLANDS (Proc. 1st Internat. Cong. Soil Sci., 1927, 4, 187—192).—The general characteristics of the profiles of Scottish soils are typical of the podsol type. Further subdivision of this type is possible on the basis of organic and moisture contents, texture, and chemical composition. A. G. POLLARD.

Substances removed by the drainage from a Scottish soil. J. HENDRICK and H. D. WELSH (Proc. 1st Internat. Cong. Soil Sci., 1927, 4, 163—171).—Results of lysimeter experiments with cropped and manured soils are recorded and discussed. The small loss of nitrate from cropped soils even when highly fertilised was noteworthy. The amount of sodium in the drainage water was nearly as great as that of calcium, although the respective proportions of these two bases in the total replaceable bases of the soil were 4.5 and 85%. No appreciable amounts of phosphates were found in the drainage even where phosphatic fertilisers were used. The leached sulphate was increased by liming, presumably as a result of stimulated bacterial decomposition of the soil organic matter. In the chalk-free soil examined considerable amounts of silica appeared in the drainage. A. G. POLLARD.

Mineral composition of the soil as a factor in soil classification. J. HENDRICK and G. NEWLANDS (Proc. 1st Internat. Cong. Soil Sci., 1927, 4, 183—186).—An outline of the process for separating the minerals in the fine-sand fraction of soils by means of bromoform is described. The proportions of the various minerals present serve as a means of classification of soils according to origin. Characteristics of the parent rock are retained in cultivated soils. A. G. POLLARD.

Changes in soil reaction effected by long-continued manuring. B. THOMAS and F. J. ELLIOTT (J.S.C.I., 1929, 48, 51—54 T).—The Tree Field plots at the Northumberland County Experiment Station,

Cockle Park, form the subject of this investigation. Changes in soil reaction and base status produced by the long-continued application of lime, basic slag, superphosphate of lime, dissolved bones, and dung are examined. It is shown that dung, from sheep fed with cake and also superphosphate produce no significant effect on p_H or exchangeable bases, dissolved bones produce only a slight increase in the bases and no effect on p_H , basic slag gives a slight increase in p_H and a large increase in exchangeable bases, and lime produces a large increase in p_H and a very large increase in exchangeable bases.

Influence of the replaceable bases on the soil solution formation in mineralised soils. F. MENCHIKOWSKY and S. RAVIKOVITCH (Soil Sci., 1929, 27, 49—68).—The distribution of replaceable bases varies with the depth of soil examined. Water extracts of soils remove amounts of bases corresponding to the proportions present in the absorbing complex, the relationship being the more definite in soils having the greater absorbing capacities. The amounts of bicarbonate and silicate ions in water extracts of soils increased with the water: soil ratio adopted. It is considered that water brings about the hydrolysis of the aluminosilicate nucleus of the colloidal complex, and that the reaction is independent of adsorbed sodium ions. The relative proportions of cations in water extracts of soils do not alter with the proportion used. The amount of ions of an individual base present in a water extract is the outcome of a definite partial decomposition pressure of compounds of adsorbed cation with the aluminosilicate complex. In highly mineral soils the nature of the soil solution reflects to a greater or smaller degree the composition of the adsorbed bases. A. G. POLLARD.

Nature of the nitrogenous compounds in fungous tissue and their decomposition in soil. A. F. HECK (Soil Sci., 1929, 27, 1—47).—Analyses of a number of fungi show the carbon contents to range between 40 and 44%, with nitrogen 1.5—7%. The carbon: nitrogen ratio of the substrate determines both the amount of mycelium produced thereon and its nitrogen content, which may vary considerably. As the available nitrogen of the substrate is decreased the nitrogen content of the mycelium falls to 2—3%. Further reduction in nitrogen supplies when the carbon: nitrogen ratio is of the order 10 or 12:1 lowers the amount of mycelium produced, without markedly affecting its nitrogen content. In dry fungous tissue 40—70% of the total nitrogen is water-soluble, and of this 80—90% is dialysable. Of the total nitrogen 80—85% is soluble in 0.05*N*-alcoholic soda; 40—65% of the water-soluble nitrogen consists of free amino-acids. No urea was found. After decomposition in soil for 26 days 40—60% of the carbon is liberated as carbon dioxide and 30—40% of the nitrogen appears as nitrate. In the absence of other sources of energy living fungous tissue liberates its own nitrogen by autolysis. Nitrification proceeds quite as rapidly as is the case with other materials of similar nitrogen contents. The rate of nitrification depends on the nature of the source of energy. Where the latter is of simple composition decomposition of fungus tissue is mainly bacterial and much nitrate is

produced. Where cellulosic matter is the source of energy, decomposition of fungous tissue is mainly effected by fungi and the yield of nitrate is small.

A. G. POLLARD.

Conditions of nitrification [in soil]. J. HENDRICK (Proc. 1st Internat. Cong. Soil Sci., 1927, 4, 175—182).—Analysis of drainage water from variously manured soils indicates that nitrification can take place freely and for long periods in acid, chalk-free soil in spite of increasing acidity resulting from continued use of ammonium sulphate. Ammonium sulphate is almost completely nitrified when used alone or in conjunction with other fertilisers, even when relatively large applications are made. Mineral fertilisers stimulate the activity of soil bacteria responsible for the decomposition of organic matter from which ammonia and ultimately nitrate is produced. The conception that the nitrogen of fertilisers may be utilised by soil organisms and stored in an insoluble form within their tissues is discredited. No evidence is obtained that nitrogen in a gaseous form is lost from soil when excessive amounts of nitrogenous fertilisers are used.

A. G. POLLARD.

Tolerance limit of seedlings for aluminium and iron and the antagonism of calcium. J. R. SKEEN (Soil Sci., 1929, 27, 69—80).—Comparison of the growth of *Lupinus albus* and *Phaseolus vulgaris nanus* in the presence of iron and aluminium shows the former to be about three times as resistant as the latter to these toxic ions. The iron ion is 5—7 times as toxic as the hydrogen ion. The antagonism of calcium for iron and aluminium in this respect is shown, and limiting values for soil solution concentrations are discussed and demonstrated in pot experiments. Both iron and aluminium ions tend to become more toxic at 29°. Hydrogen-ion concentration of soils is of little significance as an ecological factor.

A. G. POLLARD.

Deleterious action of smoke gases on vegetation. K. NOACK [with O. WEHNER and H. GRIESSMEYER] (Z. angew. Chem., 1929, 42, 123—126).—The effect of nitrous gases on vegetation as shown by the change in the absorptive power for carbon dioxide is similar to that of sulphur dioxide (cf. B., 1926, 458); in the concentrations found in smoke gases hydrochloric acid is less harmful and ammonia has scarcely any action. A certain minimum concentration of the noxious gas is required to diminish the absorptive power for carbon dioxide, but for concentrations below the minimum there is at first an increase in the amount of carbon dioxide absorbed, which after longer exposure of the plant to light falls again to the normal. The affected plant shows an increased ratio of soluble to insoluble iron compounds; by soaking in a very dilute solution of an iron salt, e.g., ferrous ammonium citrate, its absorptive power is considerably increased, but in no case is it restored to normal. The phenomenon appears to depend on a change in the condition of the iron of the chloroplasts, with a consequent reduction in its catalytic activity.

F. R. ENNOS.

Comparison of the Robinson, International, and Bouyoucos methods of mechanical analysis of non-organic soils, and the analysis of such soil

with and without preliminary treatment with hydrogen peroxide. J. H. DENNETT (Malayan Agric. J., 1928, 16, 374—377).—The International and Robinson's methods are interchangeable and values are obtainable by interpolation of the summation curves of the latter. Clay determinations after 24 hrs.' sedimentation with a sampling depth of 30 cm. by the International method are as accurate as and more convenient than the usual sampling after 8 hrs. at a depth of 10 cm. Hydrogen peroxide treatment is unnecessary in non-organic soils. Bouyoucos' hydrometer method is of value in determining general limiting values of soils for routine purposes, but the total sand is preferably determined by direct sedimentation rather than by difference.

A. G. POLLARD.

Colorimetric determination of phosphorus in acid soil extracts. W. N. C. BELGRAVE (Malayan Agric. J., 1928, 16, 361—371).—Atkins' modification (B., 1924, 483) of Denigès' method fails in acid soils where the proportion of iron to phosphate is great (cf. Greenstreet, B., 1928, 682). Reduction of the ferric salts followed by adjustment of the p_H of the solution leads to a satisfactory determination. The extract from 1 g. of soil, from which silica and nitric acid have been substantially removed by gentle ignition, is dissolved in concentrated hydrochloric acid and heated with 2—3 g. of granulated zinc. When reduction is complete (spot test with thiocyanate), but while hydrogen is still freely evolved, the liquid is filtered through cotton-wool and made up to 100 c.c. To 40 c.c. are added 3 drops of thymol-blue solution and 5M-ammonium acetate solution until the pink colour begins to fade. 1 c.c. of Atkins' reagent-A and 3 drops of reagent-B are added and the original method is proceeded with. An equal amount of thymol-blue is added to the standard solution to maintain similar tints. 0.1 mg. of phosphorus pentoxide may be determined in 50 c.c. of solution in the presence of 1 mg. of silica without decrease in the colour intensity.

A. G. POLLARD.

Improved method of fusion for soils. J. H. DENNETT (Malayan Agric. J., 1928, 16, 372—373).—In certain soils silica and sesquioxides fail to respond to fusion with "fusion mixture." A quantity of potash is prepared by fusion in a nickel crucible till all frothing ceases. A portion is removed by means of a nickel ladle holding 5 c.c. and placed in a small nickel crucible with 0.5 g. of soil. A homogeneous fusion is obtained by heating for 15 min. in a blowpipe flame. The water extract of the melt contains little or no nickel, and is satisfactory for the determination of iron, aluminium, and silica.

A. G. POLLARD.

Examination of agricultural lime. S. REYNAERT (Natuurwetensch. Tijds., 1929, 11, 25—27).—The importance of composition in connexion with price is indicated.

S. I. LEVY.

Determination of total nitrogen in urea-nitrate mixtures. R. LUCAS and W. HIRSCHBERGER (Z. angew. Chem., 1929, 42, 99—100).—About 10 g. of material are quickly weighed and dissolved in 1 litre of water. To 50 c.c. of solution are added 5 g. of powdered iron and 50 c.c. of 1:3 sulphuric acid, a funnel being placed in the neck of the flask. When

reaction has ceased, the flask is heated until the contents become turbid through separation of ferrous sulphate. After cooling, 15 c.c. of concentrated sulphuric acid are added and the concentration is continued until white fumes are freely evolved. The contents are transferred to a distillation flask, decomposed with excess of sodium hydroxide, and ammonia is absorbed in 40 c.c. of 0.5*N*-sulphuric acid. The back-titration is effected with 0.5*N*-sodium hydroxide, using nitrophenol as indicator. Adherence to the quantities indicated is essential.

J. S. CARTER.

Apparatus for incinerating plants for microscopical examination of the ash. O. WERNER (Mikrochem., 1929, 7, 110—115).—The material to be ashed is placed between two perforated aluminium discs which fit into a thin cylindrical framework so as to leave a space between the discs. The framework is carried on an asbestos-covered iron gauze stretched on a rectangular framework over Teclu burners. The ash is mounted for microscopical examination in fused phenol between two cover glasses joined around their circumferences with paraffin wax. For permanent mounting Canada balsam is used in place of phenol.

A. R. POWELL.

Soap-nicotine preparations. McDONNELL and GRAHAM.—See XII.

PATENTS.

Manufacture of a [mixed] fertiliser. I. G. FARBERIND. A.-G. (B.P. 294,654, 26.7.28. Ger., 29.7.27).—Ammonium nitrate is caused to react with a mixture of sodium and potassium chlorides in aqueous solution, the mixture of salts obtained being separated from the liquor.

W. G. CAREY.

Material for the destruction of plant pests. TEKNISKA FABR. JOFUR, N. I. BRUZELIUS, Assees. of E. G. A. WIKSTRÖM (Swed. P. 59,458, 2.3.22).—The material comprises a mixture of tall oil with liquids capable of dissolving fats and waxes, *e.g.*, trichloroethylene, tetrachloroethane, benzol, etc., preferably with the addition of alkaloids, *e.g.*, nicotine.

L. A. COLES.

Utilisation of palm leaves (Austral. P. 1298).—See V. **Superphosphate** (F.P. 626,190).—See VII. **Sewage sludge** (B.P. 305,346). **Insecticides and fungicides** (B.P. 303,932).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Reaction between amino-acids and dextrose. J. A. AMBLER (Ind. Eng. Chem., 1929, 21, 47—50).—This reaction was studied under varying conditions of concentration and heating. If the volatile products, *e.g.*, carbon dioxide and aldehydes, are removed during the reaction the loss of reducing sugar on boiling the solution is relatively high, amounting in certain cases to as much as 10 mols. per mol. of amino-acid; the reaction becomes the more complete the longer the heating, progressive dehydroxylation of the dextrose molecule taking place. When the volatile products are not removed the amount of dextrose destroyed is smaller, probably owing to side reactions of the aldehydes with

the amino-acids. Carbon dioxide is produced only with the more concentrated solutions, and then in equimolecular proportions with the aldehyde. The yield of melanoidin is also the greater in the more concentrated solutions. These highly coloured substances may be a contributing factor to the discoloration of syrup during the manufacture of sugar products, and to the formation of froth in the fermentation of syrups etc. owing to their effect on the surface tension of liquids. F. R. ENNOS.

Comparative precipitation method for the qualitative identification of each of the common gums. W. WEINBERGER and M. B. JACOBS (J. Amer. Pharm. Assoc., 1929, 18, 34—36).—A method is established for the differentiation of the following gums: acacia, tragacanth, Irish moss, agar-agar, Indian gum, and quince seed, by the characteristic precipitate which is formed when each gum is precipitated from aqueous solution by alcohol. The texture, appearance, adherent quality, and manner of settling are different for each gum and are tabulated.

E. H. SHARPLES.

PATENTS.

Better utilisation of the lime in the precipitation of calcium saccharate. C. STEFFEN, SEN., and C. STEFFEN, JUN. (G.P. 452,728, 20.7.24).—Cooling of the sugar solution is continued until lime begins to settle as a deposit, and then with continued cooling the liquid is kept in circulation until the greater part of the lime has settled. The deposited lime sludge is introduced into a cold sugar solution and fresh lime is then added.

W. J. BOYD.

Purification of crystalloid [sugar] solutions. K. KOMERS and K. CUKER (B.P. 283,564, 14.10.27. Austr., 14.1.27).—The active adsorption surfaces of vegetable cell conglomerates, such as beet slices or comminuted sugar cane, which have been chemically treated after lixiviation, are further increased by the addition of finely-divided substances such as active carbon, cellulose, metal powder, metallic oxides or salts, and/or by the formation of finely-divided precipitates in the suspension of such conglomerates, *e.g.*, by addition of iron sulphate to an alkaline conglomerate suspension.

W. J. BOYD.

Manufacture of starch. CORN PRODUCTS REFINING CO. (B.P. 286,289, 16.1.28. U.S., 3.3.27).—An improved cyclic system of starch manufacture is described in which the water is used repeatedly and at the same time the building up of the sulphur dioxide content of the wet starch to an undesirable extent is avoided.

W. J. BOYD.

Composition of gum chicle, rubber, and an aqueous colloid. A. BIDDLE, Assr. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,691,460, 13.11.28. Appl., 22.5.26).—Adhesives are obtained, *e.g.*, by dissolving gum chicle in linseed oil or other organic solvent, and adding casein or other hydrophilic colloid and dispersed rubber or rubber latex. R. BRIGHTMAN.

Crushing machines (B.P. 302,135).—See I. **Spirit and yeast from molasses** (B.P. 283,969).—See XVIII. **Separation of mixed substances** (B.P. 272,968).—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Hydrogen ions in brewing processes. IV. Influence of hydrogen-ion concentration in fermentation. II. G. HAGUES (J. Inst. Brew., 1929, 35, 51—60; cf. B., 1928, 620).—The mechanism of cell reproduction of *Saccharomyces cerevisiae* and *S. ellipsoideus* is almost identical and depends on the activities of two groups of proteolytic enzymes or of two single enzymes the optimum activities of which occur at p_H 4.0 and 8.5, respectively. With *S. Pastorianus I* and *S. Pastorianus III* (*S. validus*), a third enzyme, which may be a phosphatase, with an optimum activity at p_H 6.5 appears to be present in addition to the same groups which stimulate the reproduction of the previous two yeasts. The reproductive capacity of a white torula is probably due to the activity of only one enzyme the optimum of which is at p_H 5.0. When *S. Pastorianus I* and *S. cerevisiae* are grown in symbiosis, the presence of the one species is deleterious to the reproductive power of the other. C. RANKEN.

First chemical phases of hexose decomposition in alcoholic fermentation. A. J. KLUYVER and H. P. STRUYK (Woch. Brau., 1929, 46, 51—55; cf. A., 1928, 398).—Figures are put forward showing that a high maximum rate of evolution of carbon dioxide during the phosphorylation period and a low ratio of hexose-monophosphate to -diphosphate are brought about in cell-free fermentations by increasing the concentration of the yeast macerate. According to the theories of Harden and Henley and of Meyerhof, the molecular ratio of the increase of carbon dioxide, resulting from the addition of phosphate, to the hexosediphosphate formed should be 2:1. The authors obtain in some cases much greater values (up to 11.4) for this ratio, which is only approached as a minimum with high concentrations of yeast macerate. They consider these observations to support their views that Robison's hexosemonophosphate results from the stabilisation of an active hexosemonophosphate, and that hexosediphosphate is formed by the combination of 2 mols. of glyceraldehyde monophosphate. F. E. DAY.

Manufacture of absolute alcohol by the spirit-benzol distillation under pressure. O. VON KEUSSLER (Z. Spiritusind., 1929, 52, 36—38).—Young's method of the manufacture of absolute alcohol by distilling spirits with benzol or similar substances which form a mixture with minimum b.p. has been extended and improved by Merck, who carries out the distillation under a pressure of 10 atm. When the pressure is increased from 0 to 10 atm., corresponding to a rise in temperature of 65—144°, the amount of water removed from the alcohol by the benzol, after allowing for the inter-solubilities of the liquids, is 2.6 times greater. In practice, benzene (b.p. 90—110°) is admixed with the benzol, which then acts as a solvent between the aqueous spirit and the benzene. If the benzene is added in correct amount it prevents the formation of layers and gives a more homogeneous mixture. The danger of separation is greater at lower temperatures, and at 65° the benzol can contain only 20—40% of benzene, whereas at 144° the content may safely amount to

60—65%. The high benzene content of the added liquid gives improved dehydration, facilitating the rapid separation of the distillate into two layers and reducing to a minimum the addition of water to the distillate, which is so necessary when no pressure is used. Since the heat of vaporisation decreases with the rise of temperature and pressure, about 20% less heat is required for every kg. of vaporised liquid when the pressure is raised to 10 atm. The high pressure does not diminish the temperature difference between the top and bottom of the high-pressure column, the difference being 5° at 10 atm. and 4° with no pressure. Under actual working conditions, the yield is increased 2—2½ times at the high pressure, although theoretically it should be 10 times greater. C. RANKEN.

Neutralisation of brewing liquor. D. McCANDLISH and G. HAGUES (J. Inst. Brew., 1929, 35, 61—66).—The brewing liquor is neutralised by sulphuric acid, thorough mixing being ensured by dripping the acid into the water as the latter passes into the mash-liquor tank. Owing to the danger of tank corrosion, if the p_H value of the water is reduced below 7.0 the rate of admixture is so regulated that the p_H of the treated water is approximately maintained at that value. The low p_H value of the brewing water favours the production of a correspondingly low p_H of the final beer, which gives the beer a bacterial stability provided infection is kept at a minimum. In addition, there is rendered possible a low initial value of the p_H of the wort in which the yeast is grown, which secures a better reproduction and aggregation of the yeast and gives rise to a clearer racking beer. C. RANKEN.

Wine refining. M. RÜDIGER and E. MAYR (Kolloid-Z., 1929, 47, 141—155).—The effectiveness of the refining process is a property not of the kind of gelatin employed, but of the nature of the turbidity of the wine. From an analysis of the wine no conclusions can be reached as to the nature of the refining. The following conditions have been found most suitable for the process of refining: (a) the swelling-water should be used for dissolving the gelatin, especially with those poor in gluten; (b) the concentration of gelatin should depend on its gluten content, i.e., not above 0.25% for gelatin rich in gluten, about 1% for medium gluten content, whilst concentrated solutions may be used with gelatin poor in gluten; (c) better results are obtained by allowing the gelatin to age for 1 to 2 days before use; (d) within limits, rise of temperature favours the process; (e) the most favourable method of mixing is either a continuous addition of the gelatin solution to the wine in proportional amounts, or a rapid admixture of the entire volumes. The tannin content of wine decreases with the amount of gelatin added. A part of the refining solution remains in the wine, the nitrogen content of which increases when large amounts of gelatin are used. The viscosity of the wine is less and the surface tension greater with increasing amounts of gelatin addition. The acidity of the gelatin solution and of the wine are important factors, increasing acidity favouring the process within limits. Experiments on the charge of the colloidal particles in turbid wine during the course

of refining indicate that the process is essentially one of electrical adsorption. E. S. HEDGES.

Comparator for determination of p_H of coloured solutions. McCANDLISH and HAGUES.—See I.

PATENTS.

Manufacture of yeast. K. A. JACOBSEN (B.P. 291,770, 8.6.28. Denm., 8.6.27).—The continuous supply of nutrient solution to the fermentation vat is periodically varied in such a manner that an increased quantity is added in time to prevent the occurrence of the yeast-ripening periods. In this way the yeast is continuously kept in a budding condition and at a constant amount. The fermented wort containing the unripened yeast is led into a separate container, in which the yeast ripens under aeration. C. RANKEN.

Manufacture of spirit and yeast by working-up molasses. A./S. DANSK GAERINGSIND. (B.P. 283,969, 20.1.28. Austr., 21.1.27).—Difficulties in the purification of the waste waters are obviated by avoiding during the process, which follows known methods, the use of primary and auxiliary materials containing inorganic sulphur compounds and particularly SO_4 ions. Thus, hydrochloric acid replaces sulphuric acid for adjusting the p_H value of the mash, and yeast nutrients are added in the form of the chloride, carbonate, and phosphate of ammonium in place of the sulphate. C. RANKEN.

Material for denaturing alcohol. H. M. CHABROL (F.P. 626,495, 1.4.26).—Shale oil, b.p. 75–86°, and phenolphthalein are used. L. A. COLES.

Production of glycerin. K. and N. LÜDECKE (U.S.P. 1,698,800, 15.1.29. Appl., 15.7.26. Ger., 21.6.26).—See B.P. 278,086; B., 1927, 921.

Decolorising tartaric acid solutions (F.P. 622,649).—See VII.

XIX.—FOODS.

Peptisation of wheat flour proteins by inorganic salt solutions. R. A. GORTNER, W. F. HOFFMAN, and W. B. SINCLAIR (Cereal Chem., 1929, 6, 1–17).—The amount of protein extracted from any given wheat flour by various salt solutions of equivalent ionic concentration is markedly different, whilst the peptisation of a variety of flours by a single salt solution is equally variable. The salt-soluble protein does not represent a mixture of albumin and globulin, nor yet the non-gluten proteins, some salts extracting only a part of the non-gluten proteins and others very appreciable amounts of the gluten proteins. These differences appear to be determined by the ease of peptisation of the protein of the particular flour, and the specific properties of the anions and cations of the salt solution used.

F. R. ENNOS.

Relation of hydrogen-ion concentration and buffer value to the baking quality of flour. I. E. A. FISHER and P. HALTON (Cereal Chem., 1929, 6, 18–33).—The baking quality of two flours as revealed by the gas production and volume of the loaf produced was practically unaffected by the addition of sufficient tartaric acid to diminish the p_H value by 0.7–0.8; in one

case the fermentation was slightly hastened. A more marked effect was shown on the dough toughness and loaf flavour, but this was only pronounced with large additions of acid. It is concluded that hydrogen-ion concentration is a factor of little importance in bread making, and contributes little or nothing to loaf quality.

F. R. ENNOS.

Effect of dry skim milk on the fermentation and hydrogen-ion concentration of doughs. J. L. ST. JOHN and C. II. BAILEY (Cereal Chem., 1929, 6, 51–59).—Addition of dry skim milk to the baking mixture increases the total production of carbon dioxide and of the carbon dioxide lost from the dough, whilst the rate of increase in the volume of the dough is practically unaffected. The buffer action of dry skim milk is appreciable, the acidity of the doughs containing this ingredient being lower at all stages of the fermentation.

F. R. ENNOS.

Relation of quantity of sodium sulphate to time of digestion in protein determination. C. G. HARREL and J. H. LANNING (Cereal Chem., 1929, 6, 72–78).—The weight of sodium sulphate used in the Gunning method is a vital factor in protein determination owing to its effect on the b.p. of the digestion mixture. For a given source of heat, the time required for complete digestion can be shortened by increasing the ratio of sodium sulphate to acid and *vice versa*. A larger quantity of the salt is necessary with copper as catalyst than with mercury.

F. R. ENNOS.

Determination of thallium [in wheat] and its toxicity. C. STICH (Pharm. Ztg., 1929, 74, 231; cf. A., 1929, 164).—Wheat containing thallium is ignited with sulphuric acid, and the thallium is determined colorimetrically. A mouse fed with 5 grains (1 g.) of wheat containing 1.8–1.9% Tl (as sulphate) died in 12–15 hrs.; rats fed with 20 grains died in 2–3 days.

S. I. LEVY.

Flour colour tests. D. W. KENT-JONES and C. W. HERD (Cereal Chem., 1929, 6, 33–50).—A critical survey of the authors' method (cf. B., 1927, 762) in consequence of certain criticisms by Hooft and de Leeuw (cf. B., 1928, 909).

F. R. ENNOS.

Method of measuring colour in bread. E. GREWE, W. K. MARSHALL, and C. G. HARREL (Cereal Chem., 1929, 6, 60–68).—An application of the Munsell method to the measurement of the colour of crust and crumb of bread.

F. R. ENNOS.

Determination of chlorides in milk. G. RODILLON (Ann. Falsif., 1928, 21, 576–578).—Raquet and Kerlevo's criticisms (B., 1928, 208) of the author's method are refuted.

D. G. HEWER.

Determination of chlorine and sodium in sheep's milk. L. BARTHE and DUFILHO (Ann. Falsif., 1928, 21, 578–579; cf. B., 1928, 385).—Analysis of a series of sheep's milks showed that the chlorine and sodium present per litre was: colostrum period 0.994, 0.191; 1 month 1.207, 0.212; 2 months 1.065, 0.193; and 4 months 1.136, 0.338 g., respectively. The chlorine is present in the same proportion as in cows' milk, and the sodium, varying round 200 mg. per litre at first, then

increases to the amount in cows' milk. Sheep's milk, in respect of chlorine and sodium, approaches most nearly to cows' milk, and ruminants' milk appears to be distinct from other mammalian milks in the high proportion of sodium.

D. G. HEWER.

Rapid methods for determination of bacteria in milk. A. J. J. VANDE VELDE (*Natuurwetensch. Tijds.*, 1929, 11, 9—14).—The methods based on titration of the acid, measurement of the enzyme-reducing power, and direct counting, respectively, are described. The results of a large number of tests by the third method with different culture media are tabulated, and Skar's method is recommended as most rapid and accurate.

S. I. LEVY.

Sampling apples in the orchard for the determination of arsenical spray residue. J. W. BARNES (*Ind. Eng. Chem.*, 1929, 21, 172—174).—Determinations were made of the residual arsenic in a large number of apples sprayed with lead arsenate, the results being expressed as grains of arsenic per apple, per pound of fruit, and per unit area of apple surface. A statistical study shows that in order to obtain a result with a probable error of 5% in the value for the mean arsenical residue per pound of fruit it is necessary to analyse a sample of about 50 apples picked at random.

F. R. ENNOS.

Nutritive value of extracted cattle foods. G. B. VAN KAMPEN (*Chem. Weekblad*, 1929, 26, 98—101).—The disease appearing in cattle fed on soya-bean cake obtained by extraction of the beans with trichloroethylene is found to be due to the removal by this solvent of part of the phosphatide content of the bean. The amount removed corresponds to the reduction in milk yield of cows fed with 4 kg. per day of the cake. The phosphorus-containing organic material removed by trichloroethylene, after a thorough extraction with light petroleum, is found to contain, besides some calcium and a little magnesium, considerable proportions of silica.

S. I. LEVY.

Auxiliary tables useful in the determination of nitrogen in cattle foods. G. JORGENSEN (*Ann. Falsif.*, 1928, 21, 601—604).—For 1 g. sample of food the number of c.c. (ranging from 5.7 to 60.5) of 0.1N-acid used in the Kjeldahl titration are tabulated and arranged in consecutive horizontal lines. By direct reading from other horizontal or vertical columns corresponding values may be obtained from which the percentage of nitrogen and of protein in the sample may be deduced.

D. G. HEWER.

Fatty acids and glycerides of New Zealand butters. HILDITCH and JONES. **Coffee berry oil.** VON NOËL.—See XII.

PATENTS.

Manufacture of a vegetable alimentary product. J. PERINO (*B.P.* 279,487, 20.10.27. *Ger.*, 22.10.26).—Leguminous vegetable material is treated with a solution of alkali phosphates at a temperature below 60°, under reduced pressure, in the absence of free oxygen, and under ultra-violet irradiation. Insoluble alkali hydroxides are added to the mixture and the liquor is then expressed

from the residue. Vitamin-containing herb- or leaf-like or bulbous vegetables are treated with a solution of neutral alkali monohydrogen phosphates under the same conditions, and the resulting liquor is expressed from the residue. The two liquors may be desiccated together or separately. In the treatment of the second class of vegetables ultra-violet irradiation may be omitted.

W. J. BOYD.

Vegetable waste products (*G.P.* 452,810).—See II. **Wrapping of perishable goods** (*B.P.* 304,168 and 304,341).—See V.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Recognition and differentiation of acetone, methyl alcohol, and isopropyl alcohol present together in pharmaceutical preparations. W. MEYER (*Pharm. Ztg.*, 1929, 74, 230—231).—The liquid is subjected to a careful fractional distillation; methyl alcohol and acetone are concentrated in the first fraction, and isopropyl alcohol is found in the second. The latter is divided into two halves, one of which is tested directly, the other, after chromic acid treatment, for acetone; the two results are compared to decide if isopropyl alcohol is present. The second fraction contains methyl alcohol, detected by the usual tests for formaldehyde after oxidation, only if considerable proportions are present in the sample.

S. I. LEVY.

p-Dimethylamidobenzaldehyde as a reagent for organic medicinal substances. H. W. VAN URK (*Pharm. Weekblad*, 1929, 66, 101—108).—A table is given of the colour reactions with the principal medicinal substances. Amino-compounds give yellow to orange colorations, hydroxyl compounds yellow, blue, green, and violet, the colour usually deepening with the number of hydroxyl groups. Pyrrole and indole compounds give colorations. The carboxyl group generally inhibits. Several cases are described in detail. A second table gives results obtained with other aldehydes.

S. I. LEVY.

Examination of therapeutic preparations. K. NEUMAYER (*Arch. Pharm.*, 1929, 267, 27—30).—A lecture dealing mainly with the microscopical examination of hormone preparations etc.

S. COFFEY.

Ammonium acetylsalicylate. "Ammon-aspirin." N. E. WOLDMAN (*J. Amer. Pharm. Assoc.*, 1929, 18, 14—17).—The preparation of salicylic acid, and of acetylsalicylic acid and its ammonium salt is described. Ammonium acetylsalicylate, difficultly soluble prismatic needles, decomp. above 70°, has the same physiological action as a mixture of aspirin and free ammonia in the same proportions.

E. H. SHARPLES.

Determination of potassium iodide in tincture of iodine according to D.A.B. VI. J. WEICHERZ (*Arch. Pharm.*, 1929, 267, 36—42).—The permanganate method described in D.A.B. VI. affords inaccurate results on account of the formation of manganese dioxide and iodic acid. Satisfactory results are obtained by the addition of small amounts of manganese sulphate and

several c.c. of carbon tetrachloride, the latter serving the double purpose of preventing to a large extent the formation of iodic acid and also loss of iodine in the stream of carbon dioxide resulting from the oxidation of oxalic acid.

S. COFFEY.

Determination of tannins in drugs. O. LINDE and H. TEUFER (Pharm. Zentr., 1929, 70, 21—26, 53—60).—The determinations were made by the following methods: (a) precipitation by means of stannous chloride, the washed and dried precipitate being weighed before and after ignition; (b) precipitation by means of copper acetate; (c) extraction by means of hide powder, the difference in weight of organic content of the aqueous solution before and after extraction being observed, and an allowance made for the organic matter yielded to the solution by the powder; and (d) permanganate titration before and after extraction of the tannin from the aqueous solution by means of hide powder. The precautions adopted in each case are described. The values obtained show poor agreement, those obtained by method (c) being highest, and by method (d) generally lowest. Method (c) is regarded as most accurate, and best suited for ordinary laboratory examination. Where alkaloids are also present it is necessary to decompose the alkaloid tannates by means of alkali.

S. I. LEVY.

Odour and constitution among the mustard oils [thiocarbimides]. VI. Natural mustard oils. G. M. DYSON (Perf. Ess. Oil Rec., 1929, 20, 42—44).—The synthesis and properties of allylthiocarbimide and of some other naturally occurring thiocarbimides are described. Specifications of 8 national pharmacopœias for mustard oils are tabulated (cf. B., 1929, 188).

E. H. SHARPLES.

Determination of essential oils by bromometric and thiocyanogen methods. I. H. P. KAUFMANN (Arch. Pharm., 1929, 267, 1—27).—The following iodine values of a number of essential oils, which furnish unsatisfactory results with the Hübl and Wijs methods, have been determined bromometrically by using a solution of bromine in methyl alcohol saturated with sodium bromide: geraniol (329.3), linalool (329.3), terpineol (164.6), anethole (171.3), thymol (338.1), citral (336.6), vanillin (166.9), cinnamaldehyde (192.1), carvone (338.1), geranyl acetate (258.7), linalyl acetate (258.7), whilst the thiocyanogen method afforded the following values: citronellol (162.5), geraniol (164.6), linalool (164.6), terpineol (164.6), anethole (171.3), thymol (169), citral (166.8), citronellal (164.6), geranyl acetate (129.4), linalyl acetate (129.4). The bromometric method cannot be applied to citronellol, cinnamyl alcohol, citronellal, bromostyrene, or coumarin, and the thiocyanogen method fails with geranyl formate and safrole, although in some of these cases normal values may be obtained by carefully adjusting the time and conditions of the reaction. Thiocyanogen does not combine with cinnamaldehyde, coumarin, or vanillin.

S. COFFEY.

PATENTS.

New antirachitic preparations and their manufacture. W., K., L., W., and F. MERCK (E. MERCK)

(B.P. 285,083, 9.2.28. Ger., 12.2.27).—The unsaponifiable constituents of yeast fat or of the corresponding extracts from ergot etc. are esterified and irradiated with ultra-violet light, or irradiated and then esterified. Water-soluble products are obtained when esterification is accompanied by the introduction of residues carrying salt-forming groups.

B. FULLMAN.

Separation of mixed substances. CHEM. FABR. U. SERUMINST. "BRAM." G.M.B.H. (B.P. 272,968, 21.6.27. Ger., 21.6.26).—Separation is carried out at an optimum p_H predetermined for each particular mixture by experiment. For example, the ψ -globulins containing diphtheria antitoxins are freed from the euglobulins which cause anaphylaxis and serum disease by precipitation at p_H 6.4; this can be produced by removal of ammonium sulphate etc. by electro-osmosis. Purification of sugar solutions and glycerol lyes, and precipitation of caoutchouc from latex are other examples mentioned. [Stat. ref.]

C. HOLLINS.

Manufacture of alkylaminoalkylamino-derivatives of aromatic compounds. I. G. FARBERNIND. A.-G. (B.P. 274,058 and 302,984—5, 23.6.27. Ger., [A] 8.7.26. Addns. to B.P. 267,169; B., 1927, 379).—Amino-phenols or aromatic diamines in which the nitrogen or one nitrogen atom carries an alkyl substituent containing an alkylated amino-group, such, for example, as the intermediates required for the production of the poly-aminoquinolines etc. of B.P. 267,169, are valuable against blood-parasites. The compounds may contain an *N*-alkyl side-chain (A) united directly or (B) through nitrogen, oxygen, or sulphur atoms to the alkylated amino-group, or (c) may contain two or more alkylated aminoalkylamino-groups. The following are described: (A) *N*- β -diethylaminoethyl-*m*-aminophenol, b.p. $171^\circ/1.5$ mm.; its *N*-methyl derivative, b.p. $151^\circ/0.5$ mm.; *N*- δ -diethylamino- β -amyl-*m*-aminophenol, b.p. $171^\circ/1$ mm.; *N*-methyl-*N*- β -diethylaminoethyl-*p*-aminophenol, b.p. $164^\circ/3$ mm.; *N*-ethyl-*N*- γ -dimethylamino- β -butyl-*m*-aminophenol, b.p. $175^\circ/2$ mm.; *N*-ethyl-*N*- β -diethylaminoethyl-2-amino-*p*-cresol, b.p. $176^\circ/2$ mm.; *p*-amino-*N*-methyl-*N*- β -diethylaminoethyl-*aniline*, b.p. $161—163^\circ/3$ mm. (by reduction of the *p*-nitroso-compound); (B) compounds of the type $\text{NHR} \cdot \text{C}_6\text{H}_3(\text{OMe}) \cdot \text{OPr}^a(1:3:4)$ in which R is β -(β -diethylaminoethylthiol)ethyl (b.p. $225—227^\circ/5$ mm.), β -(β -diethylaminoethoxy)ethyl (b.p. $186—188^\circ/1.5$ mm.), and β -(ethyl- β -diethylaminoethylamino)ethyl, i.e., $\text{CH}_2 \cdot \text{CH}_2 \cdot \text{N}(\text{Et}) \cdot \text{CH}_2 \cdot \text{N}(\text{Et})_2$ (m.p. $68—69^\circ$, b.p. $145^\circ/9$ mm.); (C) *N*-methyl-*NN'*-di-(β -diethylaminoethyl)-*p*-phenylenediamine, b.p. $180^\circ/1$ mm.; 3:4-diethoxy-*NN'*-di-(β -diethylaminoethyl)aniline, b.p. $203—204^\circ/1.5$ mm.; 3:4-dimethoxy-*NN'*-di-(β -diethylaminoethyl)-*o*-phenylenediamine, b.p. $203^\circ/2$ mm.; 2:6-diisopropoxy-*NN'*-di-(β -diethylaminoethyl)aniline, b.p. $188—190^\circ/2.5$ mm.; 3-diethylamino-6-(bis- β -diethylaminoethyl)aminothioanisole, $\text{SMe} \cdot \text{C}_6\text{H}_3(\text{NMe}_2) \cdot \text{N}(\text{CH}_2 \cdot \text{CH}_2 \cdot \text{N}(\text{Et})_2)_2$, b.p. $198—204^\circ/2$ mm. The following new intermediates are also described: (A) γ -dimethylamino- β -chlorobutane; (B) 3-nitro-6-isopropoxyanisole, m.p. $54—55^\circ$, and the corresponding amine, m.p. $68—69^\circ$, b.p. $145^\circ/9$ mm.; *N*-ethyl-*N*-(β -diethylaminoethyl)- β -aminoethyl alcohol, $\text{NEt}_2 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{N}(\text{Et}) \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{OH}$, b.p. $111—113^\circ/15$

mm. (from β -diethylaminodiethylamine and ethylene oxide), and the corresponding *chloride* (by action of thionyl chloride on the alcohol); (c) 2:6-diisopropoxyaniline, m.p. 63°, b.p. 123°/3 mm. (by reduction of the nitro-compound); 4-amino-3-methylthioldimethylaniline, b.p. 135°/3 mm. (by methylation of the thiophenol).

C. HOLLINS.

Manufacture of new derivatives of aromatic amino-[hydr]oxy- and polyamino-compounds. I. G. FARBERIND. A.-G. and A. CARPMAEL (B.P. 303,093, 23.6.27. Addn. to B.P. 267,169; B., 1927, 379; cf. B.P. 274,058, preceding).—Compounds valuable against blood parasites are obtained by introducing alkylated amino-groups into aromatic polyamino- or aminohydroxy-derivatives, the alkylated amino-group forming part of an aliphatic side-chain or of a hydroaromatic or heterocyclic residue. The following compounds, derived from 1-chloro-2:4-dinitrobenzene, are described: 2:4-di-amino-N-methyl-N-(β -diethylaminoethyl)aniline, b.p. 163°/3 mm. (hydrochloride, m.p. 205°); 3-methoxy-4-isopropoxy-N-(2'-dimethylaminocyclohexyl)aniline, b.p. 173—175°/2 mm.; 3-methoxy-4-isopropoxy-N-(β -hydroxy- γ -1-piperidylpropyl)aniline, m.p. 92—94°, b.p. 225—230°/5 mm.

C. HOLLINS.

Solution of ammonium salt of hydroxyacetylaminophenylarsenic acid. E. FOURNEAU (U.S.P. 1,697,910, 8.1.29. Appl., 30.9.27. Fr., 19.1.26).—See B.P. 264,797; B., 1927, 573.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Dyes in photographic reductions and oxidations.

A. STEIGMANN (Z. wiss. Phot. 1929, 26, 299—304).—An attempt is made to explain the effect of certain dyes on the sensitivity of a photographic plate by supposing that the dye activates the halogen first split off from the silver halide, and possibly also atmospheric oxygen, and that these then bleach the silver nuclei. Alternatively, the dye may be considered to take up hydrogen from the gelatin, and according as its affinity for this active hydrogen is greater or less than that of the silver halide, it will act as a desensitiser or sensitiser, respectively. The action of a sensitiser may also depend in part on the dye protecting the silver nuclei from dissolution by the acid fixing bath.

R. CUTHILL.

Oiling of plates for ultra-violet photography.

A. C. G. BEACH (Nature, 1929, 123, 166).—The plates are treated with a 0.5% solution of vaseline in light petroleum; they may be developed without previous cleaning.

A. A. ELDRIDGE.

Determination of ripening silver. H. KIESER (Z. wiss. Phot., 1929, 26, 305—310).—Schmidt's experiments on the determination of ripening silver in a photographic emulsion (A., 1928, 1340) are criticised. The values found are almost certainly much too high, probably because the grains of the emulsion contain inclusions of silver nitrate, so that the ratio of silver to halogen seems abnormally large. Treatment of the plate with chromic acid before exposure will destroy the ripening nuclei on the surface of the grains, and as

these would normally act as exposure nuclei, the sensitivity on chemical and primary physical development will appear to be reduced. From the fact, however, that on exposing and developing again the sensitivity proves to be normal, it must be concluded that ripening nuclei are present within the grains as well as on the surface.

R. CUTHILL.

PATENTS.

Manufacture of photographic films. J. CHAMBERLAIN and H. PERIAM (B.P. 304,632, 20. and 26.10.27).—To improve the adherence of the emulsion to the celluloid film base the latter is coated with a substratum consisting of a concentrated solution of ethyl cellulose in benzol admixed with an aqueous solution of gelatin containing a small amount of sulphonated castor oil. If the film is required for colour photography, pigment particles or dyed transparent particles, usually of the three primary colours, may be dispersed in the substratum before coating.

J. W. GLASSETT.

Method of colour photography. J. F. SEVENTAHL (U.S.P. 1,697,194, 1.1.29. Appl., 31.1.23).—Separated registered images are produced in a single layer of sensitive emulsion, and, after development and fixing, the colloid is treated with a substance which retards the action of the subsequently applied bleaching agent.

J. W. GLASSETT.

Production of colour cinematograph films.

A. and O. PILNY (B.P. 296,422, 11.2.28. Switz., 1.9.27).—A film negative is first prepared which contains two different colour record images either side by side or one above the other. For printing from this negative the two images are projected simultaneously, but are separated by an opaque screen placed perpendicular to the film at the dividing line of the images. By reflection from a series of right-angled prisms the projected images are made to register on opposite sides of a doubly-coated positive film, which is moved in the same direction and at the same speed as the negative film. After development each side of the positive film is dyed its respective colour.

J. W. GLASSETT.

Method and apparatus for developing exposed photographic surfaces [by atomisation]. A. E. WHITE. From AMER. PHOTOTURE Co. (B.P. 304,636, 22.7.27).

[Producing the effect of relief or depth in] photography. L. M. DIETERICH (B.P. 304,703, 25.7.27).

XXII.—EXPLOSIVES; MATCHES.

Laws of combustion of colloidal powders. Influence of the temperature of the gas surrounding the powder particles on velocity of combustion. H. MURAOUR (Z. physikal. Chem., 1928, 139, 163—168).—A recapitulation of work already published (B., 1928, 141, 770).

PATENTS.

Explosive. A. C. SCOTT, Assr. to MEXCO, LTD. (U.S.P. 1,700,085, 22.1.29. Appl., 21.11.25. U.K., 29.11.24).—See B.P. 248,089; B., 1926, 422.

Coating [with wax the metal case of] ammunition cartridges. J. D. PEDERSEN (B.P. 304,948, 20.3.28).

Matches [in disc form]. F. KÄPPLER, and BRIT. BOOKLET MATCHES, LTD. (B.P. 304,883, 13.12.27).

XXIII.—SANITATION; WATER PURIFICATION.

Spontaneous formation of a protective layer in iron pipes by cold water. I. Adsorption of carbon dioxide by ferric hydroxide. J. TILMANS, P. HIRSCH, and K. SCHILLING (Gas- u. Wasserfach, 1929, 72, 49—53, 78—82).—Ferric hydroxide adsorbs carbon dioxide from water containing free carbon dioxide and sodium, ammonium, or calcium bicarbonate. The hydroxide may be precipitated in the solution or may be added to it, and is more active if precipitated in the cold than from a boiling solution. The quantity of carbon dioxide adsorbed increases with the quantities of iron, and of free and bicarbonate carbon dioxide present, and appears to be definitely related to the concentration of free and combined carbon dioxide. With water containing much carbon dioxide and very little bicarbonate, and *vice versa*, adsorption is practically absent. The production of a protective layer of rust and calcium carbonate in iron water-mains depends primarily on the presence in the water of oxygen, which leads to the formation of ferric hydroxide. This adsorbs bicarbonate ions from the water, liberating an equivalent quantity of hydroxyl ions which unite with the free carbon dioxide present to re-form the original quantity of bicarbonate ions. The net result is a disappearance of free carbon dioxide and, in waters in which this is not originally present in excess, the precipitation of calcium carbonate. W. T. K. BRAUNHOLTZ.

Determination of manganese in water. J. DE GRAAFF (Chem. Weekblad, 1929, 26, 103—104).—The sample is boiled with nitric acid, treated with a small excess of silver nitrate, and boiled with ammonium persulphate until clear; the permanganate formed is determined colorimetrically without filtering. If the sample is kept for some time before the determination, manganese compounds may settle out on the walls of the containing vessel. S. I. LEVY.

PATENTS.

Treatment of sewage sludge. G. H. HADFIELD (B.P. 305,346, 29.12.27).—The drying of sewage sludge for manurial purposes by natural means may be facilitated by thoroughly mixing partly dried press-cake with a proportion of fine, dry boiler ash and powdered quicklime or Portland cement. The mixing may be carried out in a pan with a perforated bottom, the final product being friable instead of sticky.

C. JEPSON.

Purification of sewage and analogous liquids. F. A. DALLYN (B.P. 305,717, 11.11.27).—A mixture of the flocs precipitated from compounds of iron and aluminium is agitated with sewage under strictly aerobic conditions; the iron floc absorbs molecular oxygen, and by releasing it in the atomic state oxidises the organic matter present while the aluminium floc coagulates and precipitates emulsified and unoxidised matter by being brought into intimate contact therewith. After

a short period of settlement, the purified effluent is discharged and the settled sludge, after re-aeration, is introduced into a fresh supply of sewage, and, by initiating the coalescence of the iron and aluminium compounds usually present, continues the process with a minimum of expense. The reaction is non-biological, and any undesirable increase in the oxygen demand, due to fermentation changes, may be controlled by partial sterilisation without deleteriously affecting the process. C. JEPSON.

Treatment of garbage. E. H. BRUNE (U.S.P. 1,700,608, 29.1.29. Appl., 25.9.26).—A mixture of 1 pt. of iron sulphate, 18 pts. of water, and 100 pts. of calcium oxide is added to garbage to deodorise it and increase its food value for animals. H. ROYAL-DAWSON.

Disinfecting agent. K. DAIMLER, F. JUST, G. BALLE, and S. FUCHS, Assrs. to I. G. FARBEIND. A.-G. (U.S.P. 1,691,228, 13.11.28. Appl., 31.7.26. Ger., 3.8.25).—Aromatic sulphonic acids containing at least one side chain of three or more carbon atoms, *e.g.*, sodium butyl-naphthalenesulphonate, dibutyl-naphthalenesulphonate, benzylbutylsulphonate, or pyridine cyclohexyltetrahydronaphthalenesulphonate, are used. R. BRIGHTMAN.

Manufacture of disinfecting, bactericidal, insecticidal, fungicidal, and vermin-destroying preparations. I. G. FARBEIND. A.-G., W. BONRATH, and W. SCHEPSS (B.P. 303,932, 17.10.27. Addn. to B.P. 232,249; B., 1925, 940).—Instead of the arsenic or arsenious salts of basic dyes, mixtures of salts of the dyes with arsenic or arsenious acid or their salts are used. C. HOLLINS.

Automatic and continuous activation of water by means of radium emanation on an industrial scale. SPA-MONOPOLE, COMP. FERMIÈRE DES EAUX ET DES BAINS DE SPA (B.P. 298,915, 15.10.28. Belg., 15.10.27).—The emanation from a slightly acid aqueous solution of a radium salt is transported by means of an inert gas to a scrubber in which it is removed by a spray of water which by operating at varying rates becomes radio-activated to any desired degree up to saturation and may be disposed of as required. The gas leaving the scrubber is freed from carbon dioxide and again bubbled through the radium salt solution. All parts of the apparatus are of glass sealed together to ensure airtightness. The direction of flow in the circuit is maintained by the introduction of suitable mercury seals, and the circulation is induced by the rise and fall of a mercury piston actuated intermittently by a suction pump so as to prevent a too copious evaporation of the radium solution by a continuous stream of gas. An accumulation of explosive gas is prevented by the introduction of a heated tube containing copper oxide in the circuit at a point preceding the scrubber.

C. JEPSON.

Apparatus for charging furnaces for burning refuse or like material. WOODALL-DUCKHAM (1920), LTD., and F. A. HORNBY (B.P. 305,451, 5.5.28).

Gas masks, respirators, etc. J. A. SADD (B.P. 305,080, 29.9.27).

Protective gas-masks. E. DRÄGER (B.P. 289,810, 30.4.28. Ger., 2.5.27).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 12, 1929.

I.—GENERAL; PLANT; MACHINERY.

Air-screening method for determining the grain-composition of powders. H. W. GONELL (*Z. Ver. Deut. Ing.*, 1928, 72, 945—950; *Chem. Zentr.*, 1928, ii, 1128).—An air-screening device is described.

A. A. ELDRIDGE.

Dephlegmation. K. V. KOSTRIN and N. M. AKOPOV (*Neft. Choz.*, 1927, 13, 311—316).—A description is given of the work performed by the following apparatus: a distilling flask carries a vertical tube (30 × 300 mm.) insulated with asbestos (50 mm.) connected by a U-tube to the top of a reflux condenser jacketed with steam. The lower orifice of this condenser is connected by a T-piece to a Liebig condenser or to a gas-trap which can be connected either with a second Liebig condenser or directly to the still.

CHEMICAL ABSTRACTS.

Manometer for determination of gases in vapours. D. F. OTHERMER (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 46—47).—In measurements with steam it is often important to detect admixture with non-condensable gases. For such work, and particularly for use in boilers, a manometer has been applied, of which one arm dips below water level, and the other terminates in a bulb surrounded by steam. This bulb contains air sufficient to give a pressure of about 400 mm. (Abs.), sealed by water, and mercury is used as the indicating liquid. When no air is present in the steam, the bulb functions as an air thermometer and gives its maximum deflection, but the addition of air to the steam tends to equalise the pressure on both sides of the manometer.

R. H. GRIFFITHS.

Apparatus for gasometric determinations in technical analysis. J. HÜBSCHER (*Süddeut. Apoth.-Ztg.*, 1928, 68, 447—448; *Chem. Zentr.*, 1928, ii, 1466).

PATENTS.

Furnaces. A. W. BENNIS (B.P. 302,471, 19.11.27).—The space beneath a firegrate is divided into at least three compartments with separate air supplies, hot air is supplied to the middle compartment(s), cool or medium air to the front, and cool air to the back. The air supplies may be controlled merely by dampers, or separate fans may be used for forced draught. Two air heaters may be used in tandem, the air to the front passing through one only, and to the middle through both.

B. M. VENABLES.

Furnace and method of treating bodies therein. W. G. BERGMAN, Assr. to D. P. OGDEN and E. F. PLUMB (U.S.P. 1,701,223, 5.2.29. Appl., 18.4.24).—A firing chamber is arranged between the inlet and outlet ends of a tunnel-like chamber through which a helicoidal flow of gases is maintained from the outlet end to the inlet end. The escape of flowing gases from the chamber

to the open air is regulated at points arranged along the chamber between the firing chamber and the inlet.

J. S. G. THOMAS.

Regulation of combustion air in furnaces or the like. ASKANIA-WERKE A.-G. (B.P. 287,132, 21.2.28. Ger., 16.3.27).—The fuel is supplied by a number of independently adjustable mechanical devices, each of which operates a measuring fan or pump which draws from a common main, so that the total amount of air or fluid withdrawn will be proportional to the total fuel burnt. In the main is a constriction, the pressure drop across which is caused to regulate the supply of combustion air.

B. M. VENABLES.

Retort. S. B. MATHEWSON and W. K. JAMISON, Assrs. to trustees of LECAR CARBON Co. (U.S.P. 1,700,684, 29.1.29. Appl., 15.2.24).—The apparatus comprises an oven chamber, a furnace communicating with the upper part thereof, flues connecting the chamber to a second chamber below the floor of the first, and an escape stack for the heated gases passing from the lower chamber. A system of piping in the upper part of the oven chamber communicating with a conduit leading outside the chamber can be detachably connected to a number of containers.

A. B. MANNING.

Rotary drum for drying machines. K. FRASER, and W. J. FRASER & Co., LTD. (B.P. 302,541, 10.5.28).—A cylindrical drum is divided by radial partitions into a number of segmental compartments, other shorter radial partitions are provided, and both sets carry lifter blades. Lengthwise the drum may be divided into zones, the partitions in one zone being staggered with reference to the next. A heating jacket may be provided and the heating fluid from the jacket may pass into the interior of the drum in direct contact with the material.

B. M. VENABLES.

Drying of paper or other material. H. W. COWAN (B.P. 279,816, 6.10.27. Can., 29.10.26).—The drying rolls, or other machine from which the moisture evolved normally passes into the atmosphere, is surrounded by a cover, to which heated air is supplied and from which air is exhausted in greater amount, so that some air is withdrawn from the room. Provision may be made for recirculating some of the air withdrawn, with or without condensation of moisture and re-heating.

B. M. VENABLES.

Grinding and mixing apparatus. D. ANDERSON & SON, LTD., and R. O. CHILD (B.P. 302,561, 20.7.28).—Two co-axial propellers have their blades of opposite inclination and are rotated at different speeds. They are submerged in a vessel of the material to be treated and work in conjunction with fixed breaker bars of various shapes.

B. M. VENABLES.

Apparatus for grinding and crushing material. J. K. BLUM (B.P. 288,527, 10.4.28. U.S., 8.4.27).—The apparatus comprises an annular hammer member co-operating with a stationary casing and anvil member. The annulus is given a combined rotating and reciprocating motion by means of an internal element rotating eccentrically, which may be a toothed pinion meshing with teeth on the interior of the annulus. The space between the annulus and the casing diverges in all directions from the point where impact takes place.

B. M. VENABLES.

Mills for grinding, crushing, or pulverising material. A. SONSTHAGEN and G. M. POVERUD (B.P. 303,227, 15.10.27).—During the progress of grinding the material (e.g., cocoa) is well exposed to the air so as to permit the escape of gases. The grinding surfaces may consist of a number of suitably spaced bars, so that the material is alternately ground between bars and deflected in a direction inclined to the general rotation.

B. M. VENABLES.

Combined drying and pulverising apparatus. SIMON CARVES, LTD., Assees. of SOC. ANON. POUR L'UTILISATION DES COMBUSTIBLES (B.P. 297,119, 12.9.28. Fr., 16.9.27).—The rotating shell of a grinding machine is surrounded by a fixed casing forming an annular space through which a current of hot gas and the material to be ground are passed before entering the grinding chamber. The annular space may be divided into two by a cylindrical envelope which rotates with the grinding machine, the material and hot gas then being caused to make two longitudinal passes before entering the grinding chamber. Movement of the material is effected by inclined blades, and means are provided to cause the hot gas to be evenly distributed over the circumference.

B. M. VENABLES.

Multi-stage apparatus for mixing, stirring, emulsifying, etc. A. L. MOND. From I. G. FARBEN-IND. A.-G. (B.P. 301,701, 12.5.28).—The liquid passes through slots in the wall of the axial inlet conduit, then through moving paddle rings and fixed guides alternately in a radial direction.

B. M. VENABLES.

Apparatus for use in the treatment of solids with liquids. W. H. CAMPBELL (B.P. 304,387, 25.10.27).—The pulverulent solid is charged on to a number of diaphragms when in a moderately inclined position, and is subjected to leaching or other treatment while the diaphragms are horizontal; it is discharged by inclining the diaphragms very steeply, preferably one at a time. During charging, the lowest one or two diaphragms are reserved empty until the end; they then receive the material which was retained in the feed passages.

B. M. VENABLES.

Apparatus for the production and dispersion of solids in liquids. W. H. WHATMOUGH (B.P. 304,178, 14.10.27).—Amorphous aggregates are dispersed in a liquid by means of an apparatus comprising a fluid-tight casing in which a beater is rotated at high speed with large clearances and is preferably eccentric to the casing. The beater may be constructed of a number of thin sheets of metal with fine teeth, or of a number of layers of wire gauze from the edge of which wire strands project, or of alternate layers of each.

B. M. VENABLES.

Mixing solids suspended in liquid. DUNCAN STEWART & Co., LTD. From P. HIRSCHFELDER (B.P. 303,339, 11.7.28).—A number of segmental compartments are arranged round a central discharge chamber, an annular feed launder being formed in the wall between the two. A pulp containing several grades of solids is supplied to one compartment, say no. 1, where the coarser settles and the fine material overflows to no. 2, and so on for several compartments. The feed is then charged to no. 2, so that coarser material is deposited on top of the finer. When discharge is required the contents of the compartment are agitated by compressed air and run out through the central discharge chamber.

B. M. VENABLES.

Apparatus for pneumatic separation or grading of solid materials. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 302,400, 8.10.27).—The air from a grinding apparatus is cleaned before admission to the fan in an apparatus designed on the principle of sudden expansion followed by gradual contraction.

B. M. VENABLES.

Separation of solid materials. H. KOLITSCH (B.P. 275,970, 2.8.27. Austr., 14.8.26).—In the "hand-picking" process of separation of materials, especially raw mining products, the controlling factor is differing behaviour to ordinary unaltered light. In this invention ordinary unaltered light is excluded, but all other forms of radiation are claimed to be able to control a separation of individual pieces of the mixture, through already-known devices such as thermo-elements or selenium cells. The radiation may come from an outside source, or may be produced by the minerals themselves, e.g., by heating.

B. M. VENABLES.

Separators [for solids and liquids]. F. H. LUENSE (B.P. 289,847, 28.2.28. U.S., 4.5.27).—The pulp is thrown from the rims of a number of rotating bowls upon the inner surface of a cylindrical screen, through which the liquid passes and upon which the solids are retained. The solids falling down the screen are lifted and thrown upon it again by means of a rotating, inverted cone at the bottom. The rotating parts are driven by a motor through a vertical shaft, and the whole rotating assembly and frame of the motor is supported only by a swivel at the top; the frame of the motor is prevented from turning by a resilient device.

B. M. VENABLES.

Linings for grinding, refining, and mixing machines. S. BRAMLEY-MOORE (B.P. 298,899, 11.10.28, U.S., 15.10.27).—A method of construction is described by means of which the interior lining of a mill may be formed from round, easily-renewable rods.

B. M. VENABLES.

Filter presses. F. B. LEHMANN (J. M. LEHMANN) (B.P. 297,354, 5.12.27. Ger., 19.9.27).—A form of filter press for very high pressures embodying the use of coiled metallic strip as the filter medium is described.

B. M. VENABLES.

Prevention of caking of crystals, powders, etc. A. C. SCOTT (B.P. 305,721, 11.11.27).—The crystals, e.g., of sodium nitrate for use as a fertiliser, are mixed intimately with about 1–5% of a finely-divided insoluble silicate, e.g., asbestos or its mixtures with talc or mica.

L. A. COLES.

Absorbing and recovering the vapours of volatile liquids. RUTH-ALDO Co., INC., Assees. of H. L. BARTHELEMY (B.P. 282,792, 28.12.27. Fr., 28.12.26).—The vapours are absorbed in a wash liquid which readily foams; *e.g.*, acetone vapour is removed from air by means of a solution of ammonium oleate and glycerin in soft water. B. M. VENABLES.

Apparatus for cooling and hardening fluid substances. S. FYHN (B.P. 302,398, 17.9.27).—The substance is spread in a thin layer on the interior wall of a vertical cylinder, which is surrounded by an annular jacket. Cooling is effected by the evaporation of a liquid within the jacket, the cooling liquid being caused to flow in a thin layer on the opposite side of the same wall, and pockets or ribs are provided to promote evaporation. B. M. VENABLES.

Evaporating ovens. CHEM. WERKE VORM. H. & E. ALBERT (B.P. 283,495, 23.12.27. Ger., 11.1.27).—An evaporator, of the type where the liquid passes through a tube rotating in an oven, is provided with another oven and tube above the first through which the waste gases pass and in which the liquid is preheated. B. M. VENABLES.

Vacuum distillation. C. R. BURCH, F. E. BANCROFT, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (B.P. 303,078—9, [A] 21.9.27 and 12.3.28, [B] 21.9.27).—Materials such as mineral oils are subjected to distillation *in vacuo* at the lowest possible temperature consistent with commercial speed of operation, whereby cracking and the formation of azeotropes are reduced. The material is distilled from a number of shallow pools each provided with a cooled condensing surface a short distance away, so arranged that practically all the molecules leaving the pools will strike the condensing surface. The distance between the pools and their condensers may be of the order of 1 cm., and is in any case less than the mean free path of the molecules of residual gas (other than the distillates) at the pressure employed, which may be of the order of 1 dyne/cm.² B. M. VENABLES.

Carrying out exothermic catalytic gas reactions and apparatus therefor. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 303,192, 23.9.27).—The catalyst is contained in a number of tubes of a bundle, the incoming gas is passed first outside the tubes and then through them, and the resistance to flow through the catalyst must be less than the resistance to flow outside the tubes. The tubes are, therefore, very closely spaced, and if round tubes are used the spaces between may be partly filled with inert material such as iron rods, but it is simpler to use square, hexagonal, or other prismatic tubes. When such tubes are used, the tube walls may touch everywhere, in which case some of them will be left empty for the first (heat-interchange) flow and others filled with catalyst for the return (reaction) flow. In a convenient construction the tubes are held in a tube plate at one end only, and are tapered at that end to allow plenty of space for distribution of incoming gas. B. M. VENABLES.

Removal of solid particles from flue gases. M. PRÉTOT and F. ULLMANN (B.P. 281,651, 28.11.27. Fr., 4.12.26).—The washing liquid such as soda solution

is sprayed by compressed air upon turbine-like blades of rotors which are of frusto-conical shape, each inverted with respect to its neighbour. B. M. VENABLES.

Method for obtaining a perfect high vacuum. S. LOEWE (B.P. 280,560, 10.11.27. Ger., 11.11.26).—After evacuation, but prior to sealing-off, a valve-like device within the evacuated bulb is moved to obstruct the stem leading to the pump, so that the gases evolved from the glass when heated for sealing almost entirely move in the direction of the pump. The valve-like device may be of mica, or of glass with an iron or metal tail which is manipulated by a magnet. B. M. VENABLES.

Manufacture of friction material for braking and coupling purposes. KIRCHBACH'SCHE WERKE KIRCHBACH & Co., and M. K. KIRCHBACH (B.P. 284,269, 26.1.28. Ger., 26.1.27).—A mixture of fibrous material and phenolic condensation product is subjected to a pressure in a mould exceeding 3000 atm.; the heat of compression causes pre-hardening sufficient to prevent warping in the subsequent drying and final hardening by heat. B. M. VENABLES.

Mixing machine. W. F. DEHUFF, Assr. to GLEN MIXER Co., INC. (Re-issue 17,215, 12.2.29, of U.S.P. 1,656,665, 17.1.28).—See B., 1928, 175.

Absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 281,710, 3.12.27. Ger., 4.12.26. Addn. to B.P. 270,319).

Absorption refrigerating systems. ELECTROLUX, LTD., Assees. of A. LENNING (B.P. 283,474, 9.1.28. U.S., 8.1.27).

Processes of refrigeration. M. HIRSCH (B.P. 290,305, 12.5.28. Ger., 12.5.27).

Sorting of granular materials. K. C. APPLEYARD, P. W. BEWICKE, (SIR) J. F. LAYCOCK, M. R. PORTALL, and W. E. MANNERS (B.P. 305,608, 5.11.27).

Joints of pipes or other vessels adapted to withstand fluid pressure. VICKERS, LTD., and D. M. ANDERSON (B.P. 305,609, 7.11.27).

Coating of materials (B.P. 304,812). Separation of gaseous mixtures (B.P. 288,216).—See II.

II.—FUEL; GAS; TAR; MINERAL OILS.

Yorkshire, Nottinghamshire, and Derbyshire coalfield. South Yorkshire area. Parkgate seam. ANON. (Dept. Sci. Ind. Res., Fuel Res., Survey Paper No. 13, 1929, 64 pp.).—The Parkgate seam has been examined throughout the South Yorkshire area by means of detailed analyses of pillars of coal cut from the entire thickness of the seam, and taken at 16 points well distributed over the area. The seam consists of three main divisions, "tops," "middle coal" or "hards," and "bottoms." The "tops" and "bottoms" consist of bright coal of the normal bituminous type, and the "hards" mainly of durain with some bright coal in the middle. A portion of the seam adjacent to the roof and floor is sometimes of inferior quality and is not mined. Of the dirt bands associated with the seam the middle dirt band occurs with the most regularity, and shows a

gradual increase in thickness from the centre of the area northwards to beyond Barnsley. The seam exhibits no marked gradation of properties across the South Yorkshire area, but slight differences are noticeable between sections taken in the north and south respectively. The moisture contents of the former are comparatively low, whilst those of sections taken from the east of Sheffield are comparatively high. The percentage of ash, in general, is very low. The volatile matter is comparatively high. The average sulphur content is less than 2%; it is nowhere under 1%, and in one sample reaches 3.17%. The phosphorus content is rather high (up to 0.03%) in certain sections. The calorific value of the seam is high, averaging 14,430 B.Th.U./lb. The m.p. of the ash is also comparatively high, varying from 1320° to 1400°. The cokes obtained in the Gray-King assay were highly swollen in all but three samples. The ash and volatile matter in the "tops" and "bottoms" are generally higher than in the "hards," which contain considerably less sulphur but generally more phosphorus than the rest of the seam. The banded constituents, vitrain, clarain, durain, and fusain, have been separated and analysed in each section. The suitability of the coal for gas making, steam raising, and the production of coke for metallurgical purposes is briefly discussed. A. B. MANNING.

Brown coals and bituminous coals of Hessen, and their utilisation. A. SANDER (Brennstoff-Chem., 1929, 10, 21—26).—The brown coals of Hessen, which are obtained from both underground and surface workings and in deposits up to 60 ft. thick, have a high water content (of the order of 60%) and an ash content varying from 2.5 to 14.9%. On low-temperature carbonisation they yield considerable quantities of tar (about 17—26%, on the dry material), which can be satisfactorily "berginised." Two low-temperature carbonisation plants, of the Kohlenveredlung A.-G. type and having a daily throughput of 600 and 1000 tons of brown coal, respectively, have recently been erected in Hessen, the semi-coke being used for generating power or for domestic purposes. This area also yields coals with a lower bitumen content, which are used directly for firing, and a lignite which is low in ash and gives a fairly robust coke on high-temperature carbonisation.

W. T. K. BRAUNHOLTZ.

Classification of coal. S. W. PARR (Univ. Illinois Eng. Exp. Sta. Bull., 1928, No. 180, 59 pp.).—A discussion of various classifications and the fundamental factors involved. The following formulæ (where A is the ash and S the sulphur) are put forward: pure coal substance ("unit coal") = $1.00 - (1.08A + 0.55S)$; unit B.Th.U. = (indicated B.Th.U. - 5000 S) / $[1.00 - (1.08A + 0.55S)]$; "unit volatile matter" = determined volatile matter - $(0.08 + 0.4S) / (1.08A + 0.55S)$.

CHEMICAL ABSTRACTS.

Swelling of coals. II. G. LAMBRIS (Brennstoff-Chem., 1929, 10, 44—50; cf. B., 1928, 916).—If coal is heated rapidly in the usual crucible tests an outer crust of coke is quickly formed which prevents further swelling. This has led to the erroneous supposition that greater swelling is obtained with more gradual heating. If the coal (1 g.) is spread on a layer of kieselguhr in a crucible

placed in a vertical tube furnace at 900° free swelling takes place and a residue of coke up to 17 cm. high is obtained. The swelling power determined by this method is about three times that found by the Bochum method, using coal of the same degree of fineness, and is still greater if the coal is more finely powdered. The swelling power is also several times greater with rapid heating than with gradual heating, but bears no relation to the coke yield. The volume of the coke is determined by immersing the piece in a mixture of paraffin wax (2 pts.) and liquid paraffin (3 pts.) at 60°, removing, wiping with cotton wool, and, when cold, measuring the displacement in water. The volumes found in duplicate determinations of swelling power agree, on an average, to within 8%. W. T. K. BRAUNHOLTZ.

Use of the Berthelot-Mahler calorimeter. V. KOSTJEJEV (Arh. Hemiju, 1929, 3, 27—29).—Powdered fuels such as coke, coal, and charcoal may, when not compressible into tablets, be burned in starch capsules, the heat of combustion of which is about 3600 g.-cal./g. A supply of these capsules may be kept in hermetically closed jars. R. TRUSZKOWSKI.

Origin and decomposition of carbon disulphide in gas making. II. Carbon-sulphur complex. W. J. HUFF and J. C. HOLTZ (Proc. Amer. Gas Assoc., 1927, 1431—1435; cf. B., 1928, 77).—When a gas oil rich in sulphur was cracked at 650°, carbon disulphide could not be detected in 2 litres of gas; at 730° it was detected, and at 980° considerable quantities were found. Possibly carbon surfaces, when exposed (at temperatures employed in gas manufacture) to gases containing sulphur, at first absorb sulphur, thereby assisting the decomposition of sulphur compounds and decreasing the exposed carbon surface; saturation is approached, and finally carbon disulphide is evolved. The decrease of the tendency of heated refractory surfaces to decompose sulphur compounds is due to the formation of a carbon-sulphur complex, as well as to the presence of carbon. Coke heated in hydrogen behaves like clean pumice, no carbon disulphide being formed at 730° except on continuous use. With higher cracking temperatures deposition of carbon and dissociation of sulphur compounds increase. Conditions favouring the fixation of high local concentration of sulphur, such as sudden heating with decreased diffusion, reduction of exposed surfaces, and the rapid removal of carbon disulphide when formed, favour the production of carbon disulphide. Any process giving the gas extended contact with heated surfaces sufficiently low in, or free from, sulphur will decrease the concentration of carbon disulphide in the gas or eliminate it.

CHEMICAL ABSTRACTS.

Removal of tar fog from gases. A. FISCHER (Chem. Fabr., 1929, 101—102).—In laboratory research on the carbonisation of lignite it is desirable to have a tar-fog separator which is complete in its action, causes no back-pressure, and does not involve the risk of interruption of an experiment. To fulfil these conditions a small Cottrell-Möller apparatus was designed. It consisted of a 50 × 5 cm. steel tube chromium-plated, internally flanged at each end to large Jena flasks, and having an axial wire. It is driven from the ordinary lighting circuit, an oil transformer giving a direct

P.D. of 10,000 volts between the wire and the tube, the latter being earthed. The throughput of gas may vary between 10 and 1500 litres/hr. without the tar separation being disturbed. C. IRWIN.

"Gasol" from carbonisation gas, and use of the gas for cutting and welding. H. SCHOLTZ (Chem.-Ztg., 1929, 53, 159).—The mixture of ethane, ethylene, and higher homologues known as "gasol" was used for welding two sheets of metal 3 mm. thick and 250 mm. long. With a gasol pressure of 0.048–0.05 atm. and oxygen pressure 0.9–1.0 atm. the operation took 6 min. and was accomplished without extinction of the flame or heating of the burner. In other experiments lower pressures and a mixture with 20% of acetylene were successfully used, the weld in the latter case surviving a 180° bending test. Operating costs with the mixture were lower than those with acetylene or gasol alone, but if the original carbonisation gas is used the economy of its employment depends on the thickness of material and on the type of burner. R. H. GRIFFITH.

Pyrogenic decomposition of wood tar in presence of hydrogen and under pressure. V. N. IPATIEV and A. D. PETROV (Ber., 1929, 62, 401–407).—The behaviour of wood tar (Kahlbaum) and of a specimen richer in pitch when heated at about 450° in hydrogen under pressure and in the presence of the oxides of iron and aluminium has been investigated. The total yield of liquid products of pyrolysis is greater than that obtained by distillation with phosphoric acid, and the proportion of hydrocarbons and fractions of low b.p. is higher. The amount of unsaturated hydrocarbons so produced is markedly lower than that obtained by cracking in the absence of hydrogen. Short heating with hydrogen decomposes phenolic ethers, unsaturated cyclic ketones, and acids with predominating formation of aliphatic hydrocarbons and phenols, the latter being produced by decomposition of the ethers and ketones. Protracted treatment with hydrogen reduces the phenols and saturated cyclic ketones to hydrocarbons of the aromatic and polymethylene series. The most favourable conditions for the formation of liquid products consist in the use of an excess of hydrogen in the presence of catalysts at 440–460°. The fractions of high b.p. from the products of pyrolysis are free from the unpleasant odour of the original wood tar; they do not darken when preserved, and may be used as solvents, extractives, or lubricants. In preliminary experiments it is shown that cyclohexanone at 420–440° gives much cyclohexane and small amounts of condensed products. Homoisophorone at 460–480° yields phenols and hydrocarbons, the proportion of the latter increasing as the experiment is prolonged. Pyrocatechol diethyl ether affords benzene and phenol at 430°, whereas palmitic acid at 460° yields aliphatic hydrocarbons.

H. WREN.

Influence of dusty filling materials on the plastic and elastic properties of bituminous substances. H. SUIDA and A. SCHMÖLZER (Petroleum, 1929, 25, 251–254).—For characterising the binding materials used in road construction, the sp. gr., softening point, penetration, and ductility were measured; the filling materials were limestone, quartz, granite, brick,

and asbestos in particles less than 0.065 mm. in diam., which gave a surface of about 2000 cm.²/g. Mixtures containing 5–70% of filler were prepared and tested for ductility, hardness, and ability to bend. The bending test employed a prismatic piece, 15 cm. long and of 4 cm.² cross-section, which was rested on two supports 11 cm. apart and observed under a load of 500 g. In the ball-hardness test a loaded steel ball, 1.8 cm. in diam., was used. It was found necessary to choose suitable temperatures, loads, and times for the different series of binding materials in order to reveal their behaviour most strikingly, so that no direct comparison of all the results could be made. It appeared, however, that increasing amounts of filler altered plastic and elastic properties continuously, granite and quartz giving very similar results. The effects of different fillings on the results of all the tests are discussed for petroleum, coal-tar, and asphalt pitches; in coal-tar pitch there are components capable of reaction with substances like chalk, so that its behaviour is different from that of other pitches where such compounds are not present. Particular properties observed with asbestos and brick dust are ascribed to the physical nature of the surfaces involved. R. H. GRIFFITH.

Rapid determination of sp. gr. of semi-solid bituminous substances. S. E. BERKENBLIT (Ind. Eng. Chem. [Anal.], 1929, 1, 39–40).—A method is described for measuring the sp. gr. of substances such as asphalt, which soften at about 50–80°. A test piece, obtained from a cylindrical brass mould of standard dimensions, is washed and dried before weighing, and then immersed in water in a special apparatus where the volume of water displaced can be read accurately and rapidly by adjustment of level in a movable burette. The measurements are all carried out at 25°. R. H. GRIFFITH.

Paraffin content of road asphalts. H. BURSTYN (Petroleum, 1929, 25, 257–260).—Asphalts which contain paraffins are not generally suitable for road construction except when these substances are not crystalline. In Polish and Russian petroleum pitches the content of paraffin is generally high, and two preparations from Galicia, known as Galbit and Molfalt, are found to contain 0.46 and 0.22%, respectively, of paraffin when analysed by Marcusson's method; they are also of good quality practically. R. H. GRIFFITH.

Improved fractionation for cracking processes. D. B. KEYES (Chem. & Met. Eng., 1929, 36, 78).—Vapours from a cracking still, or from the top of a stripping column in which crude tar is "reduced" by means of a countercurrent of steam, are led into the bottom of an ordinary fractionating column, indirectly heating liquid accumulating at that point before entering the side of the column through a valve where the pressure is reduced to 50–100 lb. Cold feed is heated by passing through a coil acting as a reflux condenser at the top of the column, and is then introduced into the column near its middle, where it is stripped of its volatile products. Re-cycle stock passes out of the bottom, whilst gasoline vapours from the top of the column are condensed and pass to a gas separator in accordance with usual practice. H. S. GARLICK.

Process for obtaining lubricating oils and vaseline from petroleum residues. M. MORGENSTERN (Motorenbetrieb, 1929, 2, 3—4).—The process consists in warming the petroleum residues by means of low-pressure steam, whereby practically quantitative separation into oil and asphalt is accomplished.

W. S. NORRIS.

Knock ratings of pure hydrocarbons. A. W. NASH and D. A. HOWES (Nature, 1929, 123, 276—277).—Since unsaturated hydrocarbons differ widely in their reactivities towards oxidising agents, it was anticipated that they would possess widely different anti-knock values; comparative experiments with cyclohexane, benzene, cyclohexene, toluene, Δ^8 -pentene, trimethylethylene, diamylene, diisobutylene, and "ethyl fluid" show that this is the case. cyclohexene has anti-knock properties equivalent to those of benzene, whilst the others, particularly diamylene and diisobutylene, are far more effective. The advantages of the two last-named compounds (cf. B.P. 253,131; B., 1927, 835) are considered. Of the olefines examined, those which are the more stable towards bromine, sulphuric acid, potassium permanganate, and potassium dichromate are the more effective in suppressing knocking.

A. A. ELDRIDGE.

X-Ray quantitative analysis of lead tetraethyl gasoline. R. H. ABORN and R. H. BROWN (Ind. Eng. Chem. [Anal.], 1929, 1, 26—27).—The passage of X-rays through solutions of lead tetraethyl in gasoline is measured by means of an ionisation chamber and galvanometer circuit, a Coolidge tube working at 35 kv. and 20 milliamp. being a suitable source of radiation. A Bragg-type ionisation chamber is used, and the liquid under examination is contained in a brass vessel with aluminium windows. It is found that the deflection produced in the galvanometer is directly proportional to the concentration of lead tetraethyl, but in order to obtain accurate measurements careful control of current and of the type of radiation is necessary.

R. H. GRIFFITH.

Production of power alcohol from waste vegetable materials. A. C. THAYSEN and L. D. GALLOWAY (Ann. Appl. Biol., 1928, 15, 392—407).—A discussion of the fermentation of grass, straw, husks, etc. by *Bacillus acetoethylicus*. The yield (gals./ton) of alcohol and acetone is: *Hyparrhenia glauca* 18, corncocks 22.5, esun grass 17, *Phormium tenax* 10, rice husks 14, rice straw 19, wheat straw 16, *Andropogon gayanus* 20, *A. tectorum* 22, sorghum stems 17, papyrus (air-dry) 16.5, sisal-hemp residue 4.8, sugar-beet residues 24.7.

CHEMICAL ABSTRACTS.

Transformations of fatty acids during geological epochs. I. G. L. STADNIKOV and A. WEIZMANN. II. G. L. STADNIKOV and Z. VOZSCHINSKAJA (Brennstoff-Chem., 1929, 10, 61—63, 81—82).—See B., 1929, 158.

See also A., Mar., 271, **Ignition temperature of combustible gaseous mixtures** (PRETTE and LAFITTE), 294, Pityrol (SHOYAMA; OGATA; HIDAKA).

Crude petroleum and seeds. MURPHY.—See XVI.

PATENTS.

Method and apparatus for washing coke, pan-ash, coal, etc. R. NORGATE (B.P. 306,170, 14.11.27).—

Water is injected as a spiral or helical swirl into a washing chamber from a stationary injector.

Coke oven. C. OTTO (U.S.P. 1,698,272, 8.1.29. Appl., 9.4.27. Ger., 3.4.26).—Each vertical heating flue of a coke oven is provided with a removable burner nozzle, which can be taken out through an opening in the top of the oven in alinement with the flue. The opening can be closed by a removable cover brick and a loosely fitting filler brick which is packed in with insulating material. Both bricks are provided with sight holes in alinement with the flue.

A. B. MANNING.

Coke oven. W. M. PERSON (U.S.P. 1,698,651, 8.1.29. Appl., 26.4.23).—A coke oven with horizontal flues and with a pair of regenerators directly below the coking chamber is provided with independent passages connecting the ends of the flues with one regenerator, the passages from the top of the regenerator to the level of the flues being substantially vertical. The other regenerator is similarly connected with the other ends of the flues.

A. B. MANNING.

Intermittent carbonising plant. N. J. BOWATER, A. H. LYMN, and CHAMBER OVENS, LTD. (B.P. 304,859, 7.11.27).—The smoke and fume emitted when the chambers of an intermittent carbonising plant are charged are removed through a flue or pipe which is attached to the charging mechanism and is so arranged that its lower end is situated closely over an aperture in the chamber additional to those through which the chamber is charged, whilst its upper end is situated under one of a number of orifices in a manifold from which the smoke can be exhausted by natural draught or by means of a fan. The orifices in the manifold are provided with valves so that those not in use may be kept closed.

A. B. MANNING.

Gasification [of coal]. P. D'H. DRESSLER, ASST. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,698,493, 8.1.29. Appl., 10.4.20. Renewed 9.5.25).—Coal is distilled in a continuous tunnel kiln, heat being transmitted to the coal by circulating the gases through a space which is partially separated from the main passageway, and in which they are heated. A decarbonising liquid is mixed with the gases passing through the separated space.

A. B. MANNING.

Coating or impregnation of granular, pulverulent, or powdered materials with liquid compositions, and manufacture of briquettes etc. H. NIELSEN and B. LAING (B.P. 304,812, 26.10.27).—The granular or powdered material is allowed to fall through a chamber countercurrent to an atomised stream of pitch or other impregnating composition. The material is first passed through a dryer or preheater in which its temperature is raised, or lowered, if the initial temperature is too high, to that most suitable for effective coating. Steam and/or hot gases are admitted to the lower part of the chamber, the temperature of which is so regulated that any surplus of the coating material is revolatilised. The coated material is passed continuously to a mixing device and thence to the briquetting apparatus.

A. B. MANNING.

Gas-producing and oil-recovery plant. Combustion chambers. F. L. DUFFIELD (B.P. 305,253 and

305,270, 1.11.27).—(A) Pulverised fuel is first heated with air at 200–250° in order to destroy its tendency to become viscid, is then carbonised, the volatile products being collected, and is finally gasified. The first two processes are carried out in an annular space surrounding a dome-shaped chamber in the lower part of which lie the combustion and gasification chambers. The coal passes down a perforated spiral trough in the annular space, which is divided into two compartments. In the upper compartment heated air is blown through the coal as it descends, whilst in the lower the coal is carbonised in a current of hot producer gas. Part of the carbonised product is fed, together with air, as primary fuel to the combustion chamber; the remainder, with gas as the carrying medium, is supplied as secondary fuel to the gasification chamber. The hot gases produced pass from the top of the dome-shaped chamber through the tubes of a steam generator and thence downwards through a heat-exchanger in which the gas used for the carbonisation process, the air for combustion, and the air for the oxidation of the fuel are preheated. (B) A primary supply of powdered fuel and air is introduced into a combustion chamber with a velocity of 150–300 ft./sec., and secondary air is introduced, with the same velocity, at two different levels. The zone of combustion within the chamber is regulated by suitably apportioning the secondary air supply between the inlets at the two levels.

A. B. MANNING.

Means for producing gas. G. L. REICHELME, Assr. to GASIFIER Co. (U.S.P. 1,699,543, 22.1.29. Appl., 7.2.20).—Finely-divided carbonaceous fuel is supplied to a vertical, cylindrical gas producer into which air under pressure is blown tangentially. Mechanical means are provided for synchronously controlling the supplies of fuel and air.

A. B. MANNING.

Separation by intense cooling of gaseous mixtures containing acetylene. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 288,216, 4.4.28. Ger., 4.4.27).—Acetylene, the presence of which causes difficulties in the separation of gaseous mixtures by intense cooling, is removed quantitatively therefrom by passage of the gas over heated catalysts, e.g., partially dehydrated hydroxides of aluminium and iron, the acetylene being thereby decomposed, hydrogenated, or polymerised. The apparatus comprises a reaction chamber containing the catalyst, and periodically reversible regenerators for bringing about heat interchange between the inflowing and outflowing gases.

A. B. MANNING.

Separating from water tarry substances dissolved therein. P. FRANKE (B.P. 279,465, 19.10.27. Ger., 21.10.26).—Tarry substances, e.g., phenols and pyridine, are separated from water by treatment with a tar-oil fraction of boiling range 240–320° and d_{4}^{20} 1.04–1.05, from which the substances are subsequently recovered by successive extractions with alkali and dilute acid, or by distillation under vacuum. In carrying out the process the tar oil and water may be caused to flow through one another in countercurrent, or the water may be passed over a layer of the oil below which is maintained a layer of alkali lye, the oil layer being kept in motion by means of a stirrer without, however, disturbing the relative positions of the layers. A portion

of the oil is withdrawn from time to time, washed with dilute acid, and returned; the lye is similarly withdrawn and replaced by fresh lye as required.

A. B. MANNING.

Manufacture of valuable products from montan wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,552, 5.9.27).—Yellow or white products, similar to carnauba wax but easier to hydrolyse than either carnauba or crude montan wax, are prepared from the latter (1 pt.) by the action of chromic acid and at least 5 pts. of a mineral acid (sulphuric or phosphoric) in 30–70% aqueous solution, at 100–130°. Bleaching and hydrolysis of the wax esters are accomplished simultaneously, the duration of the process and the quantity of chromic acid being so chosen that carbonisation is avoided.

W. S. NORRIS.

Manufacture of liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 293,719, 8.8.27. Ger., 7.8.26. Cf. B.P. 275,663; B., 1929, 45).—The process is carried out by treatment with a large excess of hydrogen or gases containing hydrogen in a circulatory system under a pressure of at least 50 atm. and in the presence of a catalyst consisting of cobalt or a compound thereof with up to 10% of metals of the 6th group of the periodic system or compounds thereof, e.g., cobalt with up to 10% of chromium oxide. Iron or other materials giving rise to the formation of methane and separation of carbon should be excluded from contact with the hot reacting materials.

H. S. GARLICK.

Manufacture of low-boiling oils and cyanides. J. C. CLANCY (B.P. 304,421, 21.11.27).—High-boiling hydrocarbon oils are brought into intimate contact with a bath of molten alkali cyanide, and distilled under cracking temperatures and pressures. Desulphurisation occurs with formation of thiocyanate. Any moisture reacts with the molten cyanide to form ammonia which in part decomposes and tends to hydrogenate the unsaturated hydrocarbons formed during the process. The evolved hydrocarbon vapours and gases are suitably collected. A portion of the cyanide destroyed during the reaction is regenerated by leading a current of ammonia into contact with the molten reagent during the distilling operation. The fusion residues are discharged from the still while still molten and treated to reconstitute the cyanide by adding sodium carbonate and subjecting the mixture to the action of a nitrogenous gas. The distillation process may be modified by running a molten mixture of commercial sodium cyanide and thiocyanate into the top of a column still filled with catalytic packing units comprising solid masses of iron, nickel, or copper, while introducing the preheated oil or vapours thereof at an intermediate part of the still.

H. S. GARLICK.

Treatment of hydrocarbons with liquid sulphur dioxide. ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 281,337, 28.11.27. Ger., 26.11.26).—In the treatment of hydrocarbons with liquid sulphur dioxide the latter is recovered from its mixtures with hydrocarbons by evaporation in stages, the low pressures necessary for complete evaporation in intermediate and final

stages being obtained by a jet apparatus operated by the sulphur dioxide vapour from the first-stage evaporators, which are heated to give the required increase of pressure. The reduction in pressure obtained by means of the jet is also employed to remove sulphur dioxide vapour from the cooler, the liquid sulphur dioxide in which is thus brought down to the temperature required for treatment of the oil. W. S. NORRIS.

Transformation of gaseous hydrocarbons. P. H. HULL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 304,914, 21.1.28).—The hydrocarbons are passed through a low-tension arc maintained between poles of tungsten, tungsten carbide, molybdenum, osmium, or tantalum. The electrodes preferably diverge in the direction of the stream of gas, and may consist of rods fastened by clips to a water-cooled tube. The process may be utilised, for example, in the production of acetylene from methane. A. B. MANNING.

Manufacture of lubricating oils. L. MELLERSH-JACKSON. From SUN OIL Co. (B.P. 305,846, 7.3.28).—The light fractions of crude mineral oil are distilled off under limited temperature and pressure in the absence of oxygen, the residue is treated with aqueous soda, the water and certain oil fractions are removed under reduced pressure, and the residue, distributed over an extended surface, is brought into heat exchange with mercury vapour, while limiting the temperature of the mercury vapour and the pressure on the oil to minimise cracking. The condensed lubricating oil distillate is then freed from malodorous volatile constituents by being distributed over an extended surface while being subjected to a high vacuum. W. S. NORRIS.

Manufacture of lubricating and insulating oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,553, 5.10.27).—Substances such as coal, tars, mineral oil, etc., if solid are extracted by means of a solvent or subjected to destructive hydrogenation, and if liquid are destructively hydrogenated or cracked, yielding liquid products; the latter, specifically those of the middle-oil type, are exposed in the coherent liquid phase to the action of high-tension electric currents, preferably of high frequency, yielding lubricating or insulating oils. Thus brown coal is hydrogenated at 450° and 200 atm., and, of the liquid products, a fraction of b.p. substantially up to 300° (viscosity 2.7° [Engler] at 50°) is subjected to a current of about 10,000 volts and 500 periods. After separating the viscous products by distilling off the more volatile constituents, the latter may be treated again. W. S. NORRIS.

Purification of petrol. W. M. KNOWLING and M. KOSTEVITCH (B.P. 287,141, 14.3.28. Fr., 16.3.27).—Tar, water, sulphur compounds, etc. are removed by treatment with a mixture of activated charcoal and activated silica gel, the latter being either of the wide-pore or the capillary-pore species, or a mixture of the two. A. B. MANNING.

Recovery of oils and naphthenic acids from still residues obtained in the purification of mineral oils. C. ARNOLD. From HUMBLE OIL & REFINING Co. (B.P. 304,926, 6.2.28).—The hot residues are discharged

on to the surface of gently flowing water, and, after stratification, the aqueous naphthenate solution and the oil layer are withdrawn separately, the oil layer being allowed to settle again in a second tank. L. A. COLES.

Mineral oil derivative and its manufacture. G. ALLEMAN, Assr. to SUN OIL Co. (U.S.P. 1,694,463, 11.12.28. Appl., 6.2.24).—Alkali precipitate (soda sludge) obtained in the refining of lubricating oil is treated with water to give, e.g., a total water content of 58%, and the mixture is heated to a steam pressure of at least 4 atm. and cooled under pressure. The mineral oil is separated and the soap and resin solutions are concentrated, the soap and resin being then filtered off from the sodium sulphate or other salts. R. BRIGHTMAN.

Apparatus for manufacture of carbon. A. LEDERER (U.S.P. 1,700,942, 5.2.29. Appl., 14.8.25. Austr., 24.4.25).—See B.P. 251,244; B., 1927, 244.

Coal washing apparatus. P. WOLF (B.P. 306,169, 14.11.27. Addn. to B.P. 206,151; B., 1925, 162).

Preparing charges containing [atomised] liquid fuel for use in internal-combustion engines. MARION STEAM SHOVEL Co., Assees. of S. P. and H. A. COWARDIN (B.P. 282,342, 2.8.27. U.S., 15.12.26).

Scavenging the residues of combustion deposited on the sliding surfaces of internal-combustion engines operated by pulverulent fuel. I. G. FARBENIND. A.-G. (B.P. 274,905, 25.7.27. Ger., 26.7.26).

Gas burners. C. H. KEMPTON and W. BISSELL (B.P. 305,786, 7.1.28).

Vacuum distillation (B.P. 303,078—9).—See I. Carbon dioxide in flue gases (U.S.P. 1,701,181).—See XI.

III.—ORGANIC INTERMEDIATES.

Analysis of acetic anhydride in the presence of strong acids by thermometric titration. T. SOMIYA (Proc. Imp. Acad. Tokyo, 1929, 5, 34—37; cf. B., 1927, 439).—The effect of strong acids in retarding the velocity of acetylation of aniline can be eliminated by addition of pyridine, and a sharp end-point obtained; under these conditions the acetylation velocities of aniline hydrochloride and aniline sulphate in acetylene tetrachloride are also sufficiently great to afford fairly satisfactory end-points. The amount of pyridine to be added is very important, and should be less than that of acetic acid in the solution to be titrated. Standardisation of solutions, and analysis of samples of aniline, aniline hydrochloride, and aniline sulphate are described in detail. C. W. SHOPPEE.

Manufacture of β -naphthol. A. I. ZAKHAROV (J. Chem. Ind. Moscow, 1928, 5, 720—721).—In presence of 1—2% of sodium carbonate the thermal bends of the fusion curve of sodium naphthalene-2-sulphonate and alkali are flattened. The fused mass does not thicken, and overheating and charring are absent. When the last portions of the sulphonate are added, the acid or even the neutral sulphonate tends to decompose on account of the decrease of total alkalinity; the presence of sodium carbonate preserves the salt from thermal

decomposition before it has been attacked by the sodium hydroxide. The fused mass contains 93–96% of β -naphthol. Excess of carbonate causes frothing.

CHEMICAL ABSTRACTS.

See also A., Mar., 265, **Equilibrium between methyl alcohol, carbon monoxide, and hydrogen** (SMITH and BRANTING). 273, **Chlorine carrier** (FIERZ-DAVID). 289, **Oxidation of methane with nitrogen peroxide** (FROLICH and others). **Intermediate products of combustion of methane** (RIESENFELD and GURIAN). 307, **Pyrogenic dissociation of condensed ring systems** (ORLOV). **Fluoranthene** (VON BRAUN and ANTON). 308, **Preparation of perylene** (MARSCHALK). 310, **Halogen derivatives of β -naphthol unsubstituted in position 1** (MARSCHALK). 316, **Use of zirconium tetrachloride in organic synthesis** (KRISHNAMURTI). 318, **Derivatives of 2-methylantraquinone and anthraflavene** (RUGGLI and MERZ). 319, **Naturally occurring anthraquinone derivatives** (MITTER and BISWAS). **Reduction products of hydroxyanthraquinones** (HARDACRE and PERKIN). 329, **Derivatives of quinoneacridone** (SCHARVIN and GALFERIN).

Naphthalene in gas. FULWEILER and others; POWELL; CUNDALL. **Power alcohol.** THAYSEN and GALLOWAY.—See II. **Polyhydric alcohol-polybasic acid reaction.** KIENLE and HOVEY.—See XIII. **Rectification of alcohol.** HOUGHLAND.—See XVIII. **Liquor cresoli saponatus.** FEIST.—See XX.

PATENTS.

Catalytic oxidation of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 291,419, 11.11.27. U.S., 3.6.27).—Oxidation catalysts give improved and more easily controlled results when mixed with "stabilisers," viz., sulphates, phosphates, halides, chlorates, nitrates, cyanides, arsenates, antimonates, bismuthates, borates, carbonates, or other innocuous salts of alkali metals, alkaline-earth metals, or other metals which form oxides not reducible by hydrogen. To these may be added "stabiliser promoters" which enhance their effect; there are usually mild catalysts, and include zeolites and other base-exchanging diluents, particularly those which are highly porous. Examples are: (1) ammoniacal silver vanadate on kieselguhr heated to expel water and ammonia, impregnated with aqueous potassium (or potassium hydrogen) sulphate, and dried at 80–100° to give a catalyst for formaldehyde from methyl alcohol, maleic acid from benzene, etc.; (2) potassium vanadomolybdate, ferric pyrovanadate, and potassium hydroxide on kieselguhr, dried at 400° and treated with burner gases at 400–500° to convert the alkali into sulphate, forming a catalyst for anthraquinone from anthracene etc.; (3) kieselguhr and silica gel, impregnated with potassium molybdate, sodium tungstate, lithium tantalate, and potassium silicate, calcined and sulphated with burner gas, to give a catalyst for chlorobenzaldehyde from chlorotoluene. Seventeen other examples are given. C. HOLLINS.

Oxidation of volatile organic compounds [acetaldehyde to acetic acid]. IMPERIAL CHEM. INDUSTRIES, LTD., S. W. ROWELL, and H. S. HIRST (B.P. 304,350, 18.10.27, 2.1. and 12.7.28).—A continuous

process for the air-oxidation of acetaldehyde in presence of a catalyst (e.g., manganese acetate and a trace of cobalt acetate) is described in which acetic acid containing the dissolved catalyst flows down a tower, meeting a countercurrent of air and acetaldehyde vapour. Acetic acid is withdrawn at the base of the tower and separated by freezing or distillation into pure acid (equal in amount to new acid produced) and catalyst acid, the latter being returned to the tower. The temperature is regulated by withdrawing liquor from a plate near the middle of the tower, cooling it, and returning it a little higher up the tower; the amount so circulated per hour may be, say, ten times the amount of acid fed per hour to the tower. C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANFY (B.P. 304,156, 15.7.27).—Charring is avoided by mixing the acid vapour with an inert gas (nitrogen, hydrogen, or steam) preheated to a temperature above that of anhydride formation; e.g., acetic acid vapour at 200–300° is mixed with steam at 800–1000°, the mixture being kept at 500–800°. C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. H. DREYFUS (B.P. 305,147, 30.7.27).—The (acetic) acid vapour is passed with hydrogen at 500–700° through a copper tube which has previously been heated to the required temperature in hydrogen. Nickel, cobalt, iron, or alloys of these or other metals, which in the form of acetates are reduced to the metallic state by hydrogen at the temperature of reaction, may be used in place of copper. C. HOLLINS.

Manufacture of esters. C. ARNOLD. From STANDARD OIL DEVELOPMENT Co. (B.P. 305,308, 21.11.27).—Esterification is performed in presence of a solvent in which the solubility of the ester is greater than that of its components or water; e.g., acetates of *sec*-butyl alcohol and *sec*-amyl alcohol are made in presence of a petroleum fraction of initial b.p. 300° and *d* 1.247.

C. HOLLINS.

Manufacture of isopropyl alcohol. G. O. CURME, JUN., and E. W. REID, Assrs. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,695,249, 11.12.28. Appl., 19.7.19). Propylene is absorbed in sulphuric acid or phosphoric acid at –10° to 0°, the temperature rising to 15–20° at the finish, and the product, containing, e.g., 80% of isopropyl sulphate, is hydrolysed by emulsification with water below 40°. R. BRIGHTMAN.

Manufacture of hydrocarbon derivatives and of unsaturated hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,603, 6.10.27).—The gases (up to pentane) from hydrogenation of coal are passed with steam or hydrogen sulphide over suitable catalysts at 700–750° to give olefines (propylene and butylene), acetic and other acids, alcohols, aldehydes, and in the case of hydrogen sulphide thiophen and other sulphur compounds. Catalysts used are manganese dioxide, alumina, molybdena, chromium oxide, and metal sulphides. C. HOLLINS.

Manufacture of organic bases. I. G. FARBENIND. A.-G. (B.P. 283,163, 5.1.28. Ger., 5.1.27).—For the production of pyridine bases from acetylene and ammonia

without formation of acetonitrile, a catalyst comprising an inorganic salt (zinc chloride, ferric chloride, cadmium chloride) capable of combining with ammonia to give complex salts is used. A typical product, with zinc chloride at 280–290°, gives fractions b.p. 60–75° (29%), 75–110° (6%), 110–125° (26%), 125–160° (24%), 200° (15%). C. HOLLINS.

Manufacture of hexahydroaniline [cyclohexylamine]. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 305,507, 5.11.27).—An 88% yield of cyclohexylamine is obtained in 30 min. by heating aniline at 250–300° in hydrogen at 100 atm. in presence of 2% of cobalt oxide, prepared, e.g., by heating cobalt oxalate at 200–400°. C. HOLLINS.

Manufacture of aromatic [hydr]oxaldehydes. I. G. FARBERIND. A. G. (B.P. 294,889, 30.7.28. Ger., 30.7.27).—Hydroxylated benzylamines are boiled with an isatin (especially a salt of isatin-5-sulphonic acid) in aqueous alkaline solution (cf. Traube, A., 1911, i, 960). The preparation of salicylaldehyde from *o*-hydroxybenzylamine, vanillin from vanillylamine, and 5-aldehyde-*o*-cresotic acid from 5- ω -aminomethyl-*o*-cresotic acid is described. C. HOLLINS.

Manufacture of substituted thioglycollic acids. I. G. FARBERIND. A.-G. (B.P. 287,858, 26.3.28. Ger., 24.3.27. Addn. to B.P. 281,290; B., 1928, 921).—The process of the prior patent is applied to the production of thioglycollic acids from halogenated benzene-sulphonic acids, e.g., 2-chloro-*p*-xylene-5-sulphonic acid, 3-chlorotoluene-6-sulphonic acid, 1:2:3-trichlorobenzene-4- or -5-sulphonic acid; in each case the sulphonic group is converted into a sulphonyl chloride group, which is reduced to thiol and condensed with chloroacetic acid. C. HOLLINS.

Manufacture of halogenated organic compounds. COMP. NAT. MAT. COL. & MANUF. DE PROD. CHIM. DU NORD RÉUNIES, ÉTABL. KUHLMANN (B.P. 294,462, 11.11.27. Fr., 23.7.27).—Aromatic amines are halogenated in the form of their arylsulphonyl derivatives. *o*-Toluenesulphon-*o*-toluidide, m.p. 134°, chlorinated with chlorate and hydrochloric acid, gives a product, m.p. 154°, from which on hydrolysis 5-chloro-*o*-toluidine is obtained. 2-Nitrotoluene-4-sulphon-*p*-toluidide, m.p. 131°, with chlorine in tetrachloroethane at 50°, gives the 3-chloro-*p*-toluidide, m.p. 152°; toluene-*p*-sulphonanilide yields the 2:4-dichloroanilide; the β -naphthylamide similarly affords a toluene-*p*-sulphon-1-chloro- β -naphthylamide, m.p. 105°; from these the bases are obtained by hydrolysis. C. HOLLINS.

Manufacture of 2:3-aminonaphthol and derivatives thereof. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 304,439, 15.12.27).—An *O*-arylsulphonate of 2:3-hydroxynaphthamide is treated in alcohol with alkaline hypochlorite or hypobromite and then hydrolysed with hot caustic alkali. 2:3-Hydroxynaphthamide *O*-*p*-toluenesulphonate, m.p. 216°, obtained from the amide and toluenesulphonyl chloride, gives 2:3-aminonaphthol *O*-*p*-toluenesulphonate, m.p. 144–145°, which may be hydrolysed to 2:3-aminonaphthol; the benzenesulphonate, m.p. 170°, behaves similarly. The *O*-*p*-toluenesulphonate, m.p. 184–185°, of 6-bromo-

2:3-hydroxynaphthamide (m.p. 295°) yields the *O*-*p*-toluenesulphonate, m.p. 105°, of 6-bromo-2:3-aminonaphthol (m.p. 222°). C. HOLLINS.

Manufacture of acenaphthene derivatives. G. T. MORGAN and H. A. HARRISON (B.P. 305,754, 8.12.27).—On nitration of acenaphthene with nitric acid in acetic anhydride at –5° to +16° 3-nitroacenaphthene, m.p. 151°, crystallises on cooling; the 5-nitro-compound may be obtained by diluting the mother-liquor. Reduction gives 3-aminoacenaphthene, m.p. 81° (cf. 1-amino-, m.p. 136°, 4-amino-, m.p. 87°, 5-amino-, m.p. 108°). The 3- and 5-isomerides may be separated by means of petroleum, b.p. 80–100°, from the reduced product of ordinary nitration. C. HOLLINS.

Manufacture of [hydr]oxydiaryl ketones. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 305,763, 16.12.27).—A hydroxyaromatic acid chloride is condensed with an aromatic hydrocarbon etc. in presence of aluminium chloride. 2:3-Hydroxynaphthoyl chloride with benzene gives phenyl 3-hydroxy- β -naphthyl ketone, m.p. 161–162°; with toluene, *p*-tolyl 3-hydroxy- β -naphthyl ketone, m.p. 152–153°; with anisole, *p*-anisyl 3-hydroxy- β -naphthyl ketone, m.p. 134–134.5°; from *m*-4-xylene-5-carboxylic chloride and anisole there is obtained 2-hydroxy-4'-methoxy-3:5-dimethylbenzophenone [*p*-anisyl 4-hydroxy-*m*-5-xylol ketone], m.p. 105–106°. C. HOLLINS.

Manufacture of naphthol ether carboxyamides alkoxy-naphthamides] and aminonaphthol ethers. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 304,441, 19.12.27).—Hydroxynaphthamides yield on alkylation *O*-ethers, which by Hofmann's reaction are converted into aminonaphthol ethers. 2:3-Methoxy-naphthamide m.p. 170°, and 2:3-aminonaphthol methyl ether, m.p. 107°; 2-carboxymethyl-3-naphthamide and 3-amino- β -naphthylglycollic acid; 1-ethoxy-2-naphthamide, m.p. 154°, and 2-amino-1-naphthol ethyl ether, m.p. 48–49°; 2:6-hydroxynaphthamide, m.p. 209° (benzyl ether, m.p. 198°; methyl ether, m.p. 224°), and the benzyl (m.p. 176°) and methyl (m.p. 156–157°) ethers of 2:6-aminonaphthol are described. C. HOLLINS.

Manufacture of salts of acid sulphuric esters of nitro-9:10-dihydroxyanthracenes. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 304,436, 9.12.27).—A nitroanthraquinone is treated with finely-divided copper or zinc in dry pyridine (or other tertiary base) in presence of chlorosulphonic acid or esters or of sulphur trioxide. The nitro-group is not reduced, the product being the water-soluble pyridine salt of the disulphuric ester of the nitroleucoanthraquinone. Products from 1- and 2-nitroanthraquinones, 1-nitro-2-methylantraquinone, and 1:5-dinitroanthraquinone are described. Treatment with acid oxidants (nitric or nitrous acid, chromic acid) regenerates the starting-material; with non-oxidising acids mixtures of nitro- and amino-anthraquinones are formed. C. HOLLINS.

Manufacture of new solid diazo-azo compounds. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 305,498, 28.10.27).—A *p*-aminoazo compound of the type, arylamine \rightarrow *o*-aminophenol ether, containing no sulphonic or carboxylic group, is diazotised and treated

with an alkali halide, or with sulphuric acid and/or a sulphate, or with a heavy-metal salt with or without sodium chloride, or with an arylsulphonate. The diazo compound separates or is isolated by evaporation. Anhydrous or partly anhydrous salts may be added to the solid diazo compound. Twenty examples are given including: *p*-nitroaniline \rightarrow cresidine, 2:5-dimethoxyaniline, 4-ethoxy-*m*-toluidine, *o*-aminophenyl benzyl ether; *o*-chloroaniline \rightarrow 2:5-dimethoxyaniline, 2-ethoxy- α -naphthylamine, cresidine, *o*-anisidine.

C. HOLLINS.

Production of 1-diazoanthraquinone-2-carboxylic acids. L. EIFFLAENDER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,700,790, 5.2.29. Appl., 23.11.26. Ger., 28.11.25).—See B.P. 262,119; B., 1928, 45.

Naphthenic acids from mineral oils (B.P. 304,926).—See II.

IV.—DYESTUFFS.

PATENTS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 279,489, 21.10.27. Ger., 23.10.26).—4:4'-Dimethylthioindigo, obtained by the usual methods from 3-chloro- or 3-nitro-*o*-toluidine by way of 3-chloro- or 3-nitro-*o*-tolunitrile, shows excellent fastness to light.

C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 280,866, 28.10.27. Ger., 18.11.26).—1-(3-Benzanthronylamino)anthraquinones carrying an *N*-alkyl group are converted into vat dyes by alkaline condensing agents; e.g., 1-(3-benzanthronylmethylamino)anthraquinone with alcoholic potassium hydroxide at 98° yields a blue-green vat dye.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAN. From I. G. FARBENIND. A.-G. (B.P. 305,082, 26.10.27).—1:1'- or 2:1'-Dianthraquinonylamines carrying at least one acridone ring (NH in 4- or 4'-position) and also an aroylamino-group are converted into vat dyes (carbazole derivatives?) by treatment with sulphuric acid alone at 30–40° or with sulphuric acid at 15–20° followed by oxidation, e.g., with nitrous acid. The dianthraquinonylamine from 4-aminoanthraquinone-1:2-acridone and 5-chloro-1-benzamidoanthraquinone affords in this way an olive dye; the isomeride from 4-chloro-1-benzamidoanthraquinone gives a greyish-green dye.

C. HOLLINS.

Preparation of yellow monoazo dyes fast to light. H. WAGNER and B. VOSSEN, Assrs. to GRASSELLI DYE-STUFF CORP. (U.S.P. 1,701,248, 5.2.29. Appl., 13.8.27. Ger., 19.8.26).—See B.P. 276,353; B., 1928, 183.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemical composition of wool, with special reference to the protein of wool fibre (keratin). H. R. MARSTON (Council for Sci. & Ind. Res., Australia, Bull. No. 38, 1928, 36 pp.).—Analyses of wools from various localities in Australia indicate that after careful purification the composition of the wool keratin from these sources is constant within the limits of experimental error. The cystine content of the fibre (determined by the Folin-Looney method after hydrolysis

with 20% sulphuric acid) was found to be 13.1%, whilst the tyrosine and tryptophan contents were 4.8 and 1.8%, respectively. For the determination of humin, amide, basic, and non-basic nitrogen a modification of Van Slyke's method was used. The preliminary acid hydrolysis introduces very serious errors unless rigidly controlled, since the *l*-cystine formed undergoes progressive racemisation, resulting, on treating the hydrolysate with phosphotungstic acid, in the formation of a racemised cystine phosphotungstate which is very soluble in comparison with the *l*-cystine compound. Further, in the determination of amide nitrogen by distilling the hydrolysate with lime *in vacuo* approximately one half of the cystine initially present is decomposed. The arginine content of wool keratin (corrected for ammonia derived from cystine) was found to be 10.2%, and the histidine and lysine contents (corrected for the abnormal yield of amino-nitrogen by cystine) to be 6.9 and 2.8%, respectively. Amide nitrogen amounted to 1.2%. The isoelectric point of wool keratin was found to be p_H 3.4. In the preparation of the wool for analysis, the samples were dehydrated in absolute alcohol, extracted for 48 hrs. with absolute ether, washed in 95% alcohol, then in 15 changes of tap water, and dried at 40° for 24 hrs. The wool was then combed, equilibrated in 0.01*N*-hydrochloric acid for 12 hrs., repeatedly washed in distilled water, and dried at 100°. Complete desiccation was then obtained by three successive treatments with absolute alcohol followed by drying for 96 hrs. at 100°. D. J. NORMAN.

Rigidity of wool and its change with adsorption of water vapour. J. B. SPEAKMAN (Trans. Faraday Soc., 1929, 25, 92–103).—The rigidity of dry Cotswold wool fibres has been studied as a function of the fibre diameter. The influence of adsorbed water on the rigidity of wool fibres has been determined under adsorption and desorption conditions, and expressions have been found relating the rigidity to the amount of water adsorbed and to the relative humidity. The reduction in relative rigidity with changing humidity can be expressed by a formula of the adsorption isotherm type and is probably caused by adsorption of water on particular groups in the wool molecule, but the simplicity of the rule is masked by the fact that water can be adsorbed in two other ways in addition.

O. J. WALKER.

Value of the ignition method for determining the cotton content of asbestos goods. H. SOMMER (Gummi-Ztg., 1929, 43, 1107–1108).—On account of the variation in the loss in weight on ignition suffered by various grades of asbestos or even by one grade at different temperatures, it is not possible to obtain trustworthy indications as to the proportion of any cotton present by an ignition method. Similarly, the hygroscopic moisture and the loss on treatment with 80% sulphuric acid fail to give accurate indications. The only satisfactory method is by means of cuprammonium solution. A shredded sample (0.5–1 g.) is weighed after exposure for several hours in air of 65% R.H., the moisture content of the material being determined in a separate sample by drying for 2–3 hrs. at 110°; any fat or starch should be removed previously. The sample is treated in a closed flask overnight, with

frequent shaking, with 50 c.c. of fresh cuprammonium solution (approx. 20 g. Cu per litre). After filtration through a Gooch crucible the residual asbestos is washed successively with cuprammonium solution and water and then dried at 110° until constant. The proportion of cotton is derived by difference. D. F. TWISS.

Cellulose ethers [alkylated celluloses]. E. BERL and H. SCHURP (*Cellulosechem.*, 1929, 10, 41—59).—A detailed account of the methylation and ethylation of various celluloses is given. Methylation of cellulose (linters) with methyl sulphate and 15% sodium hydroxide solution at the ordinary temperature (cf. Denham and Woodhouse, B., 1913, 974; 1914, 1084; 1917, 607, and Heuber and von Neuenstein, A., 1923, i, 17) affords first of all a monomethylcellulose, partly soluble in water, completely so in Schweizer's reagent, and insoluble in the usual organic solvents. Further methylation of this produces methylated celluloses with gradually increasing methoxyl content, until trimethylcellulose results (25—28 methylations). This derivative is insoluble in water and Schweizer's reagent. The solubility in water of the methylated celluloses diminishes gradually after the second methylation, finally becoming insoluble. In two of the cases examined the methoxyl content of the water-soluble portion and of the original material is the same. The solubility of the methylated products in Schweizer's reagent diminishes appreciably with increasing methoxyl content; two hydroxyl groups must be present in the cellulose molecule to give a completely soluble product. Methylation of alkali-soluble cellulose (by previous treatment with 75% sulphuric acid) with methyl sulphate under a variety of conditions gives derivatives containing 19.17—30.71% of methoxyl, according to the amount of methyl sulphate used. Methylation proceeds at the ordinary temperature, but analogous ethylation requires an initial temperature of 50° for reaction to commence. Monoethylcellulose is soluble in water and insoluble in alcohol, whilst diethylcellulose is soluble in alcohol and insoluble in water. The relative viscosity of aqueous solutions of the former increases considerably with increase of concentration, whilst an alcoholic solution of the latter also gives an increase, not nearly so marked as with the former. Ethylation of cellulose with ethyl chloride and sodium hydroxide (cf. G.P. 322,586 of 1912) gives products having varying compositions and properties, according to temperature of reaction, time of heating, presence of water, and amount of ethyl chloride used. Thus, at 100—120°, under otherwise identical conditions, the ethylated derivative is almost completely soluble in alcohol, whilst at 60—80° the product is only partially so. Further ethylation of alcohol-soluble and -insoluble products increases the ethoxyl content, does not affect the solubility in alcohol, and renders them soluble in chloroform. The viscosities of chloroform solutions of these substances vary considerably. Ethylation with 16 mols. of ethyl chloride at 115° gives a product (2.5 ethoxyl groups per mol.) partly soluble in chloroform, dichloroethylene, and benzene, insoluble in all other solvents. Further ethylation at 100° affords triethylcellulose, also obtained by direct ethylation of cellulose at 115° with 48 mols. of ethyl chloride. This derivative is soluble in chloroform, dichloroethylene,

benzene, and acetic acid. Further ethylation of a water-soluble ethylcellulose (ethoxyl content 18.2%) with ethyl chloride and sodium hydroxide at 80° increases the ethoxyl content to 38.85%; the new derivative is then insoluble in water, but soluble in alcohol. Dimethylcellulose can be ethylated with ethyl chloride and sodium hydroxide at 90—100°. It is not possible to ethylate cellulose with ethyl chloride in presence of pyridine or magnesium hydroxide. Hot sodium hydroxide solution does not hydrolyse dimethyl- or diethylcellulose.

H. BURTON.

Standard method for determining the viscosity of cellulose in cuprammonium hydroxide. COMMITTEE OF DIVISION OF CELLULOSE CHEMISTRY OF THE AMERICAN CHEMICAL SOCIETY (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 49—51).—Details of the required apparatus, of the experimental procedure, and of the preparation and standardisation of the cuprammonium solution for use in determining the viscosity of 2.5% solutions of cellulose are given. For ordinary solutions the falling-sphere method is used, the time being taken for glass beads of $\frac{1}{8}$ in. diam. to fall a vertical distance of 15 cm. through the solution maintained at 25°. When the viscosity is very low a special pipette is used of such dimensions that the shearing force applied in it is approximately the same as that due to the falling glass bead, thus making it possible to obtain the same apparent viscosity on a single solution by both methods. This pipette is calibrated by timing the outflow of an oil of known viscosity. The procedure gives satisfactory results with a wide variety of celluloses, and if satisfactory measurements cannot be made with cellulose solutions of the above concentration other concentrations may be used and the viscosity corresponding with a 2.5% solution calculated by the application of a suitable formula such as Joyner's.

B. P. RIDGE.

Determination of α -cellulose. SUB-COMMITTEE 2 OF DIVISION OF CELLULOSE CHEMISTRY OF THE AMERICAN CHEMICAL SOCIETY (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 52—54).—The α -cellulose content of two grades of cotton linters and three grades of sulphite pulp has been determined by four variations of the same method and by workers in eight different laboratories. The following results of the study of different factors affecting the determinations are given. (1) Dilution of the sodium hydroxide-cellulose solution with an equal volume of water and stirring immediately before filtering does not precipitate β -cellulose and facilitates filtration. (2) The yield of α -cellulose is not affected by washing with water at temperatures above 20°. (3) Cellulose is more soluble in sodium hydroxide solution alone than in a similar solution containing dissolved cellulose. (4) Mercerisation in air and in nitrogen give the same yields of α -cellulose. In the method which is recommended to the Cellulose Division of the American Chemical Society as a tentative standard method the cellulose is mercerised with 17.5% sodium hydroxide for 45 min., the alkali being added in several portions, an equal volume of water is added, the α -cellulose filtered off through a Gooch crucible, washed with water, 10% acetic acid, and water, and dried to constant weight at 105°. Close agreement is shown between the results from the different laboratories.

B. P. RIDGE.

Determination of α - and β -cellulose. R. BERGQVIST (Papier-Fabr., 1929, 27, 119—123).—The influence of various factors in the determination of α - and β -cellulose, such as the conditions of filtering, the ratio of sodium hydroxide solution to cellulose material, the amount of water used for dilution after the mercerisation, etc., has been investigated, and the method of carrying out each stage of the determination is discussed. The following points are emphasised: (1) The temperature of the experiments should be controlled by use of a thermostat at 20°, or a correction should be applied depending on the temperature employed. The wash-water used should also be at 20°. (2) After addition of the diluting water, filtration and washing must be done as quickly as possible; a suitable filtering medium is a piece of coarse linen fabric placed in a Buchner funnel. (3) For dilution of the sodium hydroxide solution after mercerisation of the cellulose the same quantity of water (300 c.c.) should always be used. (4) For the kneading operation 5 min. should suffice, and, if care be taken in working, reproducible results are easily obtained within the limits for α -cellulose of $\pm 0.1\%$. B. P. RIDGE.

Determination of cellulose and amount of chlorine consumed in its isolation. M. W. BRAY (Ind. Eng. Chem. [Anal.], 1929, 1, 40—43).—The solvent-extracted sawdust or wood pulp is chlorinated in a Jena glass crucible with a fritted glass bottom by passing chlorine under slight pressure from a gas burette up through the crucible and over into a Hempel gas pipette. The volumes of gas passed over and that remaining in the apparatus are measured, the contents of the crucible are washed with water (titration of the washings giving the amount of hydrochloric acid formed), treated with sodium sulphite solution, and again washed. These treatments are repeated until the lignin is completely removed and the fibres are uniformly white, when the weight of dry material remaining is determined. The time taken for the first chlorination is 3—4 min., and for each subsequent chlorination 1—2 min., but the removal of lignin and other foreign substances is as complete as when, by the older processes, $\frac{1}{2}$ —1 hr. is taken. The method has the advantages that the chlorination is more uniform and complete than when it is carried out by suction or in beakers, the time is shortened without impairing efficiency, the total chlorine used together with that forming hydrochloric acid may be measured at the various stages of the process, and there is less degradation of the cellulose as shown by the α -cellulose content. B. P. RIDGE.

Recovery of the soda lyes from the industrial delignification of Tripoli esparto. C. PADOVANI and E. SALMOIRAGHI (Annali Chim. Appl., 1929, 19, 23—45).—In the autoclave treatment of these lyes the best clarification is obtained at 350°, the pressure exceeding 200 atm. Under this treatment the lyes undergo a species of "cracking," about 80% of the original organic matter being converted into tar of high calorific value. No methyl alcohol is formed, but acetic acid is obtained in amount approximating to that originally present. In general, treatment of the lyes subsequent to chlorination is uneconomical unless they are subjected to concentration. A systematic study was

made of the influence on the results of the maximum temperature reached and of the velocity of heating.

T. H. POPE.

Can the mechanical wood-pulp content of paper be determined to a fraction of a per cent? KORN (Papier-Fabr., 1929, 27, 142—143).—The mechanical wood pulp present may be determined either microscopically or chemically. The former method needs considerable dexterity and experience, and results accurate only to $\pm 5\%$ are obtained under the best conditions. In chemical methods the complete separation of the wood pulp from the rest of the paper is very difficult, so that indirect methods with their inherent drawbacks are used. Halse's method depends on the determination of the lignin content of the paper and calculation from this of the wood-pulp content by means of a formula. The latter depends on an average value for the lignin content of sulphite-cellulose of 3%, but for the same paper the amounts of lignin found may vary within wide limits. A further complication is introduced by variations in the moisture content of the material examined. It is concluded that the determination of the wood-pulp content to within 1% is impossible. B. P. RIDGE.

Rosin sizing [of paper]. S. R. H. EDGE (Proc. Tech. Sect. Papermakers' Assoc., 1928, 9, 56—68).—See B., 1928, 564.

See also A., Mar., 245, Röntgen diagrams of cellulose (HERZOG and JANCKE). Structure of cellulose (MARK and MEYER). 299, Allylcellulose (SAKURADA). Action of fatty acids on cellulose (MALM and CLARKE). 300, Cellulose xanthamides (NAKASHIMA). 342, Relation between cystine yield and total sulphur in wool (RIMINGTON).

PATENTS.

Process for hydrophilising flax in the raw or retted state to obtain hydrophilous wadding therefrom. P. HADDAN. From SOC. DES TEXTILES ROANNAIS (B.P. 305,709, 10.11.27).—The material is boiled for 1—2 hrs. with an alkaline solution containing, e.g., 2 pts. of caustic soda, 2 pts. of sodium carbonate, 1 pt. of Castille soap, and 0.2 pt. of milk of lime per 100 pts. of water, and, after rinsing, is soaked in 0.2% sodium hyposulphite solution at not above 80° for 1 hr. After washing and drying, the fibres are treated first with 0.5% sulphuric acid, then with a bleaching agent, preferably "chlorozone," and finally for about 1 hr., with agitation, with a solution containing, e.g., water 100 pts., sulphuric acid (d 1.835) 2 pts., sodium hyposulphite 0.2 pt., and nitric acid 0.1 pt. Between each of these treatments the fibre must be thoroughly washed. D. J. NORMAN.

Manufacture of [moiré patterned] fabrics or textile materials. BRIT. CELANESE, LTD. (B.P. 280,195, 27.10.27. U.S., 4.11.26).—Moiré effects which are permanent may be produced on fabrics containing cellulose esters or ethers by application of heat and pressure according to known processes. [Stat. ref.]

F. R. ENNOS.

Manufacture or treatment [cutting] of textile products. BRIT. CELANESE, LTD., and W. A. DICKIE (B.P. 305,257, 2.11.27).—The operation of cutting

fabrics containing cellulose esters or ethers by hot knives or dies in order to seal them against fraying is facilitated if, before cutting, the fabric is sprayed with high-boiling softeners or solvents, *e.g.*, diacetone alcohol.

D. J. NORMAN.

Manufacture of acetylcellulose. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 305,601, 5.9.27).—Before acetylation, the cellulose is treated continuously, *e.g.*, by percolation, with formic acid of at least 80% concentration at ordinary or raised temperatures in the absence of esterifying agents, until it contains 4–10% by wt. of chemically combined formic acid.

F. R. ENNOS.

Precipitation of viscose solutions. I. G. FARBERIND. A.-G. (B.P. 278,716, 5.10.27. Ger., 5.10.26).—The precipitating bath used has a high content of sulphuric acid and sodium salts, the concentrations of which are so selected that on cooling to the crystallising point "trisodium hydrosulphate," $\text{Na}_3\text{H}(\text{SO}_4)_2 \cdot \text{H}_2\text{O}$, forms the first deposit.

F. R. ENNOS.

Manufacture of artificial threads, filaments, ribbon, etc. COURTAULDS, LTD., E. HAZELEY, and E. A. MORTON (B.P. 305,279, 7.11.27).—In order to prevent the threads from wrapping round the spraying device when centrifugally-spun artificial silk is washed in a rotating spinning box by means of a non-rotating spray, two or more rods, or a cylinder of wire gauze, interposed between the cake of silk and the spraying device, are so arranged that they rotate with the box.

D. J. NORMAN.

Treatment of sheets of cellulose pulp for use particularly in the manufacture of viscose, and apparatus therefor. L. C. P. JARDIN (B.P. 286,619, 5.3.28. Fr., 4.3.27).—In order to avoid the use of hydraulic presses in the preparation of alkali-cellulose, the pulp sheets are uniformly impregnated with just that quantity of caustic soda solution necessary for the formation of alkali-cellulose.

D. J. NORMAN.

Manufacture of cellulose esters and conversion products therefrom. C. EBERT and T. BECKER, Assrs. to I. G. FARBERIND. A.-G. (U.S.P. 1,701,229, 5.2.29. Appl., 17.2.27. Ger., 22.2.26).—See B.P. 289,973; B., 1928, 920.

Preparation and spinning of fibrous materials for production of yarns. M. CONLAN (B.P. 305,797, 24.1.28).

Apparatus for spinning artificial threads. RUTH-ALDO CO., INC., Assecs. of (MISS) M. KELIN (B.P. 293,424, 4.7.28. Fr., 6.7.27).

Production of twisted artificial silk threads etc. O. SINDL (B.P. 305,280, 28.11.27).

Devices for stretching artificial threads made by the dry-spinning method. RUTH-ALDO CO., INC., Assecs. of R. A. J. THENOZ (B.P. 283,139, 4.1.28. Fr., 4.1.27).

Apparatus for manufacture of films from cellulose solution. FELDMÜHLE PAPIER- U. ZELLSTOFFWERKE A.-G. (B.P. 290,233, 10.5.28. Ger., 10.5.27).

Drying apparatus for cellulose sheets etc. H. HAAS (B.P. 306,359, 17.7.28. Ger., 22.2.28).

Manufacture of plastic material [from fibres and asphalt etc.]. E. THOMAS (B.P. 305,973, 11.11.27).

Drying of paper (B.P. 279,816). **Friction material** (B.P. 284,269).—See I. Nitrocellulose solutions (B.P. 305,148).—See XIII. **Bonding of india-rubber and cellulose derivatives** (B.P. 305,745).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching of *Phormium tenax*. P. W. AITKEN (New Zealand J. Sci. Tech., 1928, 10, 236–240).—Methods of bleaching the material by means of hypochlorite and permanganate solutions are compared with the usual wiring and grassing method from the point of view of cost, ease of manipulation, permanency of the colour obtained, and conservation of the strength of the product. When adequate allowance is made for cost of the bleaching agents, labour, and other charges, considerable economy is still effected by use of the above solutions in place of the grassing. Bleaching may be carried out in the vat, or by the chain method as a continuous process, and if in the case of the permanganate solutions the temperature is raised above 54° only a few minutes' treatment is required and labour charges for a continuous process are reduced to a minimum. No loss of strength occurs as a result of these treatments beyond that occurring during grassing. The colour of the material is different from that obtained by weathering, but is reasonably permanent, and is considered superior. Use of sulphur dioxide as a bleaching agent is not a success.

B. P. RIDGE.

Colour of light sources. I. Carbon arcs. II. Daylight lamps. P. W. CUNLIFFE and H. LANIGAN. III. Sunlight and skylight. P. W. CUNLIFFE (J. Text. Inst., 1929, 20, 21–46 T).—Preliminary work to the study of the fastness to light of dyes on textile fabrics.

B. P. RIDGE.

Influence of titanium white on coal-tar dyes. KEIDEL; WAGNER.—See XIII.

PATENTS.

Bleaching of paper pulp. J. RUTHS, Assr. to RUTHS ACCUMULATOR AKTIEBOLAG (U.S.P. 1,700,647, 29.1.29. Appl., 20.10.21. Swed., 9.1.20).—Steam required in the short-time bleaching of paper pulp is supplied from a steam accumulator. D. J. NORMAN.

Production of dyeings and printings by means of vat dyes. DURAND & HUGUENIN SOC. ANON. (B.P. 281,336, 28.11.27. Ger., 29.11.26. Addn. to B.P. 220,964; B., 1925, 879).—In the process of the prior patent the acid required for accelerating oxidation of the ester salt of a leuco-vat dye is derived from a substance such as mono- or di-chlorohydrin, chloroethyl alcohol, or potassium methyl (or ethyl) sulphate which is included in the printing or dyeing composition.

A. J. HALL.

Transference of dyestuffs to fabrics. H. S. SADTLER (B.P. 284,676, 3.2.28. U.S., 3.2.27).—Coloured designs on textile fabrics are obtained by use of transfer paper consisting of grease-proof paper printed with inks prepared with water-soluble dyes and glycerin. The transfer paper is pressed upon the wet fabric after dusting

with a carbohydrate such as starch, and then is covered with a damp cloth and ironed; increased fastness of the colours on the fabric is obtained. A. J. HALL.

Production of reserves on wool and silk. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 305,269, 4.11.27).—Wool and silk are reserved against acid or neutral dyes by adding sulphonated natural resin (colophony) to the dye-bath. C. HOLLINS.

Coloration of materials made with or containing cellulose acetate. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 305,560 and 305,566, 7.11.27, Addn. [A] to B.P. 239,470 [B., 1925, 879], and [B] to B.P. 237,943 [B., 1925, 801]).—(A) The following diphenylamines, selected from the general claim of the prior patent, are used for dyeing acetate silk: 4-chloro-2-nitro-3'-methyl-, 4-chloro-2-nitro-4'-ethoxy-, 4-chloro-2-nitro-4'-acetamido-, 4-bromo-2-nitro-, 4-bromo-2-nitro-4'-methoxy-, 2-nitro-, 2-nitro-4-methyl-, 2-nitro-4'-ethoxy-4-methyl-, 4-chloro-2'-nitro-, 4-bromo-2'-nitro-, 2-nitro-4'-acetamido-diphenylamines. (B) The same compounds are used in the form of dispersions with the agents recommended in B.P. 219,349 and 224,925 (B., 1924, 906; 1925, 39). C. HOLLINS.

Revival of the colour of flowers or other patterns on carpets, rugs, mats, etc. M. E. DEANE (B.P. 304,172, 8.9.27).—The faded parts of coloured materials are painted with a suitable dye dissolved in methylated spirit. A. J. HALL.

Manufacture of nitrosoamine printing colours. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 305,787, 11.1.28. Addn. to B.P. 303,942; B., 1929, 239).—A nitrosoamine and a coupling component are dissolved separately in water, filtered from impurities, mixed, and evaporated to give a dyeing preparation of the Rapid Fast type. Premature colour-formation is prevented if the solutions are sufficiently concentrated before mixing, and a considerable amount of alkali is present. *E.g.*, Naphthol AS (100 pts.) and concentrated aqueous sodium hydroxide (110 pts.) in hot water (300 pts.), and *o*-nitroaniline (52.6 pts.) in the form of nitrosoamine salt and sodium hydroxide (8 pts.) in hot water (200 pts.), are filtered separately, mixed, and evaporated in a vacuum. The solution of the coupling component may be evaporated separately to dryness before adding the nitrosoamine solution. C. HOLLINS.

Mercerisation of vegetable materials with alkaline liquids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,509, 6.9.27).—Wetting-out agents suitable for use with highly concentrated alkaline liquors and prepared by adding a sulphonated aromatic compound to sulphonated oleic or ricinoleic acid are added to the mercerising lyes. Examples are dibutyl-naphthalenesulphonic acid with sulphonated dihydroxystearic acid, or the sodium salt of tetrahydronaphthalene-sulphonic acid with sulphonated oleic acid. A. J. HALL.

Treatment [delustring] of [viscose] artificial silk. C. and T. TERRELL (B.P. 305,828, 17.2., 14.3., and 13.6.28).—The material is impregnated with an aqueous solution of a salt, *e.g.*, barium chloride, sodium silicate, or a soluble calcium salt, dried, and treated with an aqueous solution of another salt, *e.g.*, ammonium sul-

phate, aluminium nitrate, or ammonium carbonate, which reacts with the first to form an insoluble, colourless, opaque precipitate in and/or on the filaments, which are finally softened by washing in a soap solution. F. R. ENNOS.

Treatment [fireproofing] of fabrics or other materials or articles made from combustible material. BRIT. CELANESE, LTD. (B.P. 296,344, 10.8.28. U.S., 29.8.27).—Textile materials, *e.g.*, cellulose esters or ethers, are steeped in an aqueous solution at 15–16° containing arsenic, stibinic, or phosphoric acid, rinsed, and dried. Cellulose acetate silk after steeping for 1 hr. at 40° in a 10% solution of phosphoric acid melts, but does not ignite when held in a flame. Aluminium acetate may also be added to the steeping liquor. A. J. HALL.

Dyeing and like machines. C. S. BEDFORD (B.P. 305,901, 27.6.28. Addn. to B.P. 290,138).

[Winding] process for retting, boiling, bleaching, or otherwise treating yarn or rove with liquids. M. WADDELL and H. C. WATSON (B.P. 306,206, 25.11.27).

[Apparatus for] drying or drying and bleaching fabrics. M. M. KASANOF (B.P. 301,783, 5.9.27).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Manufacture of synthetic nitric acid from nitrogen oxides under pressure. V. I. MALYAREVSKI and V. V. PAKOV (J. Chem. Ind. Moscow, 1928, 5, 682–689).—The absorption of nitrogen oxides in water under pressure is advocated; 65–67% nitric acid could thus be obtained. Water-cooling is necessary. A projected plant is described. CHEMICAL ABSTRACTS.

Determination of small amounts of silica in orthophosphoric acid. P. ALBRECHT (Chem.-Ztg., 1929, 53, 118–119).—Phosphoric acid is mixed with 5 times its weight of silver nitrate and heated in a platinum dish at 100–120° for 2 hrs., when the silico-phosphoric acids are irreversibly decomposed with formation of silver phosphate and insoluble silicic acid. Soluble salts are dissolved in warm dilute nitric acid, and the solution is filtered. The insoluble matter is ignited and weighed before and after treatment with hydrofluoric acid. J. S. CARTER.

New process for the manufacture of caustic soda and sodium carbonate. W. SIEGEL (Chem.-Ztg., 1929, 53, 145–147).—The process of the "Ring" Ges. chem. Unternehmungen for the manufacture of caustic soda from common salt consists of three steps: (1) $\text{CaF}_2 + \text{SiF}_4 + 2\text{NaCl} = \text{Na}_2\text{SiF}_6 + \text{CaCl}_2$; (2) $\text{Na}_2\text{SiF}_6 = 2\text{NaF} + \text{SiF}_4$; (3) $2\text{NaF} + \text{Ca(OH)}_2 = 2\text{NaOH} + \text{CaF}_2$. The silicon fluoride from step (2) and the calcium fluoride from step (3) are used in step (1). If sodium carbonate is required the milk of lime in step (3) is replaced by calcium carbonate. Step (3) presents no difficulties, and the caustic soda can be produced at a higher concentration than from the carbonate. Step (1) is carried on in solution acidified with hydrochloric acid, and may be written as follows: (a) $3\text{SiF}_4 + 4\text{H}_2\text{O} = 2\text{H}_2\text{SiF}_6 + \text{Si(OH)}_4$; (b) $\text{H}_2\text{SiF}_6 + 2\text{NaCl} = \text{Na}_2\text{SiF}_6 + 2\text{HCl}$; (c) $3\text{CaF}_2 + \text{Si(OH)}_4 + 2\text{NaCl} + 4\text{HCl} = \text{Na}_2\text{SiF}_6 + 3\text{CaCl}_2 + 4\text{H}_2\text{O}$. No acid

is therefore consumed, apart from small losses. The reaction is carried out in a conical vessel, into the lower part of which silicon fluoride is led. It is important to obtain a good suspension of the calcium fluoride. Step (2) presents the chief difficulties. The dissociation pressure of sodium silicofluoride reaches a workable figure at about 700°, but as decomposition proceeds the pressure falls off owing to the formation of a solid solution of m.p. 717°. The final driving-off of the last traces of silicon fluoride is a slow process, and any which may remain results in the caustic soda becoming contaminated with sodium silicate. It might be possible to work below 700° with injection of indifferent gas or superheated steam, but this would lead to difficulty in recovering the silicon fluoride. The theoretical heat balance shows a consumption of only 260 kg.-cal. per kg. of NaOH. A modification of step (3) employs calcium cyanamide, whereby sodium carbonate and ammonia are obtained; a further variation involves the reaction $2\text{CaF}_2 + \text{SiO}_2 + 6\text{C} = \text{SiF}_4 + 2\text{CaC}_2 + 2\text{CO}$, carried out in an electric furnace. By incorporating this latter in the cycle, hydrochloric acid is obtained as a by-product. The advantages of this complication are doubtful. The process here described has not been proved on a works' scale.

C. IRWIN.

Manufacture of oxygen from lime and chlorine. O. R. SWEENEY and A. W. RALSTON (Proc. Iowa Acad. Sci., 1927, 34, 215).—For the reaction of chlorine with a suspension of lime the optimal temperature is 94° and the optimal concentration of the nickel nitrate catalyst is 0.02 g. per 100 c.c. The nickel or cobalt catalyst is not easily poisoned.

CHEMICAL ABSTRACTS.

Determination of ozone in air. M. S. EGOROV (Z. Unters. Lebensm., 1928, 56, 355—364).—The iodometric methods of Wartenberg and Podjaski (A., 1925, ii, 1198) and of Macdonnell (B., 1926, 273) for determining traces of ozone are not sufficiently sensitive, and the fluorescein method of Benoist (B., 1919, 285 A) is unsuitable owing to the slowness of the reaction. The following method was found to be suitable. Fluorescein (1—2 mg.) is dissolved in several drops of 10% sodium hydroxide solution and 10 c.c. of saturated sodium hydroxide are added. The mixture is shaken for several minutes with zinc dust, filtered through asbestos, and used fresh or stored in sealed tubes. 10 c.c. of 0.5% sodium hydroxide solution are placed in a test-tube with one drop of fluorescein solution prepared as described, and air is drawn through the liquid until the intensity of its fluorescence is equal to that of a standard fluorescein solution (standardised iodometrically). Not more than 8 litres of air should be drawn through at a rate of 12—15 litres/hr. The standard fluorescein solution should not be more dilute than $1:10^8$, and the standardisation should be carried out at a dilution of $1:10^6$. Nitrogen oxides and hydrogen peroxide do not affect the reagent.

W. J. BOYD.

Automatic apparatus for determination of small concentrations of sulphur dioxide in air. II. M. D. THOMAS and J. N. ABERSOLD (Ind. Eng. Chem. [Anal.], 1929, 1, 14—15; cf. Thomas and Cross, A., 1928, 862).—The automatic recording of the change in iodine content of a solution is not easy, but the substi-

tution for it of distilled water, slightly acidified and containing 0.003—0.006% of hydrogen peroxide, makes it possible to determine the sulphuric acid produced by conductivity. The method is suitable for concentrations up to 60 p.p.m. of sulphur dioxide in air, and results agree well with those given by other methods.

C. IRWIN.

Extraction of bromine by solvent. II. B. G. PANTELEIMONOV (J. Chem. Ind. Moscow, 1928, 5, 484—489).—When kerosene is used for the extraction of bromine from aqueous solutions, solvent containing bromine is lost as an emulsion, the loss of bromine being 14—26% after 15 extractions. Absorption in sponge reduces the loss of kerosene from 1—2% to 0.5% per extraction. The bromine is thereby converted into hydrogen bromide, and the kerosene can thus be used again without treatment with alkali, the hydrogen bromide remaining in the aqueous liquid.

CHEMICAL ABSTRACTS.

See also A., Mar., 256, **Adsorption of gases and vapours by silica gels** (BOSSHARD and JAAG). 257, **Siloxen as adsorbent** (KAUTSKY and BLINOFF). **Adsorption of carbon dioxide by ferric hydroxide** (TILLMANS and others). 267, **Equilibrium between water and the nitrates and sulphates of sodium [and potassium] at 50—90°** (CORNAC and KROMBACH). 274, **Copper catalysts from precipitated hydroxide** (FROLICH and others). 275, **Electrolytic preparation of sodium perborate** (GIORDANI and INTONTI). 284, **Detection of thiocyanate, fluoride, and copper, and drop reaction for ammonia** (FEIGL and others).

Soda lyes from treatment of esparto. PADOVANI and SALMOIRAGHI.—See V. **Evaluation of stibnite.** McNABB and WAGNER.—See X. **Determination of iodine and potassium iodide.** WINTERFELD and SIECKE.—See XX. **Toxicity of hydrogen cyanide.** ALLISON.—See XXIII.

PATENTS.

Manufacture of hydrogen cyanide from cyanides of the alkali metals. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 305,860, 23.3.28).—Alkali cyanides are treated in the presence of water with a quantity of sodium bicarbonate at least 10% in excess of that required by the equation, $\text{NaCN} + \text{NaHCO}_3 = \text{Na}_2\text{CO}_3 + \text{HCN}$, and the hydrogen cyanide is removed below 50° *in vacuo*. L. A. COLES.

Synthesis of ammonia. G. F. UHDE (B.P. 272,930, 28.12.27).—Catalysts active at comparatively low temperatures (380—450°) are prepared from material containing complex cyanides by heating it in the contact chamber by the admission of an externally heated mixture of hydrogen and nitrogen from which all traces of oxygen and compounds containing it have been removed. The proportion of hydrogen in the mixture, which may be initially as low as 10%, and the temperature and pressure of the gases are raised during the preparation of the catalyst until normal working conditions are attained.

L. A. COLES.

Means for obtaining crystals of uniform coarse grain, especially of fertiliser salts. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 304,872, 24.11.27).—

A hot, saturated solution of, *e.g.*, ammonium sulphate is passed from a saturator through a crystallising tower in which the liquid is kept moving upwards by means of bubbles of air admitted at suitable intervals and by a spiral propeller. Coarse crystals fall to the bottom and are removed, whilst fine crystals pass out at the top of the tower and are returned to the saturator. In the production of ammonium bicarbonate etc., ammonia solution is charged in from the saturator, and carbon dioxide and ammonia gas are forced into the tower.

L. A. COLES.

Manufacture and purification of alumina. J. C. SEAILLES (B.P. 293,392, 3.7.28. Fr., 5.7.27).—Modifications for reducing the proportion of silica in the product obtained as described in B.P. 277,697 and 283,509 (B., 1928, 603, 230) comprise reduction of the temperature and/or time of treatment of the bauxite etc. with lime in the autoclave; addition of catalysts, *e.g.*, calcium benzenesulphonate, calcium sulphuricinate, etc., during digestion; prevention of an excess of free alkali during treatment of the alkaline-earth aluminate with sodium carbonate, *e.g.*, by previous extraction of free lime with water, by direct carbonation, or by the addition of sodium bicarbonate; and treatment of the sodium aluminate solution with material containing calcium aluminate, *e.g.*, with material freshly discharged from the autoclave.

L. A. COLES.

Production of green hydrated chromium oxide. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 304,809, 24.10.27).—Chromic acid or a chromate is heated with water and a reducing agent, *e.g.*, carbon monoxide, hydrogen, or mixtures of these (sulphur dioxide being excepted), under a raised pressure not exceeding 150 atm.

L. A. COLES.

Manufacture of pure hydrated chromic chloride. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 306,305, 23.3.28. Addn. to B.P. 271,016; B., 1927, 522).—Anhydrous chromic chloride is treated with a quantity of water equal to or not greatly exceeding that required to form the crystals, $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$, in the absence of air and in the presence of catalytic reducing agents, preferably chromous chloride.

L. A. COLES.

Impregnated gel for adsorbing water vapour. SILICA GEL CORP., Assees. of E. B. MILLER and G. C. CONNOLLY (B.P. 280,934, 17.11.27. U.S., 19.11.26).—The material comprises silica gel, with pores of such a size that it will adsorb not less than 10% of its weight of water vapour at 30° and 22 mm. pressure, impregnated with a substance which will combine reversibly with water vapour, *e.g.*, copper sulphate.

L. A. COLES.

Manufacture of carbonic snow [solid carbon dioxide]. E. Du Bois (B.P. 304,958, 3.4.28).—Liquid carbon dioxide is passed through a spiral coil terminating in a jet with an enlarged mouthpiece, situated at the top of a suitable receptacle in which the solid collects and whence it can be removed through an opening at the bottom. Carbon dioxide gas liberated by evaporation passes through a screen of fine mesh placed above the spiral coil, and, after passage through a double cavity in the walls of the receptacle, is returned to the compressor.

L. A. COLES.

Catalytic oxidation of sulphur dioxide. A. O. JAEGER, Assr. to SELDEN Co., and J. A. BERTSCH (U.S.P. 1,695,285, 18.12.28. Appl., 6.2.26).—Sulphur dioxide gases containing hydrogen chloride or catalyst poisons other than arsenic are oxidised by zeolite catalysts free from platinum and containing vanadium or other catalytically active elements.

R. BRIGHTMAN.

Recovery of ammonia from waste waters in manufacture of artificial filaments by the cuprammonium process. Recovery of ammonia from dilute solution. H. HOFMANN, Assr. to AMER. BEMBERG CORP. (U.S.P. 1,701,110 and 1,701,265, 5.2.29. Appl., [A] 21.4.25, [B] 29.1.27. Renewed [A] 13.6.28. Ger., [A, B] 6.5.24).—See B.P. 233,669; B., 1925, 955.

Manufacture of ammonium nitrate in water solution and simultaneous concentration thereof. C. TONIOLO (U.S.P. 1,700,914, 5.2.29. Appl., 27.1.26. It., 7.2.25).—See B.P. 247,227; B., 1927, 408.

Production of aluminium oxide. S. E. SIEURIN (U.S.P. 1,701,510, 12.2.29. Appl., 4.6.24. Swed., 15.6.23).—See B.P. 217,568; B., 1925, 545.

Cyanides (B.P. 304,421).—See II. **Catalytic oxidation (B.P. 291,419).**—See III. **Chromium compounds (B.P. 305,712).** **Vessels with acid-proof linings (B.P. 290,189).**—See X. **Fertilisers (B.P. 305,760).**—See XVI.

VIII.—GLASS; CERAMICS.

White stoneware and other white ceramic masses. F. SINGER (Chem.-Ztg., 1929, 53, 105—106, 126—129).—The methods of manufacturing chemical stoneware and porcelain are briefly described, and a short account of their properties and microstructure is given. The mechanical properties of stoneware or earthenware are dependent to a considerable extent on the form and distribution of the mullite produced during firing.

A. R. POWELL.

Slagging of refractory materials. II. Influence of the chemical composition of the slags. H. SALMANG and F. SCHICK (Arch. Eisenhüttenw., 1928—9, 2, 439—447; Stahl u. Eisen, 1929, 49, 187—189).—The corrosive action of various slags on a high-grade fireclay crucible at 1410° has been examined. Slags with a high content of ferrous oxide are very fluid, but have a low wetting power and high surface tension, whereas those with a high content of manganous oxide are more viscous and have a low surface tension and high wetting power; both types are highly corrosive. The slagging action of various metal oxides on fireclay decreases in the order: lead, ferrous, manganous, calcium, ferric, potassium, sodium, manganic oxide, but in molten slags the order of decreasing activity of the basic oxides is lime, ferrous oxide, manganous oxide, ferric oxide, magnesia. In highly basic slags from iron smelting, alumina acts as an acidic constituent and tends to reduce the corrosive action of the bases. Single-silicate slags containing less of the base than corresponds with $\text{RO} \cdot \text{SiO}_2$ have no corrosive action on fireclay, but with magnesium aluminosilicate slags the addition of lime causes a progressive increase in the corrosive action, as also does addition of ferrous or manganous oxide to

calcium aluminosilicate slags. The slagging power for fireclay at 1410° in 1 hr. of a slag may be expressed by the formula $K = 9F[(\text{CaO}) + 5(\text{FeO}) + 4(\text{MnO}) + (\text{MgO}) + (\text{Fe}_2\text{O}_3)]/[(\text{Al}_2\text{O}_3) + 2(\text{SiO}_2) + 4(\text{P}_2\text{O}_5) + (\text{S}) + (\text{SO}_3)]$, where F is a constant depending on the clay, and the analysis figures (%) are substituted for the oxide formulae.

A. R. POWELL.

See also A., Mar., 280, **Rehydration of metakaolin and synthesis of kaolin** (VAN NIEUWENBERG and PIETERS).

PATENTS.

Moulding of [powdered] ceramic material. H. L. CROWLEY, Assr. to ISOLANTITE Co. of AMERICA, INC. (U.S.P. 1,699,502, 15.1.29. Appl., 11.1.27).—The material is formed into a block under great pressure while the air is evacuated. The block is then subjected *in vacuo* to a second and greater pressure, which changes the shape without causing the block to crumble. F. SALT.

Manufacture of light-weight ceramic material. R. ERICSON, Assr. to U.S. GYPSUM Co. (U.S.P. 1,702,076, 12.2.29. Appl., 14.10.27).—After incorporating a tenacious foam in a fluid plastic mixture of finely-divided argillaceous material and water, the mixture is shaped, dried, and finally heated to form a hard, cellular product.

H. ROYAL-DAWSON.

Pottery decoration. LOVATT & LOVATT, LTD., and A. E. LOVATT (B.P. 303,679, 20.2.28).—Pottery ware is subjected to a light biscuit fire and then dipped in a mixed glaze, prepared by mixing colouring matter, which has been previously fired and crushed to suitable particle size, with raw or unfired glaze. The glaze is then fired on at a comparatively high temperature.

F. SALT.

Compound glass sheets. J. A. WATT (B.P. 305,404, 30.3.28).

Production of graded tinting on glass. O. P. RAPHAEL (B.P. 305,436, 5.7.28).

Apparatus for making glass threads or filaments. S. G. S. DICKER. From GOULD STORAGE BATTERY Co., Inc. (B.P. 306,332, 26.4.28).

IX.—BUILDING MATERIALS.

Determination of iron and aluminium oxides, magnesium oxide, and calcium oxide in Portland cement. J. S. PIERCE and W. C. SETZER (Ind. Eng. Chem. [Anal.], 1929, 1, 25–26).—After removal of silica, iron and aluminium oxides are precipitated at p_{H} 7, using bromothymol-blue as indicator. The slightly blue filtrate is acidified, boiled, and 10% of its volume of saturated alcoholic trinitrobenzene added. Carbonate-free sodium hydroxide solution is run in until a deep red colour is obtained. The precipitated magnesium hydroxide is dissolved in excess of standard acid, and the excess acid is titrated, using dimethylaminoazobenzene as indicator. The calcium in the filtrate is determined similarly. The method is an approximate one suitable for rapid control tests.

C. IRWIN.

Effect of temperature on the strength of concrete. C. C. WILEY (Eng. News-Rec., 1929, 102, 179–181).—Equal strength of concrete matured at 38° is obtained in about half the period required for that matured at 21° ,

which again requires only half the time of concrete matured at 2° . At all ages between 3 and 28 days the difference in strength was represented by 54 lb./in.² per degree. Frozen concrete showed practically no increase in strength, and it is recommended that all concrete should be protected from freezing for at least a week at 21° , or for twice as long at 2° . Loss in strength of concrete after 7 days' hardening was small, and it was indecisive whether freezing after this period resulted in any permanent damage, but when frozen at the age of 1 day the strength was less than half that of unfrozen material, and freezing at 3–5 days indicated a permanent loss of strength of 15–20%.

C. A. KING.

Prolonged tests on concrete made with various ballasts, especially blast-furnace slag. A. GUTTMANN (Arch. Eisenhüttenw., 1928–9, 2, 401–403; Stahl u. Eisen, 1929, 49, 213).—A series of tests extending over ten years on concretes made with blast-furnace slag, granite, limestone, and gravel as ballast showed that slag ballast was slightly more satisfactory than natural ballast. The sulphur content of the slag appeared to have no accelerating effect on the corrosion of iron embedded in the concrete.

A. R. POWELL.

Absorption of wood preservatives. J. D. MACLEAN (Eng. News-Rec., 1929, 102, 176–179).—Considerable variation in results occurs when timbers of different lengths and cross-sections are treated with preservatives in accordance with present specifications. The most satisfactory and convenient method of specification is in weight (lb.) of preservative per cub. ft. of timber, provided that consideration is taken of the ratio of surface area to volume. This proviso is unnecessary in the case of sapwood or porous wood, but the heartwood of most species will take only a limited penetration, so that absorptions widely differing from an average value are obtained with different sizes of timber. The side and longitudinal penetrations of a number of air-seasoned heartwood specimens gave an average ratio of about 1:15 for preservative oils and 1:20 for water solutions.

C. A. KING.

Bituminous substances. SUIDA and SCHMÖLZER. Road asphalts. BURSTYN.—See II.

PATENTS.

Production of Portland cement. H. R. DURBIN, Assr. to INTERNAT. CEMENT CORP. (U.S.P. 1,700,032–3, 22.1.29. Appl., [A] 12.6.26, [B] 22.1.27).—(A) Portland cement, having a tensile strength of not less than 240 lb./in.² after 24 hrs. in a 1:3 sand mixture, is prepared by burning Portland cement clinker containing a large proportion of tricalcium silicate and incompletely lime-saturated silicate intimately mixed with just sufficient lime to complete the saturation of the silicate, and grinding this clinker to cement. (B) A similar cement, having a tensile strength greater than 300 lb./in.² after 24 hrs. in a 1:3 sand mixture, is prepared by adding silica, alumina, and iron, together with an amount of lime which is theoretically necessary to saturate the silica, alumina, and iron, but which is considerably in excess of that which will combine with these elements in one burning operation. The mass is burned to incipient fusion, ground, re-burned to incipient fusion, and again ground.

F. SALT.

Direct vitreous coating of surfaces such as walls, constructional ironwork, roofing, fencing, etc. R. F. BANKS (B.P. 306,256, 21.1.28).—The surfaces are sprayed with a mixture of cement and solutions of sodium silicate and barium chloride, with or without a solution of acid potassium oxalate, and are thereby glazed and rendered waterproof.

H. ROYAL-DAWSON.

Aggregating particles of wood. F. LAUTER, Assr. to LIGNEL CORP. (U.S.P. 1,702,013, 12.2.29. Appl., 18.11.27).—Wood particles containing substantial amounts of natural resin are treated with an aldehyde solution, dried, and subjected simultaneously to the action of heat and pressure; the artificial resin produced acts as a binder for the wood. H. ROYAL-DAWSON.

Manufacture of [recessed] bricks or building blocks. P. W. STEWART and G. H. C. RACLIFFE (B.P. 306,210, 29.11.27).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Corrosion of iron in sodium chloride solution. W. VAN WÜLLEN-SCHOLTEN (Arch. Eisenhüttenw., 1928—9, 2, 523—530; Stahl u. Eisen, 1929, 49, 212—213).—When a smooth and a rough iron plate are immersed in sodium chloride solution and connected outside the vessel through a galvanometer a current flows from the smooth to the rough plate inside the cell. Agitation of the solution causes a reversal of this direction of flow, which is permanent with vigorous agitation, but gradually reverts to the original direction with slow stirring. In an atmosphere of hydrogen agitation has no effect. Potential measurements in the non-agitated solution show that the potential of iron to ferrous hydroxide is -0.569 volt and to ferric hydroxide -0.220 volt, but in the presence of oxygen a solid solution of these two oxides is obtained as a single phase, the ratio $\text{Fe}^{3+}/\text{Fe}^{2+}$ in the phase being dependent on the oxygen concentration in the solution. A. R. POWELL.

Sulphur in electrolytic iron and its removal. K. OMA (Bull. Inst. Phys. Chem. Res. Tokyo, 1929, 8, 126—130).—Iron deposited electrolytically from ferrous ammonium sulphate baths may contain 0.001% of sulphur as sulphur derived from the anode material, and 0.02—0.03% as sulphate derived from the electrolyte. At high temperatures the latter may be reduced to sulphide and thence to hydrogen sulphide, and the rate of desulphurisation effected by heating in hydrogen above 800° is a function of time, temperature, and grain size. J. GRANT.

Manufacture of hollow drill steel in the electric furnace. T. M. PUGSLEY (Chem. Eng. Min. Rev., 1929, 21, 131—132).—The furnace is charged with scrap steel covered with 3% of lime and some fluorspar, and heating is continued until the metal contains less than 0.02% P, more lime and fluorspar being added if necessary. The slag is removed and the metal carburised to 0.6% C by stirring in anthracite; more lime-fluorspar slag is added, together with anthracite, to produce carbide in the slag, followed by the requisite quantity of manganese. The steel is then deoxidised

with a small quantity of ferrosilicon and poured. Details of the method of pouring and of working-down the ingots are given. A. R. POWELL.

Effect of the final rolling temperature on the properties of steel rails. R. STUMPER (Stahl u. Eisen, 1929, 49, 177—187).—The mechanical properties and structure of steel rails containing 0.4% C, 0.1% Si, 1.0% Mn, 0.06% P, and 0.04% S have been examined after finishing the rolling at temperatures between 950° and 1150° . The tensile strength, elongation, yield point, and impact strength decrease generally with increase of rolling temperature, which also causes a rapid increase in grain size. Hence the rails should be finished at as low a temperature (above 950°) as is compatible with convenient handling in the rolling mill. A. R. POWELL.

Resistance of some steels to chemical action in relation to their nickel, chromium, and carbon content. F. SCHMITZ (Z. Metallk., 1929, 21, 64—65).—The work of Guertler and Ackermann on carbon-free iron-chromium-nickel alloys (B., 1928, 753) has been repeated on nickel-chromium steels with similar results, chromium being the sole protective agent against corrosion in oxidising media and nickel in non-oxidising media. A high carbon content improves the resistance of plain carbon steels to mild corrosive agents, but tends to reduce the passivity of alloy steels, although it decreases scaling at high temperatures.

A. R. POWELL.

Systems iron-silicon, iron-chromium, and iron-phosphorus. P. OBERHOFFER and C. KREUTZER (Arch. Eisenhüttenw., 1928—9, 2, 449—456; Stahl u. Eisen, 1929, 49, 189—190).—Röntgenographic examination of the iron-silicon system shows the γ -field to be bounded by a smooth curve joining 1400° and 900° on the temperature axis and extending to 2.2% Si at 1150° . The heterogeneous ($\alpha + \gamma$)-field is bounded by a similar curve extending to 2.5% Si at 1180° . X-Ray analysis affords no evidence of the formation of an intermetallic compound in the iron-chromium system; the γ -field extends to about 15% Cr. In the iron-phosphorus system, alloys containing up to 1.11% P have the simple α -lattice structure, those with more than 2.28% P show in addition the lines of the compound Fe_3P , and alloys with 14.2—21.2% P contain both Fe_3P and Fe_2P . With larger proportions of phosphorus new lines appear in the röntgenogram due to the presence of an, as yet, unknown constituent. A. R. POWELL.

Anomalies of annealing cold-worked copper and brass. F. EUGÈNE (Rev. Mét., 1928, 25, 685—706; 1929, 26, 29—42; cf. B., 1928, 861).—The changes in the physical and mechanical properties of cold-worked copper and brass on annealing through the recrystallisation zone are shown in a series of tables and graphs, and photomicrographs of the structural changes that take place in the zone between the two anomalies A1 and A2 are reproduced. The maximum softness and Erichsen value of copper are obtained by annealing at A2, whereas the malleability of brass increases with rise of annealing temperature between A2 and the temperature at which recrystallisation is complete. Micrographic examination shows that all the strained crystals have disappeared

completely at A2, but internal stress is relieved as soon as the individual strained crystals are completely isolated from one another by areas of recrystallised metal. Dilatometric tests on cold-worked copper showed contraction to take place on annealing in all directions simultaneously, whereas with brass contraction takes place in a direction transverse to the direction of rolling and expansion in the direction of rolling; this effect is the more pronounced the higher is the zinc content.

A. R. POWELL.

Copper-cuprous oxide eutectic in copper refining. W. STAHL (Chem.-Ztg., 1929, 53, 137).—The formation of a cuprous oxide-copper eutectic containing 3.4—3.5% Cu_2O is responsible for the fibrous structure which copper acquires during refining by poling. Similar behaviour is observed when pure copper is oxidised.

J. S. CARTER.

Brittleness in arsenical copper. II. C. BLAZEY (Inst. Metals, March, 1929. Advance copy. 6 pp.).—Following an observation of a type of brittleness in copper which disappeared on annealing, and re-appeared when the metal was cold-worked (cf. B., 1927, 280), the influence of phosphorus, lead, and bismuth has been determined. Phosphorus or antimony up to 0.1% was without ill-effect, but 0.004% of bismuth was harmful when no phosphorus was added before pouring, and the effect was characteristic of low-temperature annealing only.

C. A. KING.

Testing of electrodeposits on aluminium. G. B. BROOK and G. H. STOTT (Inst. Metals, March, 1929. Advance copy. 9 pp.).—Methods designed to determine the probable durability of electroplating on aluminium included the determination of weight, thickness, and degree of porosity of the deposit. The measure of adhesion was shown by bending a strip of the plated metal helically around an iron rod under standard conditions, and the resistance to corrosion by immersing specimens in brackish water at half-tide level for three months. Considerable variation in thickness, soundness, and crystalline structure was found in specimens representing commercial practice. Generally, failures, e.g., cracking, blistering, peeling of the plating, were confined to single-metal deposits, some multiple deposits remaining free from these defects for long periods. A particular chromium-plated specimen showed evident poor adhesion.

C. A. KING.

Rapid determination of tin in tinplate. W. A. MASEL (Chem.-Ztg., 1929, 53, 108).—A weighed sample is placed in a short test-tube with a small hole in the bottom and the tube is lowered, by means of a piece of iron wire round the neck, into a cylinder filled with a solution of chlorine in tin tetrachloride. Dissolution of the tin coating is complete in 3—4 min. when no further bubbles develop on the iron. The tube is removed and the iron thoroughly washed with benzine, dried, and weighed. The loss in weight, plus 0.1% for tin retained as a compound in the outer iron layer, is the amount of tin in the sample taken.

A. R. POWELL.

Evaluation of stibnite. I. **Determination of sulphur.** W. M. McNABB and E. C. WAGNER (Ind. Eng. Chem. [Anal.], 1929, 1, 32—35).—The evolution method for the determination of sulphur in stibnite

gives results which, though concordant in themselves, are lower than those given by bromine oxidation. The difference was suspected to be due to the presence of free sulphur. Specimens of Chinese stibnite gave on extraction with carbon tetrachloride up to 0.10% of free sulphur. They contained about 0.01% of sulphate sulphur. The same samples were analysed by the bromine oxidation and evolution methods, and differences approximating to the actual free sulphur content were found. The evolution method, in which the hydrogen sulphate is evolved in a current of carbon dioxide or hydrogen, absorbed in ammoniacal cadmium solution, and the cadmium sulphide titrated with iodine, is therefore accurate. It permits of the antimony being determined from the same sample.

C. IRWIN.

Variations in silver assaying. F. C. JOHNS (Chem. Eng. Min. Rev., 1929, 21, 152—154).—In the assay of lead-zinc sulphide ores and concentrates for silver equally good results are obtained by the scorification and pot-running (nitre) methods, whereas slightly low results are obtained by using an iron rod in the pot assay. With bone ash cupels there is a greater absorption of silver than with Mabor or Morganite cupels. Examples are given of the variations that may be expected in the assay of these products for silver; even with careful adjustment of the cupellation conditions differences of 1—4 oz./ton on material assaying 70—80 oz./ton may be obtained.

A. R. POWELL.

Detection of gold and the platinum metals [in ores]. SPONCQ (Bull. Soc. chim. Belg., 1929, 38, 21—24).—The ore is smelted in the usual way to obtain a lead button, which is cupelled and the resulting bead is heated in a porcelain crucible with a few drops of nitric acid. When no further action is seen, a few drops of hydrochloric acid are added and the solution is evaporated on the water-bath to dryness. The residue is moistened several times with hydrochloric acid and dried to expel nitric acid completely. The final residue is moistened with 1 drop of hydrochloric acid, and 2 drops of a 20% solution of stannous chloride in 1:1 hydrochloric acid are added. If gold is present a blackish-blue colour is obtained, and after keeping for some time a black precipitate separates; if platinum is present the supernatant liquid will be brown, and if palladium is present brick-red. Detection of as little as 0.01 g. of these metals per ton of ore is claimed when 100 g. are taken for the test.

A. R. POWELL.

Annealing of metals *in vacuo*. L. GUILLET and A. ROUX (Rev. Mét., 1929, 26, 1—11).—Three samples of commercial steel were annealed *in vacuo* and the rate of evolution of gas and its composition determined. With mild steel (0.04% C) gas continued to be evolved for 23 hrs. at 950°, the total volume expelled being 3.3 times the volume of the steel; the gas contained 40% H_2 , 37% CO , 22% N_2 , and 1% CO_2 by vol. From a cementation steel (0.13% C) 0.67 vol. of gas was evolved in 5 hrs., the gas containing 43% H_2 , 40% CO , 8% N_2 , and 9% CO_2 by vol., and from a steel with 0.45% C, 0.53 vol. of gas containing 41% H_2 , 39% CO , 14% N_2 , and 6% CO_2 by vol. was evolved in 4 hrs. Except in the case of mild steel, annealing *in vacuo* had little effect on the mechanical properties. The resistance to shock of mild

steel was, however, considerably increased by vacuum-annealing. Brass containing 67% Cu evolved 0.55 vol. of gas containing 65% H₂, 14% CO, 7% N₂, and 14% CO₂ by vol. on annealing for 2 hrs. at 720°, whereas 60:40 brass evolved only 0.39 vol. of gas containing 56% H₂, 43% N₂, and 9% CO₂ by vol. under the same conditions. A pure nickel wire evolved 22 times its volume of gas in 2 hrs. at 950° *in vacuo*; the gas contained by vol. 36% H₂, 33% CO, 26% N₂, and 5% CO₂. Aluminium and duralumin commence to evolve gas at 300–400° *in vacuo*; the volume and composition of the gas vary very widely according to the previous history of the metal, but usually the gas is very rich in hydrogen and poor in nitrogen, and it frequently also contains methane.

A. R. POWELL.

Determination of gases in metals. W. HESSENBRUCH (Z. Metallk., 1929, 21, 46–55).—A review of recent work, with especial reference to steel, giving diagrams of suitable apparatus and a summary of the results obtained by previous authors. A. R. POWELL.

Explosibility of sulphide dusts in metal mines. E. D. GARDNER and E. STEIN (U.S. Bur. Mines Rep. Invest., 1928, No. 2863, 11 pp.).—Sulphide dusts can be ignited by blasting, and combustion may proceed with explosive violence. CHEMICAL ABSTRACTS.

Cathodic yield in nickel-plating with high current density. Influence of oxidising agents and hydrogen-ion concentration. BALLAY (Compt. rend., 1929, 188, 556–559).—Nickel-plating on steel from a solution containing 450 g. of nickel sulphate, 22 g. of nickel chloride, and 22 g. of boric acid per litre for 15 min. at 50° with a current density of 10 amp./dm.² gives a yield of over 95%, which is only slightly influenced by small amounts of oxidising agents, though excess of nitric acid or nitrates, or to a smaller extent potassium permanganate, diminishes it considerably. The optimum *p*_H range is 4.4–5.5. J. GRANT.

Importance of design and setting of large kettles used for refining and low m.p. alloys. H. C. LANCASTER (Inst. Metals, Mar., 1929. Advance copy. 6 pp.).

See also A., Mar., 248, **Recrystallisation of metals** (KARNOP and SACHS). 259, **Colloidal gold** (NICOL). 270, **Passivity of metals** (EVANS). 271, **Mechanism of corrosion** (EVANS). 275, **Electrolytic precipitation of metals** (ARNDT). **Theory of electrodeposition of chromium** (MÜLLER and ERWALL). 287, **Microtitration of bismuth** (STRAUB). **Determination of tantalum and niobium** (SEARS). **Determination of osmium** (CROWELL and KIRSCHMAN). 289, **Iron ores of S. Africa** (KRENKEL).

Gas for cutting and welding. SCHOLTZ.—See II. **Testing for corrosion.** DUFFEK.—See XI.

PATENTS.

Apparatus for roasting and/or sintering ores etc. COMP. DES MÉTAUX OVERPELT-LOMMEL (B.P. 294,197, 30.3.28. Belg., 20.7.27).—Sintering apparatus of the Dwight-Lloyd type is provided with one or more joints in series with the usual joint between the movable grate and the suction or blowing box so as to form one or more chambers inside which a pressure is maintained,

by means of a fan, equal to that in the suction or blowing box. An automatic regulating device is provided for keeping these pressures in equilibrium.

A. R. POWELL.

Reduction of ores. F. L. DUFFIELD (B.P. 304,174, 10.10.27).—A charge of iron ore, carbonaceous material, and lime is introduced into the base of a vertical furnace by means of a horizontal ram working in conjunction with a vertical ram which intermittently elevates the charge into progressively hotter zones of the furnace, the reduced metal being discharged at or near to the top of the chamber. A one-way stop-valve supports the charge on the downward stroke of the ram. Gases evolved in the reducing chamber are burned in an adjacent heating chamber, the gases passing in a downward direction and providing the necessary heat for the reducing process. C. A. KING.

Chill-cast iron alloys. Grey-iron alloys. INTERNATIONAL NICKEL Co., Assees. of P. D. MERICA, J. S. VANICK, and T. H. WICKENDEN (B.P. 279,414–5, 29.9.27. U.S., 21.10.26).—(A) A chilled cast-iron alloy, a portion at least of which is of chilled white iron, contains 0.25–2% Si, 2–10% Ni, 1–4% Cr, and 0.25–2% Mn, besides the usual carbon content of cast iron. (B) A cast-iron alloy resistant to wear contains 0.25–3% Si, 1–12% Ni, up to 3% Cr, 2–4% C, and the usual content of manganese, sulphur, and phosphorus. [Stat. ref.] A. R. POWELL.

Manufacture of grey cast iron with low carbon content and of any desired composition. MASCHINENFABR. ESSLINGEN (B.P. 291,112, 25.5.28. Ger., 27.5.27. Addn. to B.P. 260,990; B., 1927, 960).—On the basis of the relation between the thickness of wall, the composition of metal, and the formation of structure of grey cast iron, the carbon content is maintained constant and the structure determined by variation of the silicon content in accordance with a graph correlating the wall thickness and the silicon content for a given percentage of carbon. C. A. KING.

Manufacture of malleable cast iron. C. E. KLUJTMANS and W. H. W. PROCTOR (B.P. 288,980, 14.4.28. Fr., 16.4.27).—A charge consisting of equal parts of steel scrap and the residues from previous melts is smelted in a cupola with the addition of ferrosilicon and ferromanganese to obtain an iron containing up to 2.9% C, 1.1–1.5% Si, 0.42–0.75% Mn, 0.1–0.2% P, and less than 0.2% S. The castings are embedded in sand or waste iron ore, heated for 12 hrs. at 680–730°, 12 hrs. at 800–830°, and 48 hrs. at 880–900°, cooled to 700° at the rate of 5–8°/hr., and then cooled rapidly to produce black-core malleable iron. A. R. POWELL.

Increasing the elastic limit or yield point of tough or plastic metals. G. KERÉKGYÁRTÓ (B.P. 295,390, 7.8.28. Hung., 11.8.27).—Wrought iron or steel shafts, tubes, etc. are subjected to a torsional stress beyond the elastic limit or yield point, and then annealed at a low temperature to relieve internal stress.

A. R. POWELL.

Proofing of iron and steel against rust. W. H. COLE (B.P. 305,386, 28.2.28. Addn. to B.P. 289,906; B., 1928, 488).—The pickling solution is made by

dissolving in hot phosphoric acid (1 litre of acid, *d* 1.45, diluted to *d* 1.09) 150 g. of zinc oxide, 250 g. of ferric oxide, 10 g. of chromium oxide, 10 g. of aluminium powder, 3 g. of potassium dichromate, 4 g. of potassium chromate, 2 g. of ammonium phosphate, and 6 g. of naphthalene.
A. R. POWELL.

[Heat-resisting chromium-iron] alloys. T. D. KELLY (B.P. 304,893, 23.12.27).—Iron of a good quality is melted by means of a flame produced from powdered anthracite, and to every 100 lb. of iron is added an alloy consisting of (a) 5–20 lb. of chromium, 5–20 lb. of nickel, and 5–20 lb. of copper, (b) 5–20 lb. of chromium, 10–20 lb. of nickel, 10–20 lb. of copper, and 60–80 lb. of carbon-free iron, or (c) 5–20 lb. of chromium and 5–20 lb. of copper. In addition, the alloy may contain 2–20 lb. of manganese and/or silicon and 0.2–2% of vanadium, molybdenum, or tungsten.
A. R. POWELL.

Prevention of corrosion of articles made of aluminium and aluminium alloys. B. JIROTKA (B.P. 305,149, 28.10.27).—The articles are immersed in nitric acid of a concentration greater than 25% whereby a protective oxide film is formed. Coloured films are produced by addition of salts of heavy metals, *e.g.*, chromium, cobalt, copper, to the nitric acid bath, and the duration of pickling may be shortened by connecting the article to a rod or plate of graphite or of a heavy metal so that electrolytic action ensues. To obtain a rough surface for subsequent varnishing hydrochloric acid is added to the pickling bath.
A. R. POWELL.

Production of coatings on aluminium and its alloys. B. JIROTKA (B.P. 286,729, 9.3.28).—To the baths containing zinc-cyanogen compounds, or a mixture of zinc salts and alkali cyanides, or zinc compounds and alkali carbonates, an alkali borate is added; *e.g.*, the bath may consist of a mixture of zinc carbonate, alkali cyanide, and borax. The hot solutions may be sprayed or painted on to the metal.
M. E. NOTTAGE.

Decomposition of chromium ore and manufacture of chromium compounds free from iron. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 305,712, 10.11.27).—The ore is opened up either by quenching it, or by melting it with an alkali hydroxide or carbonate in a non-oxidising atmosphere. It is then treated with hydrogen chloride and/or chlorine at a temperature not above 800°, preferably 500–650°, in the presence or absence of a reducing agent.
M. E. NOTTAGE.

Recovery of tin contained in the residues of tin plate manufacture. LES PETITS FILS DE F. DE WENDEL & CIE. (B.P. 304,639, 15.3.28. Fr., 23.1.28).—The residues are agitated in a perforated rotating trommel immersed in a hot solution containing 2% of sodium hydroxide and 2% of sodium carbonate. The oily material separates as a scum on the surface of the liquid, and the earthy impurities and oxides fall through the perforations in the trommel, leaving a clean metallic residue from which a high-quality tin is recovered by reverberatory liquation.
A. R. POWELL.

Metallurgical process [for nickel]. J. H. WHITE, ASST. to BELL TELEPHONE LABS., INC. (U.S.P. 1,700,460,

29.1.29. Appl., 15.12.26).—Small amounts of vanadium and magnesium are added to molten nickel to enable it to be forged.
H. ROYAL-DAWSON.

Silicon-containing magnesium alloys for use with pistons for internal-combustion engines. I. G. FARBENIND. A.-G. (B.P. 293,359, 4.7.28. Ger., 4.7.27).—The alloys contain up to 15% Al, 1–6% Si, and copper, zinc, or cadmium, either alone or together, in such amounts that the m.p. does not fall below 400°.
A. R. POWELL.

Tantalum-alloy pen. E. W. ENGLE and M. M. AUSTIN, ASSES. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,701,299, 5.2.29. Appl., 27.5.27).—An alloy containing 90% Ta and 10% W is used for the nib.
H. ROYAL-DAWSON.

Decomposition of ores of zirconium and other rare-earth metals, and of titanium. DEUTS. GASGLÜHLICHT AUER-GES.M.B.H. (B.P. 291,004, 26.4.28. Ger., 23.5.27).—The ore is treated with sulphuric acid and the earths are separated as sulphates or basic sulphates in the usual way; the mother-liquor is evaporated and the residue heated to drive off excess sulphuric acid and to decompose any sulphates, *e.g.*, ferric sulphate. The mixed gases from this operation and from the calcining of the sulphates of the earths are mixed with air and passed over a fresh charge of ore to effect partial sulphatising of the earths. The product is then digested as usual with sulphuric acid. The process effects considerable economies in the amount of acid required to decompose the ores.
A. R. POWELL.

Vessels having an acid-proof lining. I. G. FARBENIND. A.-G. (B.P. 290,189, 25.1.28. Ger., 9.5.27. Addn. to B.P. 283,964; B., 1929, 100).—The lining consists of silicon alloy slabs containing not less than 50% Si. Alloys of silicon with chromium, copper, manganese, or tungsten are suitable.
H. ROYAL-DAWSON.

Treatment of minerals. McK. STOCKTON, ASST. to CELITE CO. (Re-issue 17,212, 12.2.29, of U.S.P. 1,611,791, 21.12.26).—See B., 1927, 114.

Processing sheets to be used in can making. E. W. BLISS CO., ASSEES. of P. KRUSE (B.P. 289,052, 12.4.28. U.S., 22.4.27).

Disintegration of [low-grade, ore-bearing] clays and similar materials. J. F. NEWSOM (B.P. 302,420, 24.9.27).

Separation of materials (B.P. 275,970).—See I. Refining of aluminium (B.P. 293,353). Electrodeposition of chromium (B.P. 301,478). Metal foils and sheets by electroplating (U.S.P. 1,698,472 and 1,698,486).—See XI.

XI.—ELECTROTECHNICS.

Improved form of electric resistance furnace. W. ROSENHAIN and W. E. PRYTHERCH (Inst. Metals, March, 1929, Advance copy. 5 pp.).—A resistance furnace developed primarily for laboratory use at temperatures above those obtained by means of wire-wound furnaces is heated by means of carbon resistor elements. These elements consist of pellets or short rods of carbon or graphite loosely fitting into a sheath

of refractory material. High temperatures are obtained by the passage of a relatively small current through the contact surfaces between successive rods or pellets, and the use of solid end-pieces provides the resistor with comparatively cool ends without the necessity of water-cooling. Oxidation of the carbon is very slow even when working at 1300° or 1400°. A small muffle and an experimental steel-melting furnace have been heated by means of a number of sheathed resistors, and the type evolved may be suitable for more extended use.

C. A. KING.

Recent developments in electric furnaces. D. F. CAMPBELL (Inst. Metals, March, 1929. Advance copy. 20 pp.).—In melting practice, as applied largely to brass works, the development of the Ajax-Northrup and Ajax-Wyatt induction furnaces has eliminated the use of expensive crucibles. In the modern furnace the lining is formed by filling the space between a metal liner and the induction coil with a suitable refractory composition, and fritting the material by means of heat induced in the liner, which is later melted out in the first charge. Ajax-Wyatt furnaces of 600 or 1200 lb. capacity are now preferred for intermittent working rather than the heavier types, and the type having a vertical secondary channel is in most common use. Mention is made of furnaces for heat-treatment and also of a double-track, tunnel pottery kiln heated by nickel-chromium spiral resistors inserted into grooved bricks.

C. A. KING.

Theoretical considerations in electric tunnel kiln design. J. KELLEHER (Amer. Electrochem. Soc., May, 1929. Advance copy. 6 pp.).—A method is described for designing an electric tunnel kiln for heat-treating materials according to a specified temperature-time cycle.

II. J. T. ELLINGHAM.

Control of the current density in electrolytic baths. W. PFANHAUSER (Chem.-Ztg., 1929, 53, 129—130).—An instrument for measuring the current density in electrolytic baths comprises two circular metal electrodes each 0.5 dm.² in area set into a hard rubber case so that they are parallel to but insulated from one another. The plates are connected, by means of wires inside a tube attached to the rubber case, to a measuring instrument which records directly the current density when the plates are immersed between the anode and the cathode. A second scale shows the time required to obtain a definite thickness of deposit, using the current density recorded on the first scale.

A. R. POWELL.

Electrical resistance of liquid insulating materials. A. GYEMANT (Physikal. Z., 1929, 30, 33—58).—A critical review of the literature relating to the breakdown of liquid dielectrics. The relations between chemical constitution and physical structure and the breakdown field strength of organic liquids are considered, together with the rôle of electrical conditions, temperature, and pressure.

R. A. MORTON.

Dissolution of plain and amalgamated zincs in electric batteries. J. N. FRIEND (Inst. Metals, March, 1929. Advance copy. 4 pp.).—The results of comparative treatments of plain, high-grade (99.9%) zinc and ordinary commercial (98—99%) zinc in 10% sul-

phuric acid, and in solutions of ammonium chloride in ordinary use in Leclanché cells, showed that the use of pure zinc was not warranted. In the dilute acid amalgamated pure zinc may be less resistant to attack than ordinary amalgamated rod, though little difference was noted in ammonium chloride solutions. Plain high-grade zinc is not recommended in place of amalgamated commercial metal.

C. A. KING.

Apparatus for testing liability to corrosion. V. DUFFEK (Gas- u. Wasserfach, 1929, 72, 127—129).—The material to be tested is immersed, together with a mercury electrode, in a vessel containing the electrolyte (e.g., water) the action of which is to be examined. External connexion is made through a milliammeter and a resistance, the latter being sufficiently great that the normal corrosive action of the electrolyte on the material is not affected by the current passing. The whole is placed inside a closed vessel, and provision is made for passing a stream of oxygen through the electrolyte during the test. If no corrosion takes place the current passing between the material and the mercury electrode gradually falls, and a sudden increase in the latter indicates the beginning of corrosive action. Working with 60 mm. pressure (water-gauge) of oxygen in the apparatus, the velocity of corrosion is about 120 times that under normal atmospheric conditions. The apparatus may be used for determining the liability of metals and alloys to corrosion by liquids, in the presence of air or other gases, at various pressures, or the efficiency of various protective coverings on metals etc.

W. T. K. BRAUNHOLTZ.

See also A., Mar., 240, **Deterioration of quartz mercury-vapour lamps** (GILLAM and MORTON). 274, **Preparation of hydroxylamine** (STSCHEBAKOV and LIBINA). 275, **Preparation of sodium perborate** (GIORDANI and INTONTI). **Electrolytic precipitation of metals** (ARNDT). **Theory of electrodeposition of chromium** (MÜLLER and EKWALL).

Removal of tar fog from gases. FISCHER. **Analysis of gasoline.** ABORN and BROWN.—See II. **Colour of light sources.** CUNLIFFE and LANIGAN.—See VI. **Nickel-plating.** BALLAY.—See X.

PATENTS.

Electric induction furnace. E. F. NORTHROP (B.P. 305,620, 8.9.27. Cf. B.P. 201,954; B., 1929, 101).—Current is supplied to the furnace coil in a number of phases by a polyphase transformer in which the number of secondary phases is a multiple of the number of primary phases, whereby greater dissipation of energy in the charge is attained and the power factor improved.

J. S. G. THOMAS.

Induction furnace. E. F. RUSS (B.P. 306,172, 15.11.27).—Part of the secondary circuit is used as a heating element, the secondary circuit comprising, in series, substantially smooth, corrugated, or ribbed surfaces of high resistance forming a substantially rectilinear muffle or trough, and a secondary low-resistance member surrounding the primary and heating a preheating chamber.

J. S. G. THOMAS.

[Electrodes for] alkaline accumulators. J. BACSA (B.P. 305,680, 8.8.27 and 12.4.28).—Electrodes.

are prepared by the repeated anodic oxidation of nickel, cobalt, or iron electrodes in alkaline solution and cathodic deposition of the metal from neutral solution and simultaneous conversion from the higher into the lower state of oxidation. Alternatively, cadmium repeatedly deposited from a neutral cadmium salt solution upon a cadmium or zinc electrode is subjected to anodic oxidation and subsequent cathodic reduction in an alkaline electrolyte. J. S. G. THOMAS.

Calomel electrode vessel. D. S. HAKE, Assr. to ACHESON GRAPHITE Co. (U.S.P. 1,700,796, 5.2.29. Appl., 2.11.26).—The cell has an inlet tube near its upper end for the introduction of potassium chloride solution, and a contact tube extending from the cell at a point below the inlet the end of which extends into the contact tube. H. ROYAL-DAWSON.

Production of cathodes for electric discharge tubes. VEREIN. GLÜHLAMPEN U. ELECTRICITÄTS A.-G. (B.P. 289,763, 18.11.27. Austr., 30.4.27).—Vapours of alkaline-earth metals liberated by a reduction process started by heating, *e.g.*, by reduction of barium hydroxide with magnesium or calcium, are deposited upon a core of platinum, copper, nickel, or chrome-nickel.

J. S. G. THOMAS.

Manufacture of thermionic cathodes for electric discharge devices. M. O. VALVE Co., LTD., A. L. REIMANN, and J. W. RYDE (B.P. 306,281, 20.2.28).—A metallic core, *e.g.*, of platinum-rhodium alloy, is coated with a mixture of alkaline-earth oxides and metallic thorium. J. S. G. THOMAS.

Manufacture of liquid or solid products by gaseous reaction under the influence of silent electrical discharge. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 304,623, 20.10.27).—The cooling fluid of the discharge chamber is controlled so that the temperature rises above the saturation point of the gaseous mixture, whereby deposition of reaction products upon the walls of the chamber is prevented or reduced. If desired, a liquid not taking part in the reaction is caused to flow over the chamber walls in order to dissolve or wash away reaction products. [Stat. ref.] J. S. G. THOMAS.

Glow-lamp filaments. SOC. FRANÇ. DES LAMPES À INCANDESCENCE "LUXOR" (B.P. 288,557, 11.4.28. Fr., 11.4.27).—A compound filament is made of two layers of the same metal having different resistances and bent preferably into U-shape. J. S. G. THOMAS.

Electrolytic apparatus and method of operation. E. THOMSON, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,701,346, 5.2.29. Appl., 25.3.26).—Annular electrolytic cells subdivided by permeable partitions are arranged in stacked relation at right angles to the axis of rotation of a receptacle having a hollow shaft which is used to collect separately the gases evolved from the positive and negative parts of the cells. J. S. G. THOMAS.

Electrolytic apparatus for refining aluminium etc. SOC. ANON. COMP. DE PROD. CHIM. & ÉLECTRO-MÉTALLURGIQUES ALAIS, FROGES, & CAMARGUE, Assees. of P. L. HULIN (B.P. 293,353, 19.6.28. Fr., 4.7.27).—In electrolytic apparatus in which the cathode metal is removed periodically while the anode is reconstituted

suitably, either by admixture of impure light metal or by partial or complete replacement of the mass, there are three superposed fused layers, *viz.*, an anode of a dense alloy of the metal to be extracted, a halogenated bath forming the electrolyte, and a light metal forming the floating cathode. The inner lining comprises a horizontal, refractory frame containing the anode, a horizontal insulating frame containing electrolyte which solidifies on a frame through which water is circulated, and a frame composed substantially of carbon surrounding the cathode and insulated from the metal shell.

J. S. G. THOMAS.

Electrolytic deposition of chromium. LANGBEIN-PFANHAUSER-WERKE A.-G. (B.P. 301,478, 2.1.28. Ger., 1.12.27).—Chromium is electrolytically deposited from neutral or slightly acid solutions of chromium compounds, *e.g.*, chromium acetate, sulphate, chloride, etc., to which is added sodium citrate, Rochelle salt, or other substance, *e.g.*, oxalic acid, phenolsulphonic acid, alcohol, glycerin, sugar, etc., for retaining chromium oxide in solution. The p_H value of the electrolyte is maintained between 4 and 6, and, if desired, colloids or other substances having a capillary action, *e.g.*, derivatives of aromatic hydrocarbons containing hydroxyl groups, which prevent the growth of crystals, may be added to the bath when bright, polished deposits are required. The current density employed is greater than, but of the order of, 0.25 amp./dm.² Bags containing chromium carbonate may be suspended in the catholyte, which is separated from, and has a higher concentration than, the anolyte. J. S. G. THOMAS.

Making metal foils. J. R. CAIN, Assr. to RICHARDSON Co. (U.S.P. 1,698,472, 8.1.29. Appl., 3.5.26).—A film of metal powder deposited and floating on the surface of an electrolyte is maintained in contact with a spongy electrode, and an electric current is passed between this electrode and an anode. J. S. G. THOMAS.

Making metallic sheets by electroplating. G. YUNGBLUT, Assr. to RICHARDSON Co. (U.S.P. 1,698,486, 8.1.29. Appl., 19.9.27).—A cathode shaped like the desired article but of a different metal is plated on one face with the required metal. The cathode metal is then stripped from the plated body in an electrolytic cell in which a similar cathode is used and upon which the stripped metal is plated. J. S. G. THOMAS.

Apparatus for the determination of the amount of carbonic acid in flue gases. H. GRÜSS, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,701,181, 5.2.29. Appl., 11.10.27. Ger., 25.10.26).—In an apparatus of the catharometer type, a double coil is inserted in the diagonal branch of the Wheatstone bridge, and the current supply to the bridge is such that the testing wire surrounded by flue gases and the wire surrounded by the comparison gas are at the same temperature between 200° and 350°. J. S. G. THOMAS.

Manufacture of electrical condensers and materials therefor. E. W. JODREY (B.P. 305,515, 3.11.27 and 1.2.28).—Paper coated with an adhesive, continuous, conducting layer of copper or other metal foil is coated with cellulose esters, *e.g.*, cellulose acetate or collodion. J. S. G. THOMAS.

Manufacture of moulded articles such as switch bases, fuse holders, etc. J. A. CRABTREE and J. R. DOLPHON (B.P. 306,249, 12.1.28).—Shields of refractory and insulating material, *e.g.*, asbestos, are caused to adhere (a) by heat, pressure, shrinkage, etc. to parts of the moulding liable to become hot due to arcing etc., or (b) by means of a cement after treatment with chemicals, *e.g.*, dilute hydrochloric acid, for extraction of organic and/or inorganic constituents. J. S. G. THOMAS.

[Cathode for] electric discharge tube. G. HOLST and E. OOSTERHUIS, Assrs. to N. V. PHILIPS' GLOEILAMP-ENFABR. (U.S.P. 1,701,849, 12.2.29. Appl., 28.7.23. Fr., 21.2.23).—See B.P. 211,825; B., 1924, 551.

Electric discharge device. BRIT. THOMSON-HOUSTON Co., LTD., Asses. of A. W. HULL (B.P. 282,766, 23.12.27. U.S., 23.12.26).

Filament for incandescence lamps etc. F. SKAUPY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,701,342, 5.2.29. Appl., 20.4.25. Ger., 3.5.24).—See B.P. 233,294; B., 1925, 952.

[Coir-bound electrodes for] electric secondary batteries or accumulators. H. F. JOEL (B.P. 305,738, 21.9.28).

[Holding plates in] electric storage batteries. R. BOSCH A.-G. (B.P. 283,490, 3.8.27. Ger., 11.1.27).

Conveyors for baths for treatment of objects, more particularly electrolytic baths. SIEMENS & HALSKE A.-G. (B.P. 298,184, 17.2.28. Ger., 5.10.27).

High vacua (B.P. 280,560).—See I. **Transformation of gaseous hydrocarbons** (B.P. 304,914). **Lubricating and insulating oils** (B.P. 305,553).—See II. **Insulated wire** (B.P. 305,084).—See XIII.

XII.—FATS; OILS; WAXES.

Density of lard. W. HALDEN and R. KUNZE (Chem. Umschau, 1929, 36, 61—62). The recorded observations of the density of lard were collected and corrected by calculation to the values for d_{15}^{15} for comparison: according to the large majority of observations this value falls between the limits 0.915—0.923 (never exceeding 0.93), and this range should be substituted for the higher values current in many text-books.

E. LEWKOWITSCH.

Analysis of fatty substances in Wood's light. A. G. NASINI and P. DE CORI (Annali Chim. Appl., 1929, 19, 46—54).—Addition of natural pigments soluble in oils (carotinoids, chlorophyll) may impart to refined olive oil the optical properties in Wood's light characteristic of virgin olive oil. Results are given of a comparison of the fluorescence and absorption spectra of solutions of chlorophyll in refined olive oil and other natural oils. The fluorescence of various cholesterol is due to the presence of impurities. T. H. POPE.

[Catalytic] hydrogenation of highly unsaturated acids. II. **Course of hydrogenation of methyl esters of highly unsaturated acids with a platinum catalyst.** Y. TOYAMA and T. TSUCHIYA (Chem. Umschau, 1929, 36, 49—52; cf. B., 1926, 286).—The methyl esters of the highly unsaturated acids from Japanese sardine oil were hydrogenated in alcoholic solution in

the presence of platinum-black at ordinary pressure; by this method the formation of solid acids proceeds from the beginning of the reaction. The amount of acids yielding insoluble bromides decreases steadily during hydrogenation, and it is shown that practically no acids of the series $C_nH_{2n-6}O_2$ and $C_nH_{2n-5}O_2$ yielding ether-insoluble bromides are formed; on the other hand, acids of the series $C_nH_{2n-6}O_2$, and possibly of $C_nH_{2n-4}O_2$, yielding bromides insoluble in light petroleum, are produced in appreciable quantities. Solid acids of the series $C_nH_{2n-2}O_2$ are formed, having m.p. notably higher than those of the naturally occurring acids of this series: solid acids of the series $C_nH_{2n-4}O_2$ with 20 and 22 carbon atoms are also formed.

E. LEWKOWITSCH.

Polymerisation of highly unsaturated [fatty] acids. I. Y. TOYAMA and T. TSUCHIYA (Chem. Umschau, 1929, 36, 45—49).—The highly unsaturated fatty acids (iodine value 351, ether-insoluble bromides 129%), isolated from Japanese sardine oil by the sodium salt-acetone method and fractionation of the methyl esters, were polymerised by heating at 240—320° in an atmosphere of carbon dioxide; the products were examined at various stages of the reaction. As heating proceeds the iodine value falls, the sp. gr. and refractive index rising approximately proportionally; the specific viscosity, however, rises more rapidly, the curve being almost vertical when the iodine value has dropped to 250. The acid value and saponif. value decrease, whilst the ester value increases. The mol. wt. of the fatty acids (camphor f.p. method) rises to about double its original value. The actual degree of temperature to which the oil is raised produces greater effect than prolonged heating. By extraction of the syrupy polymerisation products with light petroleum, insoluble resinous substances (m.p. 69—70°) were obtained. Since the yield of ether-insoluble bromides decreases more rapidly (none, when iodine value is 198) than the mol. wt. increases, it is concluded that, in the first stages of polymerisation, intramolecular change—probably ring-formation—preponderates over extramolecular polymerisation.

E. LEWKOWITSCH.

Fats. XIII. **Partial addition of halogen to polyunsaturated fatty acids.** **Glyceryl β -elaeostearate and wood [tung] oil.** II. P. KAUFMANN [with C. LUTENBERG] (Ber., 1929, 62, 392—401; cf. B., 1926, 447, 758).—Glyceryl β -elaeostearate when treated with a 100% excess of bromine in carbon tetrachloride in the absence of light absorbs 2 mols. of halogen in the interval 3—8 hrs., after which addition commences at the third double linking. Determination of the corresponding iodine value in tung oil is effected by mixing 0.1—0.15 g. of the sample in 10 c.c. of pure carbon tetrachloride with 30—40 c.c. of 0.1N-bromine in the same solvent, preservation of the mixture in the dark for 4—5 hrs., addition of aqueous potassium iodide, and titration of the liberated iodine. Subsequent addition of potassium iodate to the solution causes only very slight liberation of iodine, so that substitution has not occurred to an appreciable extent. For the determination of a single unsaturated linking, glyceryl β -elaeostearate or tung oil (0.1—0.12 g.) is dissolved in a mixture of equal volumes of chloroform and carbon tetrachloride (distilled

over phosphoric oxide). A solution (20 c.c.) of bromine and iodine is added, prepared by addition of the requisite weight of iodine to a 0.1*N*-solution of bromine in methyl alcohol containing sodium bromide. After 3—4 hrs. the mixture is treated with 5% aqueous potassium iodide and the liberated iodine is titrated. The "partial iodine value" thus determined coincides with or differs by 1—2 units from the thiocyanogen value. H. WREN.

Catalytic polymerisation of fatty oils. J. MARCUSON (Chem. Umschau, 1929, 36, 53—54).—Anhydrous tin tetrachloride was used as catalyst (cf. Staudinger and Bruson, B., 1926, 719) for the polymerisation of tung, linseed, and fish-liver oils. Solid polymerised products, insoluble in acetone, were obtained from tung, linseed, and cod-liver oils yielding thick oils from which the fatty acids, recovered by saponification, had mol. wts. of 590, 494, and 330, respectively. The polymerised fatty acids from tung oil could be separated into (1) solid resinous acids (acid value 186, saponif. value 199.5, iodine value 81) yielding sodium salts which were soluble in water but insoluble in alcohol; and (2) pale, unimolecular, semi-liquid acids soluble in light petroleum and forming sodium salts soluble in alcohol. The solid acids of (1) on being heated at 105° or on exposure to air became partially insoluble in organic solvents due to anhydride formation.

E. LEWKOWITSCH.

Negative autoxidation catalysts for fatty oils. V. RUTSCHKIN (Oil Fat Ind. Russia, 1928, No. 6, 27—30; Chem. Zentr., 1928, ii, 1504).—Heating and spoilage of moist oil seeds is prevented by 0.01% of β -naphthol, but not phenol. Addition of phenol, resorcinol, or β -naphthol (0.01%) to moistened seeds retards their growth.

A. A. ELDRIDGE.

Determination of the oil content of seeds without heating. N. SPASSKI (Oil Fat Ind. Russia, 1928, No. 6, 19; Chem. Zentr., 1928, ii, 1506).—By means of the author's apparatus an accuracy of 0.1% is attained.

A. A. ELDRIDGE.

Dependence of chemical composition of oils on climate. J. DAVIDSON (Chem. Umschau, 1929, 36, 64—67).—Ivanov's work ("Climate of the World and the Chemical Functions of Plants") is reviewed. A notably higher proportion of linolenic and linoleic acids is found in the oils from seeds (linseed, sunflower seed, etc.) that have ripened in cold climates (northern latitudes or high altitudes), which give oils of high iodine value, than in the oils from the same seed grown in hotter climates, which favour the production of oleic acid. The decrease in linolenic acid content may also be correlated with the increasing intensity of the light in low latitudes. (Cf. Eibner and Brosel, B., 1928, 761.)

E. LEWKOWITSCH.

See also A., Mar., 245, **Higher fatty acids** (MORGAN and HOLMES). 293 and 294, **Determination of higher unsaturated fatty acids** (ESCHER). 294, **Distillation of palmitic acid** (SHOYAMA). **Conversion of higher fatty acids into barium salts** (ESCHER). **Distillation of oleic acid** (OGATA). **Acids of high mol. wt. from arachis oil** (HOLDE and others). 312, **Cholesterol** (MONTIGNIE). **Colour reaction for ergosterol and its transformation products** (HÄUSSLER and BRAUCH-

LI). 337, **Micro-detection of glycerol** (ALBER). 359, **Vitamin content of irradiated olive oil** (RIGOBELLO). **Colour reaction for ergosterol** (ROSENHEIM).

Lipolysis of worked butter. GRATZ.—See XIX.

PATENTS.

Production and employment of cleansing, emulsifying, and wetting agents. CHEM. FABR. MILCH A.-G. (B.P. 275,267, 2.8.27. Ger., 2.8.26).—Neutral fats, fatty acids, naphthenic acids, etc. (e.g., technical oleic acid) are sulphonated with excess of chlorosulphonic acid especially in presence of aromatic hydrocarbons (e.g., benzene) to yield products with soap-like properties.

C. HOLLINS.

Treatment of tall oil. OEL- U. FETT-CHEMIE G.M.B.H., Assees. of A. SCHULTZE & Co. OELFABR. (B.P. 278,697, 26.9.27. Ger., 11.10.26).—Tall oil is subjected to esterification (e.g., by refluxing with alcohol and sulphuric acid) to such an extent that the fatty acids alone are esterified. The mixture is then treated with soda lye to neutralise the excess of mineral acid and the unattacked resinic acids: the esters are extracted with light petroleum and the fatty acids recovered by hydrolysis.

E. LEWKOWITSCH.

Oil from hops (B.P. 305,066).—See XVIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Detection of adulteration in oil of turpentine. M. H. BARRAUD (Ann. Falsif., 1929, 21, 5—18).—Oil of turpentine in France should fulfil the official definition adopted by the 4th Congress of Industrial Chemistry at Bordeaux, 1924. 200 c.c. of the sample are first fractionally distilled, and four fractions of 40 c.c. collected, the fifth consisting of the residue in the flask and a few drops from the end of the condenser. Values for d , $[\alpha]$, n_D^{20} , and acidity of the whole sample and of each of the five fractions are then determined. In the first fraction any non-terpene adulterant will be present (usually white spirit) and the normal adulterants (rosin oil and rosin) will be in the last. From variations in the differences between each of these values for the various fractions adulteration may be detected. Standard values for the turpentine oils and of an oil adulterated with white spirit are tabulated. 2% of white spirit may thus be detected. If oil of turpentine is mixed with glycerin the Christianson effect results, and the temperature of the colour change from indigo-blue to violet indicates the point where the refractive indices of turpentine and glycerin are the same in yellow light. 1—2 c.c. of glycerin are added to 10 c.c. of turpentine oil in a test tube in which a thermometer is fixed. The temperature of the change is lowered for a very pure oil and rises with the proportion of impurities in the last fraction, particularly with products of oxidation. It is necessary always to compare oils from the same type of pine and to standardise the temperature change with a sample of known purity. 50 samples of standard maritime pine oil gave the value 23—26°.

D. G. HEWER.

Adhesives and paint vehicles. E. STERN (Farben-Ztg., 1929, 34, 1312—1313).—The nomenclature adopted by Wagner and Kesselring (B., 1929, 218) in their communication on the subject of casein emulsions is

discussed; the author restates his conception of the static-colloid paint system and the dynamic-colloid processes of the paint film. S. S. WOOLF.

[Adhesives and paint vehicles.] H. WAGNER (Farben-Ztg., 1929, 34, 1313).—A reply to Stern (cf. preceding abstract). S. S. WOOLF.

Paint testing. H. WOLFF, W. TOELDT, and G. ZEIDLER (Farben-Ztg., 1929, 34, 1179—1181).—Paint testing should not be modified in the direction of increasing the accuracy of existing methods to a point where undue significance is given to quantitative differences that are not justified in practical tests, subject at the best to accidental influences. Records of paint failures should take the form of well-marked stages in the deterioration, recognisable with certainty. A sand-abrasion test and a test wherein the electrical conductivity of the whole surface of a paint film is examined are detailed. Five "degrees of failure" are indicated for each test. S. S. WOOLF.

Temperature-humidity control cabinet for testing paint, varnish, and lacquer films. G. G. SWARD (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1929, Circ. No. 345, 91—98).—Drying, hardness, elasticity, etc. tests on films are carried out in the laboratory of the above Association in a cabinet fitted with devices for controlling temperature and humidity, details of which are given. S. S. WOOLF.

Influence of titanium white on the fastness to light of coal-tar dyes. E. KEIDEL (Farben-Ztg., 1929, 34, 1242—1243).—Exposure tests indicate that coal-tar dyes bleach much more rapidly when mixed with titanium white than with zinc oxide, lithopone, or white lead. The results are very definite in water-paint vehicles, but similar tendencies are indicated in oil media. S. S. WOOLF.

Titanium white catalysis? H. WAGNER (Farben-Ztg., 1929, 34, 1243—1244; cf. preceding abstract).—The abnormal bleaching of coal-tar dyes in the presence of titanium white (which may be attributable to autoxidation and to the sulphuric acid content of titanium white) may be remedied by the addition of zinc oxide, but this in turn causes a slight tendency towards darkening. The preparation of a "neutral pigment" is proposed. S. S. WOOLF.

Rapid drying finishes for wood surfaces. F. W. HOPKINS (Amer. Paint & Varnish Manufs.' Assoc., Feb., 1929, Circ. No. 343, 61—64).—A brief survey of the scope of lacquers in the wood-finishing field. Types of failure of nitrocellulose lacquers are discussed, and the desirable qualities of a new type of material are indicated. S. S. WOOLF.

Systematic formulation of nitrocellulose lacquers. S. DE LANGE (Farben-Ztg., 1929, 34, 1245—1246).—The work of Hofmann and Reid (B., 1928, 648) is criticised. The extension of the method to three-dimensional graphs (i.e., triangular prism co-ordinates) is considered to afford much more significant information. Thus flexibility may be correlated with proportion of nitrocellulose, resin, and plasticiser in lacquers, or compatibility with quantity of ester, alcohol, and hydrocarbon in the solvent mixture. S. S. WOOLF.

Use of ultra-violet radiations in distinguishing between amber and its imitations. G. KOSTKA (Chem.-Ztg., 1929, 53, 117—118, 138—139).—Under the influence of ultra-violet radiation (440—280 μ) amber shows a yellowish-green to bluish-white fluorescence. Pressed amber (ambroid) behaves similarly, the fluorescence being rather less pronounced. The fluorescence of clouded amber differs somewhat from that of the clear variety. The difference is mainly one of degree only, and is due to structural differences and not to the presence of impurities. Phenol-formaldehyde condensation products absorb ultra-violet radiation, but are not excited to fluorescence. Urea-formaldehyde condensation products show a faint fluorescence, easily distinguishable from that of genuine amber. Urea products are transparent to ultra-violet radiation. The differing transparencies towards the radiation permit of a ready distinction between urea and phenol condensation products. The easily-recognisable casein and cellulose derivatives fluoresce under the influence of ultra-violet radiation. J. S. CARTER.

Polyhydric alcohol-polybasic acid reaction. I. Glycerol-phthalic anhydride. R. H. KIENLE and A. G. HOVEY (J. Amer. Chem. Soc., 1929, 51, 509—519).—The isothermal resinification of a mixture of glycerol (2 mols.) and phthalic anhydride (3 mols.) at 195—230° involves esterification only, since water alone is evolved and the saponification value of the mixture remains nearly constant. The reaction proceeds rapidly at first with heat evolution and a corresponding rapid decrease in acid value and increase in ester value of the product, followed by a more gradual change until gelation takes place. The flow-point, colour, and consistency of the product show similar changes. The density increases, but there is little change in the refractive index. Esterification is about 50% complete during the first minute, 75—79% at gelation, and 99% on prolonged heating. The time of gelation is inversely proportional to the absolute temperature. There is no indication that the reaction proceeds in stages.

H. E. F. NOTTON.

PATENTS.

Nitrocellulose solutions. W. J. JENKINS and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 305,148, 26.8.27).—The flash point of nitrocellulose solutions containing petrol or benzene hydrocarbons, e.g., clear lacquers or enamels, may be raised by addition of not more than 8% by wt. of carbon tetrachloride.

F. R. ENNOS.

Cellulose ester or ether compositions. E. I. DU PONT DE NEMOURS & Co. (B.P. 279,520, 25.10.27, U.S., 25.10.26).—Tetrahydrofurfuryl alcohol is claimed as a desirable ingredient of brushing lacquers.

S. S. WOOLF.

Coating of articles [insulated wire]. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 305,084, 28.10.27).—A liquid insulating coating is applied to wire etc. and hardened by heat solely obtained from the combustion of its own volatile constituents. The liquid coating material is contained in a suitable sealed chamber through which the wire may be drawn continuously at high speed. Suitable apparatus is described. S. S. WOOLF.

Manufacture of coating compositions [chlorinated rubber varnishes]. C. ELLIS, ASSR. to CHADELOID CHEM. CO. (U.S.P. 1,695,638—1,695,642, 18.12.28. Appl., [A] 29.8.21, [B] 5.5.23, [C—E] 22.3.24. Renewed, [B] 30.4.25, [E] 10.9.27).—(A) A varnish consists of chlorinated rubber, a volatile solvent or thinner, and a drying oil, *e.g.*, tung oil. (B) A straw-hat varnish consists of chlorinated rubber (over 66% Cl) and a colouring matter dissolved in a volatile solvent. (C) In order to give a dull finish, the varnish contains less drying oil, *e.g.*, linseed oil, than chlorinated rubber. (D) A solution of chlorinated rubber (67% Cl or more) is stabilised by the addition of a soluble organic amino-compound. (E) A varnish reviver consists of chlorinated gutta-percha, an aromatic solvent therefor, and a resinous homogenising agent; a phenolic phosphate may also be present. F. G. CLARKE.

Cement [from chlorinated rubber]. C. ELLIS, ASSR. to CHADELOID CHEM. CO. (U.S.P. 1,695,637, 18.12.28. Appl., 22.3.24).—An adhesive consists of a solution of chlorinated rubber. F. G. CLARKE.

Phenol resin and its manufacture. BAKELITE CORP. (B.P. 280,521, 27.10.27. U.S., 11.11.26).—A phenol is condensed with anhydroformaldehydeaniline (or a homologue) and aniline excess is removed. According to the temperature employed a reactive or non-reactive (*i.e.*, non-hardening) resin is obtained; the latter may be made reactive by addition of hexamethylenetetramine, or this reagent may be added initially. C. HOLLINS.

Artificial resin compositions. BAKELITE CORP., Assees. of J. H. SCHMIDT (B.P. 275,604, 26.7.27. U.S., 3.8.26).—A high-boiling solvent (b.p. above 170°), *e.g.*, ethyl oxalate, glycol diacetate, etc., is added to a "glyptal" resin that has previously been taken to the stage of incipient gelatinisation in the substantial absence of solvent, and the "curing" is continued in the presence of the incorporated solvent, until gelatinisation once more sets in, when medium-boiling solvent (b.p. 110—160°) may be added. S. S. WOOLF.

Production of a viscous condensation product. SCHIEFERWERKE AUSDAUER A.-G. (B.P. 286,731, 9.3.28. Ger., 10.3.27).—A mixture of phenol, *p*-dichlorobenzene, and formaldehyde is heated to boiling in the presence as catalyst of a salt mixture composed of sodium chloride, ammonium chloride, and hexamethylenetetramine, the water formed is removed, and volatile vapours are expelled from the product, which is then washed with water. The light-coloured syrup thus obtained may be readily hardened at 80°. S. S. WOOLF.

Production of highly glazed surfaces. R. ARNOT, Assec. of ELEKTROCHEM. FABR. KEMPEN-RHEIN, DR. BRANDENBURG & WEYLAND G.M.B.H. (B.P. 288,303, 4.4.28. Ger., 6.4.27).—A film of hardened artificial resin of highly polished surface is applied under pressure at normal or elevated temperatures to articles previously covered with glue solution etc. S. S. WOOLF.

Methods of enamelling and apparatus therefor. W. E. BEATTY, From BELL TELEPHONE LABS. INC. (B.P. 305,611, 8.11.27).

Green hydrated chromium oxide (B.P. 304,809).—See VII. Resins from hops (B.P. 305,066).—See XVIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Report of the Raw Rubber Specifications Committee. E. B. SPEAR, C. R. BOGGS, H. E. SIMMONS, H. L. TRUMBULL, and N. A. SHEPARD (Ind. Eng. Chem. [Anal.], 1929, 1, 55—56).—As the result of tests in five laboratories using a rubber-sulphur mixing and one containing also zinc oxide and di-*o*-tolylguanidine, the conclusion is drawn that the physical testing of raw rubber is not in a very satisfactory condition, and further investigations are recommended with a view of securing more definite standardisation and greater uniformity. D. F. TWISS.

Cold vulcanisation [of rubber] (Parkes' process). P. BOURGOIS (Bull. Féd. Ind. Chim. Belg., 1929, 8, 3—8).—The method of vulcanisation of rubber by immersion in a solution of sulphur chloride is reviewed, with especial reference to certain practical details and difficulties. The vulcanising effect is attributed to nascent sulphur formed from the sulphur chloride by the action of natural moisture in the rubber. D. F. TWISS.

Vulcanisation and structure of rubber. E. LINDMAYER (Kautschuk, 1929, 5, 35—38).—Vulcanisation of rubber is regarded as comprising a primary stage producing soft rubber and a secondary stage leading to vulcanite; these frequently overlap in practice, but with care can be distinguished. With the aid of the "needle structure" for rubber it is possible to explain the mechanism of vulcanisation and to illustrate it by means of three-dimensional formulæ. D. F. TWISS.

The accelerator [of vulcanisation] "Tuads" and its substitutes. F. LÖBLEIN (Kautschuk, 1929, 5, 38—43; cf. Esch. B., 1929, 28).—Contrary to the earlier statement, "Tuads" and "Vulkacit Thiuram" (commercial forms of tetramethylthiuram disulphide) are of comparable quality—the latter, indeed, being somewhat purer. Experimental evidence is given. D. F. TWISS.

Production of lustrous spread rubbered material with the aid of vulcanisation by ultra-violet light. R. DITMAR (Gummi-Ztg., 1929, 43, 1172).—The material is spread in the usual way with a benzene solution of a mixture containing crêpe rubber (100 pts.), colloidal sulphur (3.5 pts.), tetramethylthiuram disulphide (2 pts.), sulzin (ZnSO₄·5NH₃, 7 pts.), anhydrous sodium thiosulphate (2 pts.), selenium-red (1.8 pts.), and titanium oxide (2 pts.). It is then led directly under a battery of quartz mercury lamps, the exposure being for 5 min. at a distance of 8 cm.; complete vulcanisation results with production of a non-tacky rubbered surface of high lustre. D. F. TWISS.

New physical test for vulcanised rubber. D. D. WRIGHT (Ind. Eng. Chem. [Anal.], 1929, 1, 17—20).—The test-piece is a strip of rubber which has been trisected for a considerable portion of its length; tension is then applied between the uncut end and the end of the central tongue until rupture occurs. This test subjects the sample to a combination of tensile and shearing stresses,

the latter predominating. It is frequently a more sensitive index than the ordinary stress-strain test for over-vulcanisation or depreciation by accelerated ageing.

D. F. TWISS.

See also A., Mar., 305, **Polystyrene** (STAUDINGER and others). 305—6, **Highly polymerised compounds** (STAUDINGER and others). 321, **Isoprene and caoutchouc** (STAUDINGER and others).

PATENTS.

Vulcanisation of rubber. Production of accelerators for vulcanising rubber. NAUGATUCK CHEM. CO., Assecs. of S. M. CADWELL (B.P. 279,815 and 298,537, [A, B] 5.10.27. U.S., 1.11.26).—(A) Vulcanisation is accelerated by the chlorination derivative of a condensation product of an amine and an aldehyde containing an open chain of 2—7 carbon atoms, such as heptaldehyde-aniline. The reaction products with 1—3 chlorine atoms accelerate well; a pentachloro-product is also obtainable which is not an accelerator, but by hydrolysis with aqueous alkali is converted into a very active tetrachloro-compound. Bromine and iodine derivatives can be used similarly. (B) Heptaldehyde-aniline and other condensation products and amines with an aldehyde containing an open chain of several carbon atoms are treated with a halogen or a halogen compound such as hydrogen chloride for the production of new vulcanisation accelerators. D. F. TWISS.

Reclamation of rubber. DUNLOP RUBBER CO., LTD., D. F. TWISS, and F. THOMAS (B.P. 305,826, 17.2.28).—Scrap vulcanised rubber is treated with a solution of caustic alkali in a polyhydroxy-compound such as glycerol, *e.g.*, at 190—200°. D. F. TWISS.

Production of rubber articles from natural rubber latex. ANODE RUBBER CO., LTD. (B.P. 283,871, 23.12.27. Ger., 18.1.27).—In order to modify the properties of articles made directly from rubber latex, the latter is previously mixed with an artificial aqueous dispersion of masticated rubber. D. F. TWISS.

Bonding of articles or parts formed respectively of india-rubber or the like and cellulose derivatives. C. MACINTOSH & CO., LTD., S. A. BRAZIER, and G. F. THOMPSON (B.P. 305,745, 1.12.27).—The india-rubber surface is first treated with a mutual solvent for rubber and cellulose derivatives, *e.g.*, cyclohexanol. A varnish or lacquer of cellulose acetate or nitrate, into which a mutual solvent may have been introduced, is then applied to the softened surface. An increased strength of adhesion is thus ensured. D. F. TWISS.

Treatment of fabric with rubber solution or the like. C. MACINTOSH & CO., LTD., S. A. BRAZIER, and J. D. CAMPBELL (B.P. 305,264, 4.11.27).—Rubber in solution or aqueous dispersion is applied to fabric, the treated material being transported in festoons through a drying chamber; the material may pass progressively through another application of rubber and a drying process, and finally to a vulcanising chamber. D. F. TWISS.

Chemical product [chlorinated rubber] and its manufacture. C. ELLIS, ASSR. to CHADELOID CHEM. CO. (U.S.P. 1,695,636, 18.12.28. Appl., 17.6.22).—Solid rubber, plasticised with not more than 20% of benzene

or other solvent, is chlorinated under pressure at, *e.g.*, 20—30°. R. BRIGHTMAN.

Production of an elastic composition. W. T. GALBRAITH and A. E. MILLS (B.P. 305,249, 29.2.28).—A mixture of paper pulp (50 lb.) or its equivalent of rubber (synthetic, natural, or reclaimed), heated vegetable oil (50 lb.), "synothin" (5 lb.), gluten (20 lb.), water-glass (3 quarts), glue (20 lb.), gum (40 lb.), casein (2 lb.), and asbestos (5 lb.) is immersed for a few seconds in sulphuric acid, diluted with water or paraffin, washed repeatedly with dilute ammonia, digested with alcohol and sal-ammoniac, and, after the addition of 2% of copper oxide, dried by treatment with superheated air for 5 hrs. Any tendency to perish is overcome by treating with sulphur fumes and shellac. The elasticity is increased by incorporating tannic acid, the sheeted material being finally immersed in aniline water. The dried product is elastic and fire-proof.

D. F. TWISS.

Manufacture of rubber articles. W. B. WESCOTT, ASSR. to RUBBER LATEX RES. CORP. (U.S.P. 1,702,225, 12.2.29. Appl., 1.6.26).—See B.P. 272,187; B., 1928, 101.

Mixing machines for rubber compositions etc. F. H. BANBURY (B.P. 306,371, 1.9.28. U.S., 3.3.28)

Manufacture of articles [rubber tubes] containing material at one stage plastic, and particularly extrusion of the plastic content. DUNLOP RUBBER CO., LTD., and F. FELLOWES (B.P. 305,152, 31.10.27).

[Embossed] rubber goods and their manufacture. BROADHURST & CO., LTD., and W. J. DEXTER (B.P. 306,276, 11.2.28).

Coating compositions (U.S.P. 1,695,637—1,695,642).—See XIII.

XV.—LEATHER; GLUE.

Removal of chromium salts from leather. F. C. HEMMINGS and M. C. LAMB (J. Soc. Leather Trades' Chem., 1928, 12, 599—602).—The chromium compounds are removed from wet or dry chrome-leather shavings most rapidly by solutions of oxalic acid, which is more effective than ammonium oxalate alone or with acid or ammonia. Dry shavings are stripped of their chromium more readily than wet shavings. The liquor obtained is acid to litmus, but the chromium is present in an anionic form. 15% of oxalic acid on the weight of the dry shavings is necessary to remove the chromium salts, and the stripped product should be washed with dilute ammonia because ammonium chromoxalate is more soluble than chromoxalic acid. Oxalic acid is also useful for "wetting back" dry chrome leather.

D. WOODROFFE.

Effect of heat on wetted vegetable-tanned leathers. II. W. J. CHATERS (J. Soc. Leather Trades' Chem., 1928, 12, 608—614; cf. B., 1929, 257).—Shrinkage curves have been derived in the author's apparatus for wet-salted calf pelt after treatment with water, acid and alkaline solutions, respectively, and for oak bark-tanned leathers containing different amounts of free mineral acid. The irregularities in the curves are indicative of the physical and chemical conditions in the leathers, and could be used for control purposes in manufacture.

D. WOODROFFE.

Determination of the strength of a bate. A. BORDIN (J. Soc. Leather Trades' Chem., 1928, 12, 614—626).—Experiments have been made on casein substrates using two bacterial bates and two pancreatic bates. Complete precipitation of casein is obtained only at p_H 5. The use of buffer salts to dissolve casein diminishes the hydrolytic power of the enzymes. In the absence of buffer salts, the methods of weighing the casein and of determining the soluble nitrogen yield identical results for the casein digested. The casein is sufficiently buffered when dissolved in a small quantity of sodium hydroxide to prevent variations >0.2 in the p_H value during the digestion. If buffer salts are present, the "soluble nitrogen" method yields more accurate results for the amount of digested casein than are obtained by weighing the unchanged casein, since the latter will absorb salts when precipitated. Pancreatic enzymes and bates are more reactive the nearer the p_H value is to 8, but bacterial products are reactive over a wider range of p_H values. The results are not affected by dissolving the casein at 40° or on a boiling water-bath. The soluble nitrogenous matter is a constant for any particular casein, but varies with different products. The digestion of the casein is not proportional to the time of digestion; 1 hr. is the best period at 40° , and $35-40^\circ$ is the best temperature range. The unchanged casein cannot be precipitated if more than 84% of the total casein originally present has been hydrolysed.

D. WOODROFFE.

Specifications for standard gelatin. Report of Committee on Standard Gelatin appointed by Division of Leather and Gelatin Chemistry. C. E. DAVIS, S. E. SHEPPARD, and M. BRIEFER (Ind. Eng. Chem. [Anal.], 1929, 1, 56).—For standard gelatin for physico-chemical work the following features are tentatively specified:—calfskin gelatin from green stock extracted at 54.4° and dried at not less than 5% concentration; ash, max. 0.05%; constant isoelectric point; definite viscosity and jelly strength under standardised conditions; clear, colourless, and free from preservatives; fat, probable max. 0.1%; heat-coagulable protein probable max. 0.2%; arsenic, max. 0.5 p.p.m. (as trioxide); zinc, max. 20 p.p.m.; copper, max. 10 p.p.m.; other heavy metals, max. 50 p.p.m.

D. F. TWISS.

See also A., Mar., 353, **Specific action of proteolytic enzymes** (WALDSCHMIDT-LEITZ).

PATENTS.

Tanning of hides and skins. E. G. STIASNY and B. JALOWZER (B.P. 305,562, 7.11.27).—The materials are tanned with, or treated prior to or after treatment with other materials with, a compound of iron and (a) an aliphatic organic acid containing more than one carbon atom in its molecule, e.g., ferric lactate, or (b) a sulphonic acid of an aromatic hydrocarbon, e.g., naphthalene, anthracene, or mixtures forming these. Water-soluble carbohydrates, e.g., sulphite-cellulose extract, may be added to the solution of the iron compound.

D. WOODROFFE.

Employment of highly sulphonated Turkey-red oils in tanning. M. FLESCH (B.P. 282,710, 4.8.27. Ger., 27.12.26).—An acid-resisting, highly sulphonated Tur-

key-red oil, containing over 6% of organically combined sulphuric acid, is used, either alone or with organic acids, in the pretreatment and first stages of tanning of hides and skins.

D. WOODROFFE.

Hydrolysis of casein or casein-containing bodies. R. ARNOT (B.P. 306,168, 12.11.27).—Casein or material containing it, e.g., horse-chestnuts, soya beans, lupin seeds, etc., is treated under pressure and at 135° or below with water in the presence of alkali carbonates, alkaline-earth or magnesium hydroxides, or organic bases. The products may be used for the preparation of foodstuffs or of adhesives.

L. A. COLES.

XVI.—AGRICULTURE.

Soil-sampling tube. E. J. VEIHMAYER (Soil Sci., 1929, 27, 147—152).—The tube is characterised by a detachable cutting point shaped to minimise compression of the soil beneath it and of the core sample within. A specially constructed head withstands hammering and allows of easy withdrawal. Nearly dry sand and gravelly soils may be sampled without difficulty.

A. G. POLLARD.

Measurement of "suction forces" in soil. C. F. SHAW (Soil Sci., 1929, 27, 111—115).—Apparatus involving the suction of water by soil through porous tubes etc. cannot give true values for the "suction forces" of soils. The porous materials themselves have high "suction forces," and soil enclosed within them does not exert any greater suction than in taking up water from a free surface.

A. G. POLLARD.

Usefulness of capillary potential to soil-moisture and plant investigators. L. A. RICHARDS (J. Agric. Res., 1928, 37, 719—742).—Capillary potential in soil is explained and utilised as a basis of a general consideration of soil-water relationships. The vertical and horizontal flow of water in soil can be mathematically expressed in a manner analogous to that for the flow of heat and electricity, viz., as capillary conductivity \times potential gradient. Experimental results illustrating the application of the potential theory are recorded, and apparatus for the determination of the factors concerned is described. The availability of the soil water to plants depends on the tenacity with which the water is held by the soil and the ease of its transmission towards the plant root. The capillary potential is a direct measure of the first factor. A porous clay apparatus for the control of soil moisture in pot experiments is described.

A. G. POLLARD.

Determination of the fineness of marl. E. P. DEATRICK and C. DORMAN (Soil Sci., 1929, 27, 121—122).—Current methods for the examination of marl involve screening the dried but not deflocculated material. More satisfactory results are obtained by passing an alkaline aqueous suspension of marl through a battery of fine sieves.

A. G. POLLARD.

Condition of formation and constitution of the humic-clay complex of soils. A. DEMOLON and G. BARBIER (Compt. rend., 1929, 188, 654—656).—The fixation of humic acid by brick clay decreases as the p_H value of the medium increases. Experiments on the influence of the concentrations of humate and calcium

ions indicate that colloidal clay is a factor in the fixation of humic colloids in soils, the properties of the complex depending on the cations adsorbed by the former.

J. GRANT.

Colorimetric determination of nitrates in soils and waters. L. U. DE NARDO (Compt. rend., 1929, 188, 563—565).—A 2.5% solution of pyrogallolsulphonic acid (0.5 c.c.) is added to 10 c.c. of a solution containing 0.0005—0.1 mg. of nitrate (as KNO_3) and 20 c.c. of sulphuric acid, and the brown-red to olive-green coloration matched after 1 hr. against that produced with a standard nitrate solution. For greater amounts 0.5 c.c. of a 2.5% solution of pyrogallol preserved with 0.1% of sodium bisulphite is added to 5 c.c. of solution and 25 c.c. of sulphuric acid. Ferric salts and iodides give a violet colour, chromates yellow, and organic matter brown, and extracts of soils should therefore be treated in succession with solutions of baryta (saturated and boiling) and basic lead acetate (50%), the excess of which is removed by means of a saturated solution of sodium sulphate, and the solution filtered. Nitrites, which react as nitrates, are eliminated by shaking with 1 c.c. of sulphuric acid and a drop of a concentrated solution of urea for 10 min.

J. GRANT.

Influence of manganiferous soils on the accuracy of the quinhydrone electrode. W. T. McGEORGE (Soil Sci., 1929, 27, 83—88).—The quinhydrone electrode gives abnormally high p_H values in soils containing appreciable quantities of manganese. The potentiometer drift during the experiment is sufficient indication of the doubtful nature of the results.

Significance of hydrogen-ion concentration in soil nitrification studies. H. HUMFELD and L. W. ERDMAN (Proc. Iowa Acad. Sci., 1927, 34, 63—66).—Nitrification proceeds when p_H 4.4—4.8 is reached; thereafter it is slow. Whenever the final p_H is below 4.2 the amount of ammonium sulphate nitrified is small. Nitrification is considerably increased by addition of calcium carbonate. The final reaction of the soil is somewhat more acid than the initial reaction.

CHEMICAL ABSTRACTS.

Determination of the nutrient value of soils by pot experiments. H. WIESSMANN (Z. Pflanz. Düng., 1929, 8B, 76—89).—The criticisms of Maiwald (B., 1928, 764) are replied to and details of the author's methods explained.

A. G. POLLARD.

Application of nitrogenous fertilisers. O. ENGELS (Z. Pflanz. Düng., 1929, 8B, 49—61).—An explanatory survey of the usually accepted tenets of nitrogenous fertilisation is given. The use of chemical fertilisers is discussed from the point of view of their effects on soil reaction, physical properties, and cropping.

A. G. POLLARD.

Effects of different nitrogenous fertilisers. GERLACH (Z. Pflanz. Düng., 1929, 8B, 89—91).—Average figures from numerous field trials indicate calcium nitrate to be as efficient as Chile saltpetre and superior to ammonium sulphate. No increase in crop yields is attributable to the iodine in Chile saltpetre.

A. G. POLLARD.

Influence of organic matter and lime on soil

moisture and on the percentage of carbon and nitrogen in field soils. J. F. MULLER (Soil Sci., 1929, 27, 137—141).—Generally speaking, soils treated annually with organic manures had higher moisture contents than when minerals alone were used. Liming decreased the moisture content of the soils to an extent approximating to one half the increase resulting from the use of dung. Both the nitrogen and carbon contents of soils treated with organic manure increased during the period of examination. In all other cases the proportions of these constituents decreased.

A. G. POLLARD.

Physico-chemical effects of organic soil colloids. W. F. LOEWING (Proc. Iowa Acad. Sci., 1927, 34, 149—152).—Liming of certain acid soils is injurious; decreased potash assimilation is associated with the change from acidity to alkalinity or with release of some toxic substance, e.g., iron. Administration of potash following lime injury does not restore the yield on organic soils.

CHEMICAL ABSTRACTS.

Effect of crop growth on replaceable bases in some Californian soils. J. C. MARTIN (Soil Sci., 1929, 27, 123—136).—Variations in the amounts of replaceable bases in soils lie within narrower limits among soils of similar classification than among soils of different origin. Clays and silty clay loams have much larger proportions of replaceable bases than sandy loams. Soils cropped annually for a lengthy period showed no marked difference in the content of replaceable bases from those cropped twice only with a long fallow interval. Continuous cropping decreased the proportion of replaceable potassium in soils without significant change in the total bases present, since the replaceable calcium and magnesium, comprising 90% of the total, remained unaltered. The replaceable potassium removed by twelve annual barley crops represented 32% of the amount originally present and 82% of the total potassium contained in the crops. Replaceable bases in soil as measured by extraction with 0.05N-acid are appreciably greater than when ammonium acetate extracts are utilised. In the latter case results agree more closely with the amounts of ammonia adsorbed by the soil. Acid extraction probably results in some decomposition of the adsorbing complex.

Effect of sodium chloride and carbonate on the growth of asparagus. B. L. HARTWELL, J. B. SMITH, and L. C. DAMON (Rhode Is. Agric. Exp. Sta. Bull., 1928, No. 213, 16 pp.).—Potassium chloride or alkali carbonate decreased, whilst sodium chloride increased, soil acidity. Heavy rainfall over two months reduced active acidity and active alumina. Omission of sodium decreased the yield; in plots deficient in potassium, sodium chloride and carbonate increased the yield.

CHEMICAL ABSTRACTS.

See also A., Mar., 262, **Dual emulsions for spraying of trees** (WOODMAN). 289, **Geology and mineralogy of soils** (HART). 337, **Determination of iodine in soil** (SCHWAIBOLD). 360, **Forcing of plants by warm baths** (BORESCH). **Influence of radiations on germination of seeds and growth of plants** (MEZZADROLI and VARETON). 362, **Chlorosis of fruit trees** (WALLACE).

PATENTS.

Manufacture of fertilisers. R. E. SLADE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 305,760, 13.12.27).—A mixture of, e.g., 90% ammonium nitrate solution containing 43 pts. of ammonium nitrate with 57 pts. of calcium carbonate obtained as a by-product in the manufacture of ammonium sulphate from ammonium carbonate and calcium sulphate, is evaporated to dryness by heating at 50–100° with constant stirring in a vacuum evaporator. When dry, the mixture is sieved and oversize particles and fines are mixed with a subsequent charge. The product contains 15% N. L. A. COLES.

Cold frames and like transparent roofings for use especially in horticulture. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,242, 2.1.28).—Sheets of transparent material comprise cellulose ethers, urea-formaldehyde condensation products, etc. strengthened by an embedded network. L. A. COLES.

Caking of crystals (B.P. 305,721).—See I. Fertiliser salts (B.P. 304,872).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Analysis of maple products. X. **Modification of the Canadian lead method.** XI. **Composition of the Canadian lead precipitate.** D. E. FOWLER and J. F. SNELL. (Ind. Eng. Chem. [Anal.], 1929, 1, 8–13).—X. In order to eliminate errors due chiefly to formation of fissures in the precipitate during washing, the following method is proposed. The prepared syrup containing 25 g. of dry matter is weighed, made up to 100 c.c. at 20°, and 20 c.c. are pipetted into a large test tube; to this 2 c.c. of basic lead acetate solution are added, the tube is corked, and, after 2 hrs., the contents are filtered with suction through an asbestos mat at least 3 mm. thick. Four washings with cold water are made, care being taken to keep the precipitate covered and that filtration is not quicker than 40 drops per min. The precipitate is dried at 100° and weighed. Somewhat higher results are thereby obtained.

XI. Analysis of the lead precipitates from three maple syrups showed that the total lead content is greater (72.64–67.58%) and the basic lead content slightly higher the lower are the Canadian lead values. The difference between total and basic lead exceeds considerably the lead equivalent of the acids freed by hydrogen sulphide, and 97% of these latter acids was malic acid as determined polarimetrically in a composite sample of more than 20 syrups. Substances more carbonaceous than lead malate are present in the lead precipitate since they contained 11.1–12.2% C and 1.46–1.15% H. Flavouring and colouring matters appeared to be precipitated. D. G. HEWER.

Determination of moisture in sugar syrups. E. W. RICE (Ind. Eng. Chem. [Anal.], 1929, 1, 31–32).—Half of a 10 g. roughly weighed portion of dried Filter-Cel is put in a flask followed by 15 g. of the syrup (weighed in a capsule of thin waxed paper), and the rest of the Filter-Cel is added. Distillation is then carried out as described by Bidwell and Sterling (B., 1925, 268). The whole operation occupies only 4 hrs.,

no blackening or decomposition of the residue occurs, and the same rate of distillation of the toluene can be maintained with the oil bath at 127° as at 143° without Filter-Cel. D. G. HEWER.

Determination of the adhesive power of starch by Saare's method. W. EKHARD (Z. Spiritusind., 1929, 52, 70–71).—A metal plate 22 mm. in diam., having a rod attached perpendicularly to its centre, is suspended 3 cm. below the surface of 200 g. of a carefully prepared solution, containing 9 g. of starch, by means of a small transverse rod which rests in a slot in an adjustable metal bridge fixed to the rim of the 250 c.c. beaker. After allowing the starch solution to set for 24 hrs., the vertical rod is connected by means of a hook at the end to one beam of a balance, and sand is gradually added through a funnel to the pan attached to the other beam until the metal plate is pulled out of the starch paste. The weight of sand required is taken as a measure of the adhesive power of the starch.

F. R. ENNOS.

Quantitative separation of dextrins and gum arabic. A. HAMY (Ann. Falsif., 1929, 21, 24–26).—To 20 c.c. of the gum arabic syrup, previously diluted to twice its volume, are added 23 c.c. of 95% alcohol and the solution is made up to 55 c.c. with water; after a few hours the insoluble dextrins are deposited, no gum being precipitated in alcohol of this concentration. 50 c.c. of the solution are filtered and 90 c.c. of water containing 5 g. of dextrose and 8 g. of sucrose are added, followed by 42 c.c. of 95% alcohol and 15 c.c. of lead subacetate, and, after shaking, is left to settle overnight. The supernatant liquid is decanted, and the residue centrifuged in 2 tubes, the precipitate and tubes being washed with 50 c.c. of 50% lead acetate. The precipitate in each tube is mixed with 4.3 c.c. of a 75% sugar solution, which effects partial dissolution, and 35 c.c. of a mixture of 120 c.c. of 95% alcohol, 27 c.c. of lead subacetate, and 160 c.c. of water are added. The precipitate, after being centrifuged, is dissolved in 4% acetic acid, and the gum precipitated by alcohol. When precipitation is complete, the gum is filtered, dried at 110°, and weighed, then calcined and weighed again. D. G. HEWER.

See also A., Mar., 267, **Saturation of sugar-lime solutions** (ATEN and others). 272, **Mutarotation of galactose** (LOWRY and SMITH). 297, **Precipitation of carbohydrates by alkaloids** (ROSENTHALER). 298, **Colorimetric determination of dextrose** (SCHACKELDIAN). **Distillation of sucrose** (HIDAKA). 299, **Starch acetate** (BRIGL and SCHINLE). **Disruption of maize starch granules** (TAYLOR and BECKMANN). 341, **Colour reactions of the carbohydrates** (DISCHE). 352, **Enzymic fission of sucrose** (WEIDENHAGEN). 354, **Conversion of dihydroxyacetone into hexose by fermentation** (NEUBERG and KOBEL; IWASAKI). 360, **Nature of waxy starch of maize** (BRINX).

XVIII.—FERMENTATION INDUSTRIES.

Maintenance and regeneration of warm-stored bottom- and top-fermentation yeasts. F. WINDISCH (Woch. Brau., 1929, 46, 71–74, 81–86).—Yeasts the reproductive and fermentative powers of which had been weakened by storage under beer for three days

at 18–20° maintained their weakened character without further deterioration when similarly stored between four successive fermentations. The lower reproduction and fermentation rates as compared with yeasts stored at 2–6° are not due to the slightly higher proportion of dead cells. After four warm-storage periods the yeasts regained their original reproductive and fermentative activity when kept for 3 days under beer at 2–6°.

F. E. DAY.

Drying of hops in vacuo. K. BEHRENDT (Woch. Brau., 1929, 46, 86–87).—Four samples of hops were dried under partial vacuum (100 mm.) in a current of partly dried air. The air entered the drying vessel at 50–52° and left at 20–25° at the start, rising to 30–36° at the end of drying, which occupied 5–8 hrs. The hop moisture was reduced from 64.4–70.2% to 6.13–14.85%. The dried samples were compared as regards humulone and resin content with portions of the same samples dried on trays in air for 2–3 days, and with samples from the kiln-dried bulks. In all cases the vacuum- and air-dried samples contained similar amounts of humulone, which in three cases exceeded that in the kiln-dried material by 3–30%. The exceptional sample had been damaged in transit, and the results indicate that vacuum-drying causes as little harm as air-drying and less than kilning.

F. E. DAY.

Importance of hydrogen-ion concentration for the distillery. II. W. DIEMAIR and K. SIEBERT (Biochem. Z., 1929, 204, 414–430; cf. B., 1928, 797).—During the whole period of fermentation maize mashers differ considerably as regards acidity and state of combination of the nitrogen from potato mashers, the latter having a lower p_H maximum and producing more titratable acid than the former. The difference in amount of titratable acid is due to alterations in the composition of the yeast which, in the maize mashers, cannot obtain sufficient nitrogen for growth and so decomposes, leaving much protein and protein decomposition products in the substrate. Hence, in this case, the buffering power is at a maximum. No relation could be found between the forms of the curves of acidity and buffering power. The results of fermentation are best when the maxima of the two curves do not coincide. W. MCCARTNEY.

Estimating heat consumed in batch rectification of alcohol. G. S. HOUGHLAND (Chem. & Met. Eng., 1929, 36, 89–91).—A graphical method is described for determining the average theoretical heat consumption during intermittent rectification, and is applied to the concentration of ethyl alcohol from aqueous solutions of 1–20%, producing distillate containing 92.46% of alcohol and exhausting the residue to 0.1% of alcohol, in columns containing 9–16 perfect plates. The composition of the residue in the kettle and of the vapours arising therefrom being known, the theoretical heat quantities evolved at the instant when a given weight of distillate has passed over may be calculated, and these values may be plotted against the weight of distillate to give a curve the area under which represents the desired total heat consumption. H. S. GARLICK.

See also A., Mar., 352, Separation of enzymes of barley malt (PRINGSHEIM and THILO). Action of light on takadiastase in presence of sensitizers (KAM-

BAYASHI). Enzymic fission of sucrose (WEIDENHAGEN). Liberation of invertase from yeast (WILLSTÄTTER and GRASSMANN). 353, Specific action of proteolytic enzymes (WALDSCHMIDT-LEITZ). Specific action of erepsin, trypsin, and trypsin-kinase (ABDERHALDEN and others). 354, Conversion of dihydroxyacetone into hexose (NEUBERG and KOBEL; IWASAKI). 355, Lactic acid fermentation (VIRTANEN).

Power alcohol. THAYSEN and GALLOWAY.—See II.

PATENT.

Extraction of oils and resins from hops. **Drying of hops.** L. WEBER (B.P. 305,066 and 305,076, 27.10.27).—(A) Hops are drawn by means of a vacuum into a cylindrical still with a vertical axis furnished with rakes and so arranged that the hops are confined by two perforated plates between top and bottom dome-shaped jacketed covers. After drying the hops by the method described under (B), steam is passed under reduced pressure through the hops and the volatile oils are recovered from the condensed vapour. When the hops have again been dried as before, concentrated alcohol, preferably 96%, is circulated through the hops to extract the resins. When extraction is complete, the alcoholic solution is concentrated and run off, and the alcohol and resins are separated by distillation under as high a vacuum as possible. Following the passage of warm air which removes and permits the recovery of the alcohol remaining in the hops, warm water is circulated through the hops to extract the remainder of the resins, tannins, etc. (B) Using the still charged with hops as in (A), air or alcoholised air at 21–32° is drawn in through an inlet in the bottom cover of the still and allowed to emerge by an outlet in the top cover, the stirring gear being maintained in motion during the process. The dried hops are removed from the still by suction and baled for the market. The oil and vapour collected during the passage of the warm air are condensed and collected.

C. RANKEN.

XIX.—FOODS.

Improvement of flour. M. VUK and P. SPANYÁR (Z. Unters. Lebensm., 1928, 56, 292–308).—The effects on the baking qualities of flour of addition of wheat germ, sugar, dextrin, potassium bromate, ammonium persulphate, sodium borate, "novadelox," and of treatment with nitrogen peroxide and "gologas" (chlorine containing 0.5% of nitrosyl chloride) were studied. Wheat germ improves the baking qualities, but cannot be used owing to its effect on colour and taste of the bread. Sugar or, to a smaller extent, dextrin is beneficial, but addition of the above salts gave no definite improvement. Novadelox resulted in improved texture and bleaching, nitrogen peroxide in bleaching only, but gologas had a very favourable effect especially on bread volume and texture. Gologas is a flour improver rather than a maturing agent, as flour matured by ageing can be further improved thereby. Its action appears to be due to the production of acidic protein substances in the flour. W. J. BOYD.

Semi-microchemical method for the determination of gluten in flour. A. C. RÖTTINGER (Mikrochem.,

1929, 7, 106—109).—The flour (1 g.) is moistened with two drops of water in a bag of suitable material and, after 30 min., the paste is kneaded until no further starch is removed. The small ball of moist gluten obtained is removed from the bag and dried in an evacuated tube immersed in a water-bath. The weight of dried material obtained multiplied by 3 gives approximately the weight of moist gluten obtained in the usual macro-test using 30—40 g. of flour.

A. R. POWELL.

Heat-resistant and heat-loving bacteria in their relation to the pasteurisation of milk. R. S. BREED (New York State Agric. Exp. Stat., Bull. No. 559, 1928, 19 pp.).—A review of recent investigations on the subject. Under certain conditions milk may contain more living bacteria after pasteurisation than before, owing to the introduction into the raw milk of both heat-resistant and heat-loving bacteria. The former, which survive pasteurisation temperatures, tend to grow well at 38—45° and produce the so-called "pin-point" colonies when incubated at 37° on agar plates; they are probably derived from insufficiently sterilised dairy utensils. The latter, which are not only not killed, but actually multiply rapidly at the pasteurisation temperature, do not grow on agar plates at 37° as the temperature is too low; their presence may therefore be overlooked in ordinary routine work, but they may be detected by incubation at 63° or as rod-like bacilli by microscopical examination of the pasteurised milk. The source of these heat-loving bacteria is the dust, hay, silage, etc. in proximity with the milk, and their development is favoured by faulty procedures during pasteurisation, such as holding the milk at pasteurisation temperature for longer than 30 min., repasteurisation of left-over milk, and the incomplete removal of one batch of milk before introduction of the next. There is no evidence to show that the presence of these bacteria in milk is dangerous to human health, but in excess they cause a noticeable change in the acidity of the milk and also disagreeable flavours.

F. R. ENNOS.

Types of bacteria which produce a "caramel" flavour in milk. C. D. KELLY (Trans. Roy. Soc. Canada, 1928, [iii], 22, V, 227—232).—The four strains of organisms studied are classified (on fermentative properties) as variants of *Streptococcus lactis* (Lister) according to Orla-Jensen's classification.

F. C. HAPPOLD.

***Streptococcus lactis* strain that produces "caramel" odour and flavour in dairy products.** W. SADLER (Trans. Roy. Soc. Canada, 1928, [iii], 22, V, 243—248).—The fermentative action of the organism on different carbohydrates has been quantitatively studied. The acid produced was titrated against 0.25N-caustic soda and recorded as lactic acid.

F. C. HAPPOLD.

Casein-splitting properties of starters [in ripening of cheese]. C. BARTHEL and W. SADLER (Trans. Roy. Soc. Canada, 1928, [iii], 22, V, 233—235).—The amounts of soluble nitrogen produced by pure cultures of lactic acid bacteria of the *Streptococcus* group is comparable with that produced by "starters," but the amount of amino-nitrogen produced is much

less with the former. This indicates that the hydrolysing action of the different *Streptococci* present in cheese is of a symbiotic nature.

F. C. HAPPOLD.

Proportion of the citrates of milk incorporated in the curd during cheese-making. N. S. GOLDING (Trans. Roy. Soc. Canada, 1928, [iii], 22, V, 237—242).—Only small amounts of the milk citrate are held by the curd; these amounts cannot affect significantly the ripening of cheese.

F. C. HAPPOLD.

Action of rennin. W. GRIMMER and E. HINKEL-MANN (Milch. Forsch., 1928, 6, 274—301; Chem. Zentr., 1928, ii, 1399).—Rennin, freed from pepsin by treatment with a small quantity of alumina, gave a product-curve which rose after a preliminary fall, and corresponded with the equation $J_1 = p/2(c_1^x + c_2^x)$; "peptolab," after purification, gave a falling logarithmic curve. In presence of phosphate "peptolab" gave almost constant product-values. A purified rennin preparation behaves like an unpurified preparation towards souring milk; the coagulation time decreases with increasing acidity.

A. A. ELDRIDGE.

Lipolysis of worked butter several days after preparation. O. GRATZ (World's Butter Rev., 1928, 2, No. 7—8, 21—22).—Bacteria develop most rapidly on the outer layer of butter; mixed butters have a greater acid content when the outer layer is not removed, and lipolysis in the butter may be greater after 14 days.

CHEMICAL ABSTRACTS.

Cacao beans and cocoa products. VI. H. FINCKE (Z. Unters. Lebensm., 1928, 56, 312—334; cf. B., 1928, 911).—Observations on the calculation of the composition of chocolate preparations are made in view of the specifications drawn up by the Verbande Deutscher Schokolade-Fabrikanten. Addition of coffee to chocolate does not necessitate modification of methods or calculations. The effect of fermentation on the appearance, taste, odour, and chemical composition of cacao beans has been studied. The fat of immature beans does not differ from that of riper beans. Fermentation does not change the properties of the fat. A case of an abnormal cacao butter from a consignment of Accra beans derived from only a few trees is reported. The analytical data indicate an unusually high content of unsaturated fatty acid of low m.p., probably oleic acid. Similar divergence from the normal could not be found in consignments from many different sources nor in the fat of individual beans. It is probable that the theobromine in cacao exists partly free and partly in loose combination with tannin, from which it can be liberated by moistening the material with addition of magnesium oxide as well as by treatment with dilute sulphuric acid.

W. J. BOYD.

Comparison between complete and incomplete digestion of sprayed apple foliage in determining arsenic by the Gutzeit method. J. M. GINSBURG (J. Econ. Entomol., 1928, 21, 588—592).—When the material is boiled for 30 min. with 10% nitric acid or 20% hydrochloric acid, the amount of arsenic recovered is equal to that obtained after complete digestion with nitric and sulphuric acids.

CHEMICAL ABSTRACTS.

Use of 2 : 6-dichlorophenol-indophenol as reduction indicator in the examination of foodstuffs. J. TILLMANS, P. HIRSCH, and E. REINSHAGEN (Z. Unters. Lebensm., 1928, 56, 272—292).—The behaviour of the indicator in the presence of milk, meat extract, and lemon juice was studied. Milk showed no well-defined action on it, but the other products exhibited definite reduction. In no case was it found possible to determine the reduction-oxidation potential colorimetrically thereby. The reducing effect of lemon juice on 2 : 6-dichlorophenol-indophenol provides a method of distinguishing the natural from the artificial product.

W. J. BOYD.

See also A., Mar., 337, Determination of iodine in milk (SCHWAIBOLD). 358, Vitamin content of honey (HOYLE). 362, Chemical changes in stored apples (HAYNES and ARCHBOLD). Composition of juices of some American apples (CALDWELL). Boron compounds in fruits and vegetable products (DODD).

PATENTS.

Making wheat-meal bread. D. FRAME (B.P. 305,845, 7.3.28).—The yeast is grown in an emulsion consisting of sweet milk, yolk of eggs, malt extract, and treacle, and then mixed with wheat meal, butter, and salt. The dough is divided, then placed in a chamber at 30° for 15 min., and baked at about 205°.

W. J. BOYD.

Egg product and its production. A. K. EPSTEIN (B.P. 305,844, 6.3.28).—Egg yolk, before being frozen, is treated with an amount of a neutral edible salt sufficient to produce in the product, when thawed, a viscosity greater than that of untreated yolk before freezing, and less than that of untreated yolk after being frozen and thawed. The addition of 2—10% of sodium chloride gives good results, but other neutral salts or neutral mixtures of salts may be used.

W. J. BOYD.

Food product. M. NILSSON, Assr. to FLEISCHMANN Co. (U.S.P. 1,701,081, 5.2.29. Appl., 2.3.26).—Compressed yeast, dried at about 30° to a moisture content of 55%, is mixed with dry sugar material, so that the resulting product contains about 20% of moisture and about 60% of sugar.

H. ROYAL-DAWSON.

Chewing gum. CANADIAN ELECTRO PRODUCTS Co., LTD., Assecs. of H. W. MATHESON (B.P. 280,247, 8.11.27. U.S., 8.11.26).—A gummy product is prepared by reaction between an aliphatic vinyl ester (e.g., vinyl acetate) and a saturated aliphatic aldehyde (e.g., acetaldehyde); sugar, flavouring ingredients, or gum chicle may be added.

W. J. BOYD.

Manufacture of ordinary bread. J. MATTI, Assr. to AUGMENTINE HOLDING Soc. ANON. (U.S.P. 1,701,580, 12.2.29. Appl., 25.6.27. U.K., 25.6.26).—See B.P. 266,967; B., 1927, 376.

Preservation of perishable [tinned] food products. D. ROBERTSON (B.P. 276,646, 4.8.27. N.Z., 30.3.27).

Mills for grinding etc (B.P. 303,227).—See I. Hydrolysed casein (B.P. 306,168).—See XV. Sterilisation (B.P. 274,909).—See XXIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of free iodine and potassium iodide in tincture of iodine (D.A.B. VI). K. WINTERFELD and H. SIECKE (Apoth.-Ztg., 1928, 43, 852—854; Chem. Zentr., 1928, ii, 1363).—The tincture (1—1.5 g.) is mixed with aqueous potassium bromide (3 g. in 10 c.c.) and diluted to 100 c.c. The iodine is determined by titration of a portion with thiosulphate. For the determination of potassium iodide, 10 c.c. are mixed with 25 c.c. of potassium hypobromite solution; after 15 min. phenol is added, and then hydrochloric acid to acidity (methyl-orange). Potassium iodide and phosphoric acid are then added, and after 20 min. the iodine is titrated with thiosulphate.

A. A. ELDRIDGE.

Determination of chloral in chloral syrup. C. LORMAND (J. Pharm. Chim., 1929, [viii], 9, 151—153; cf. Fleury and Malmy, B., 1929, 188).—10 g. of chloral syrup containing about 0.5 g. of chloral are added to a mixture of ammonia solution (*d* 0.925) 50 c.c., silver nitrate 4 g., and potassium hydroxide 5 g. The mixture is kept for 24 hrs., boiled on a water-bath to remove excess of ammonia, and then acidified with a slight excess of nitric acid. After diluting to 100 c.c. with water, the liquid is warmed to coagulate the silver chloride, which, after cooling, is collected and weighed. It is claimed that this process gives more accurate results than the alkalimetric titration process.

E. H. SHARPLES.

Liquor aluminii acético-tartarici, D.A.B. VI. H. MATTHES and P. SCHÜTZ (Apoth.-Ztg., 1928, 43, 1023—1024; Chem. Zentr., 1928, ii, 1469).—The (German) official method of analysis is criticised.

A. A. ELDRIDGE.

Determination of cresol in liquor cresoli saponatus. K. FEIST (Apoth.-Ztg., 1928, 43, 1024—1025; Chem. Zentr., 1928, ii, 1469).—5% of the light petroleum extract (D.A.B. VI) is treated with formaldehyde solution (5 c.c.) and 25% hydrochloric acid (4 c.c.), and the mixture dried to constant weight (1.0—1.1 g.) at 100° (cf. B., 1929, 188).

A. A. ELDRIDGE.

Nicotine dusts. W. R. HARLAN and R. M. HIXON (Iowa State Coll. J. Sci., 1928, 2, 313—316).—Losses of nicotine on adsorbents are due to adsorption, and not to chemical reaction. Nicotined bentonite, when extracted with ether, retains 88% (3% dust), 45% (6% dust), or 20% (14% dust) of nicotine, but less in presence of calcium hydroxide. The adsorbed nicotine can be recovered by steam-distillation. CHEMICAL ABSTRACTS.

Determination of pilocarpine. P. BOURCET (Ann. Falsif., 1929, 21, 23—24).—Jaborandi leaves (25 g.) are bruised, passed through a no. 30 brass sieve, moistened with 200 c.c. of 10% sodium carbonate, extracted for 3 hrs. with warm benzene, and the cooled solution is immediately extracted 4 times with 1% sulphuric acid, with shaking. The alkaloids dissolve, and the filtered solution, neutralised to Congo-red with ammonia, is oxidised by 10% potassium permanganate, rendered alkaline with ammonia, and repeatedly extracted with chloroform. The chloroform solution, filtered over sodium carbonate, is neutralised with nitric acid (1 : 50).

evaporated to dryness, and the residue extracted with acetone, which dissolves impurities, leaving the pilocarpine nitrate. After filtering and drying, the latter is weighed; the m.p. should be 174—175° and not less than 165°. In certain samples if the benzene solution of alkaloids is converted into sulphate and left for 24—48 hrs., particularly in light, the proportion of pilocarpine is found to have diminished by half, but if a benzene or light petroleum extract of the leaves themselves is treated with sodium carbonate and then with benzene this loss does not occur.

D. G. HEWER.

Use of the refractometric method in determination of oil in avocados. B. E. LESLEY and A. W. CHRISTIE (Ind. Eng. Chem. [Anal.], 1929, 1, 24).—The oil content of avocados may be rapidly determined within an error of 0.3% by transferring 5 g. of finely-ground and well-mixed avocado paste into a warm mortar, pipetting in 5 c.c. of Halowax oil (a commercial substituted monochloronaphthalene), grinding for 5 min., determining the refractive index on one drop of the oil mixture, which readily separates, and repeating the process after further intervals of grinding until a constant value is obtained, which is then corrected to 25° by adding or subtracting 0.0004 for every degree above or below 25°, respectively. The percentage (*c*) of oil present is $[(a - 0.0032) - b]/d$, where *a* and *b* are values of n_D^{25} for Halowax oil and the oil mixture, and *d* is the change in n_D per percentage of oil in the sample. The value obtained for several samples is 0.001555.

D. G. HEWER.

See also A., Mar., 297, **Catalytic reduction of geraniol and citronellal** (SUZUKI). 297, **Precipitation of carbohydrates and glucosides and by alkaloids** (ROSENTHALER). 313, **Resolution of *dl*-thyroxine** (HARINGTON). **Derivatives of thyroxine** (ASHLEY and HARINGTON). 315, **Products with an odour of musk** (RUZICKA). 323, **Transformation of pinocarveol and sabinol** (SCHMIDT). **Essential oil and alcohol from *Camphorosma Monspeliacum*** (MAZZA and SPAGNOLO). 335, **Amine oxides of hydrastine and narcotine** (POLONOVSKI). **Dehydrogenation of yohimbine** (MENDLIK). 336, **Organo-antimony compounds** (NIYOGI). 337, **Determination of iodine in thyroid extract** (SCHWABOLD). 351, **Lipo-soluble bismuth compounds** (LEVADITI). 357, **Standardisation of insulin** (FREUDENBERG and DIRSCHERL). 361, **Constancy of essential oil of seedlings of aniseed fruit** (IVANOV and GRIGOROJEVA). **Total alkaloids of *Datura fastuosa* and *alba*** (MARANON).

PATENTS.

Blood coagulant. M. BYE, Assr. to W. S. MERRELL Co. (U.S.P. 1,697,162, 1.1.29. Appl., 11.2.27).—A brine extract of lung or other tissue is treated with excess of acid, and the resulting precipitate is dissolved in caustic alkali. The water-white, neutral solution obtained may be evaporated to dryness.

F. G. CLARKE.

Therapeutic agent for treatment of bodily lesions. H. L. JOHNSON (U.S.P. 1,696,958, 1.1.29. Appl., 2.3.27).—Amniotic fluid of cows and other mammals, after removal of impurities and micro-

organisms by filtration, is applied to lesions of the human body during or after operations.

F. G. CLARKE.

Manufacture of solutions of the active principle of organs or glands with internal secretion. I. G. FARBENIND. A.-G. (B.P. 283,493, 7.12.27. Ger., 11.1.27. Addn. to B.P. 279,123; B., 1929, 226).—The process of the prior patent is applied to preparation of active principles from other organs, especially male germ-glands or the anterior lobe of the hypophysis.

C. HOLLINS.

Manufacture of optically active phenylpropanol-methylamines [β -methylamino- α -phenyl-*n*-propyl alcohols; *l*-ephedrine]. I. G. FARBENIND. A.-G. (B.P. 297,385, 11.9.28. Ger., 20.9.27).—By resolution of *dl*- β -methylamino- α -phenyl-*n*-propyl alcohol (ephedrine) with *l*- instead of with *d*-tartaric acid the *l*-tartrate of *l*-ephedrine, m.p. 148° (hydrochloride, m.p. 215—216°, $[\alpha] - 34^\circ$), crystallises before the *d*-ephedrine *l*-tartrate (*d*-base, m.p. 39—40°; hydrochloride, m.p. 215°, $[\alpha] + 34^\circ$). The *l*-base is therapeutically more valuable than its enantiomorph.

C. HOLLINS.

Manufacture of urethanes [carbamates] of secondary alcohols. I. G. FARBENIND. A.-G. (B.P. 300,177, 6.11.28. Ger., 7.11.27).—Carbamates of secondary alcohols containing an aryl group γ to the hydroxyl group are powerful antipyretics. The alcohol is treated first with carbonyl chloride and then with ammonia. Examples are δ -phenyl- β -*n*-butyl carbamate, $\text{CH}_2\text{Ph} \cdot \text{CH}_2 \cdot \text{CHMe} \cdot \text{O} \cdot \text{CO} \cdot \text{NH}_2$, m.p. 63°, ε -phenyl- γ -*n*-amyl carbamate, m.p. 88°, Δ^a - δ -benzyl- ε -*n*-hexenyl carbamate, $\text{CH}_2 : \text{CH} \cdot \text{CH}_2 \cdot \text{CH}(\text{CH}_2\text{Ph}) \cdot \text{CHMe} \cdot \text{O} \cdot \text{CO} \cdot \text{NH}_2$, m.p. 42°, and δ -*p*-tolyl- β -*n*-butyl carbamate, m.p. 77°.

C. HOLLINS.

Preparation of monocyclic lactones with 14 to 18 ring members. SOC. ANON. M. NAEF & CIE. (B.P. 306,352, 26.6.28. Addn. to B.P. 294,602; B., 1929, 235).—Cyclic ketones, C_{13} to C_{17} , when warmed at 50° with Caro's acid yield lactones of the corresponding ω -hydroxy-acids; cyclopentadecanone is disclaimed. The lactones described are: C_{13} (lactone of ω -hydroxy-tridecoic acid), m.p. 26—27°, b.p. 148°/15 mm., d_4^{25} 0.9590; C_{14} , m.p. 29—30°, b.p. 165°/15 mm., d_4^{25} 0.9528; C_{16} , m.p. 35—36°, b.p. 188°/15 mm., d_4^{25} 0.9396; C_{17} , m.p. 41—42°, b.p. 194°/15 mm., d_4^{25} 0.9326; ξ -hydroxy- γ -methylpentadecanoic lactone, b.p. 180°/15 mm., d_4^{25} 0.9305, from muscone; ξ -hydroxy- α -methylpentadecanoic lactone, b.p. 178°/15 mm., d_4^{25} 0.9301, from 2-methylexaltone.

C. HOLLINS.

Manufacture of metal mercaptocarboxylic acid esters. SCHERING-KAHLBAUM A.-G. (B.P. 282,427, 15.12.27. Ger., 16.12.26).—*o*-Thiolbenzoic esters, best obtained from the acid chloride of 2:2'-dicarboxy-diphenyl disulphide by interaction with alcohols and subsequent reduction, are heated with oxides or salts of gold, silver, arsenic, antimony, or bismuth to yield metalthiol derivatives soluble in olive oil, sesame oil, glycerol triethyl ether, and other solvents suitable for injection. *n*-Butyl *o*-thiolbenzoate [“thiosalicylate”], b.p. 163°/20 mm., gives a bismuth compound, m.p. 72°; the methyl ester, b.p. 130°/20 mm., yields an antimony

compound, m.p. 88°. Aliphatic thiol-substituted acids give similar compounds. C. HOLLINS.

Manufacture of arsenobenziminazolones. I. G. FARBENIND. A.-G. (B.P. 281,690, 1.12.27. Ger., 2.12.26).—Benziminazolonearsinic acids carrying *N*-substituents (cf. B.P. 256,243; B., 1927, 670) give on reduction arseno-compounds more stable and effective than known arsenicals. Arsenobenziminazolones from 1-methyl-, 1-propyl-, and 1-allyl-benziminazolone-5-arsinic acids are described. C. HOLLINS.

Manufacture of a therapeutically active iron preparation having yeast as its basic substance. G. M. CLARK. From MATRO GES.M.B.H. (B.P. 304,895, 24.12.27. Cf. B.P. 300,039; B., 1929, 73).—To ordinary or acetonised yeast suspended in 10 pts. of water is added a solution of a colloidal basic iron salt (liquor ferri chloroxidi, or liquor ferri chloroxidi dialysati). The liquid is shaken overnight, and the solid filtered off, treated with alcohol and ether, and dried at low temperature. B. FULLMAN.

Manufacture of tobaccos. SOC. ZIHN (B.P. 298,151, 26.6.28. Fr., 4.10.27).—Low-grade tobacco is, preferably after fermentation of the leaves, treated by immersion, spraying, etc. with a solution obtained by diluting 1 pt. of the extract of leaves, waste, or stalks of high-grade tobacco with 10 pts. of alcohol or other volatile solvent. Finished cigarettes or cigars may be treated by injection. The product is allowed to dry. B. FULLMAN.

Improvement of perfumes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 305,555, 29.10.27).—Substances such as ionones, geraniol, citral, clove oil, etc. are subjected to partial hydrogenation with or without the presence of a catalyst; e.g., 500 g. of methylionone are treated at 60–90° with 30 litres of hydrogen in presence of 5 g. of catalytic nickel for 1 hr.; after distilling in a vacuum, a colourless or nearly colourless methylionone free from the usual empyreumatic odour is obtained. E. H. SHARPLES.

Complex antimony compound. H. HANL, Assr. to WINTHROP CHEM. Co., INC. (U.S.P. 1,701,234, 5.2.29. Appl., 10.2.27. Ger., 22.4.26).—See B.P. 288,370; B., 1928, 427.

Derivatives of cinchona alkaloids. E. BURCKHARDT, F. MÜLLER, and E. ROTHLIN, Assrs. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,701,138, 5.2.29. Appl., 30.11.27. Switz., 17.12.26).—See B.P. 282,356; B., 1928, 769.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effect of environment on photographic sensitivity. II. Effect of certain salts. S. E. SHEPPARD and E. P. WIGHTMAN (Phot. J., 1929, 69, 134–141; cf. B., 1929, 151).—Potassium citrate, whether present during or immediately after exposure, has no effect on the formation of the latent image, but potassium chloride under the same conditions produces a slight decrease in sensitivity which, however, is attributed to a development effect. Potassium bromide, when present during exposure, markedly inhibits the formation

of the latent image, but when present immediately after exposure is almost without action. Although acid silver nitrate causes a strong acceleration of the rate of print-out of the visible image, it has no such effect on the formation of the latent image. The results are discussed in terms of the transfer of electrons from bromide ions to silver ions of the lattice, and it is suggested that the electrostatic field of the adsorbed double layer is a modifying factor of differing importance for visible- and latent-image formation.

J. W. GLASSETT.

See also A., Mar., 277, Photolysis of silver bromide (MUTTER). Sensitometry of desensitised films (ARENS and EGGERT). 359, Action of the cholesterol of cod-liver oil on the photographic plate (HUGONENQ and COUTURE).

PATENTS.

Colour photography. S. DE PROCOUDINE-GORSKY (B.P. 293,038, 25.6.28. Switz., 30.6.27).—A positive is made on sensitised celluloid film from each of the three colour record images, using a hardening developer such as pyrogallol. The positives are fixed, washed, dried, and then immersed in a saturated solution of iodine in 1% potassium iodide, which converts the reduced silver into silver iodide. Each image is then treated in a separate bath containing sodium thiosulphate and a basic dye of suitable colour (e.g., methylene-blue, Auramine, red-magenta), which fixes the dye in the images and slowly removes the silver iodide.

J. W. GLASSETT.

Colour photography. O. E. WHEELER (B.P. 305,775, 28.12.27).—Bromide prints are made from the three negatives representing the blue, red, and yellow impressions. These are squeezed in contact with an uncoloured, gelatin-coated tissue sensitised as in the "Carbro" process and, after separation, the tissues are transferred to a celluloid support and developed in warm water. The relief images so obtained are used, by means of a dye imbibition method of printing, to obtain prints on any suitable support.

J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Thermodynamic treatment of explosive reactions. I. A. SCHMIDT (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 41–46).—Methods are given for calculating for a given explosive its heat of explosion, explosion temperature, the composition and volume of the explosion gases at that temperature, and the explosion pressure when the heat of formation and the stoichiometric composition of the explosive in question are known. Nitroglycerin, nitrocellulose, and trinitrotoluene are taken as examples of explosives with excess, sufficiency, and deficiency of oxygen, respectively. S. BINNING.

Electrical conductivity of the boiled [washing] water [in the purification] of cellulose nitrate. K. ATSUKI and N. HONDA (J. Cellulose Inst., Tokyo, 1929, 5, 3–5).—In the stabilisation of nitrocellulose by repeatedly boiling with water, the electrical conductivity of the washing water gives useful indications of the degree of purification of the nitrocellulose. When the specific electrical conductivity of the wash water reaches a

constant minimum value, which, however, is still higher than that of the water used, the ignition point of the nitrocellulose becomes constant, though the Abel heat test becomes higher with further boiling, presumably due to the decomposition of the nitrates of degraded celluloses. D. J. NORMAN.

See also A., Mar., 260, **Behaviour of cellulose nitrate gels in polarised light** (PHILLIPS).

PATENTS.

Explosives. A. C. SCOTT (B.P. 305,722, 11.11.27).—Crystalline oxygen carriers, *e.g.*, potassium chlorate, potassium nitrate, or sodium nitrate, are ground with asbestos, and the mixture is then impregnated with a combustible substance, *e.g.*, kerosene, nitrobenzene, or nitrotoluene. Previous to impregnation the mixture may be moistened and then pressed with subsequent granulation. S. BINNING.

Separation and after-separation of nitroglycerin, or nitroglycol, and similar explosives from the acids used in their preparation. A. SCHMID and J. MEISSNER (B.P. 284,701, 28.1.28. Ger., 5.2.27).—Nitroglycerin and nitroglycol are separated continuously from their emulsions in the waste acid by passing the emulsion into a vessel containing inclined plates between which the small drops of nitroglycerin etc. separate out easily and collect on the under-surface of a plate. S. BINNING.

Nitration of cellulose. HERCULES POWDER CO., Assees. of M. G. MILLIKEN (B.P. 291,082, 11.4.28. U.S., 28.5.27).—Cellulose is nitrated continuously by feeding it through a vertical hopper shoot on to a horizontal worm working in an enclosed perforated cylinder. Excess of nitrating acid is poured on to the cellulose when it enters the cylinder, and more is added through the perforations. The length of the cylinder and speed of the worm are adjusted so that complete nitration has taken place by the time that the cellulose reaches the end of the cylinder. The nitrocellulose passes through a perforated head box on to a conveyor, which passes it under a roller to remove excess acid, and then through another roller through which air passes and removes more waste acid. The nitrocellulose then passes to a submerging vessel, and thence through a flume to any suitable washing plant. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Toxicity of hydrogen cyanide. J. B. ALLISON (Iowa State Coll. J. Sci., 1928, 2, 243—252).—Data for cockroaches, rice weevils, and albino rats are recorded. The equation, toxicity = concentration \times time, is not universally applicable. CHEMICAL ABSTRACTS.

Mosquito breeding [in water] and malaria in relation to the nitrogen cycle. K. B. WILLIAMSON (Bull. Entomol. Res., 1928, 18, 433—439).—The following conditions, but not the p_H of the water, are important: efficient oxidation of the products of protein degradation in the water, the ratio of oxidised to ammoniacal nitrogen, the amount of dissolved oxygen, and the presence of micro-organisms. CHEMICAL ABSTRACTS.

Olszewski-Keyl water-sampling apparatus. W. OLSZEWSKI (Chem.-Ztg., 1929, 53, 149).—The apparatus serves for taking a water sample for bacteriological testing from a determined depth. A falling weight causes the frame carrying the bottles to rotate on its base plate. This allows the water to enter and a 1.5 litre sample bottle is filled in 1 min. At the same time the temperature is recorded. C. IRWIN.

Determination of borate in natural waters. M. D. FOSTER (Ind. Eng. Chem. [Anal.], 1929, 1, 27—28).—1 c.c. of hydrochloric acid (4 c.c. of acid, d 1.178—1.183, to 96 c.c. of water) and 1 c.c. of turmeric solution (5 g. of powdered turmeric repeatedly washed in water, dissolved in 500 c.c. of alcohol, and filtered before use) are added to 10 c.c. of sample and slowly evaporated to dryness. A golden residue indicates absence and a pink colour presence of borate. The quantity present is found by comparison with standards which may range from 0.0 to 0.5 mg. of BO_3 , a difference of 0.0025 mg. of BO_3 showing an appreciable difference in tint, but above 0.01 mg. the standards must have a wider range, 0.01, 0.03, 0.06, 0.10, 0.25, and 0.5 mg. of BO_3 being suitable. If a larger amount of sample is necessary it is more satisfactory to evaporate the whole bulk rather than instalments. D. G. HEWER.

Waters of Argyle lagoon. I. P. BLALOCK and T. G. THOMPSON. II. G. H. HITCHINGS, S. P. TODD, and T. G. THOMPSON (Pub. Puget Sound Biol. Sta., 1928, 5, 325—332, 341—353).

Carbon dioxide tension of the Fraser River and its lower tributaries and of certain tributaries of the Columbia River. E. B. POWERS and T. A. HICKMAN (Pub. Puget Sound Biol. Sta., 1928, 5, 373—380).

Manganese in Iowa City waters. E. BARTOW and W. T. BAILEY (Proc. Iowa Acad. Sci., 1927, 34, 191—195).

See also A., Mar., 284, **Distinguishing between tap water and distilled water and detection of alkali in water** (FEIGL and others).

Nitrates in water. DE NARDO.—See XVI.

PATENTS.

Method and apparatus for sterilisation. G. A. KRAUSE (B.P. 274,909, 25.7.27. Ger., 26.7.26).—Foodstuffs etc. liable to undergo bacterial decomposition are preserved by storage in or impregnation with water, salt solutions, etc. which have been oligo-dynamically activated by contact with silver or copper or their alloys. The activated water may be concentrated by evaporation, and may be stirred, *e.g.*, by agitation with bubbles of air etc. during use. L. A. COLES.

Sewage treatment by activated sludge combined with a preliminary tank treatment. K. IMHOFF (U.S.P. 1,700,722, 29.1.29. Appl., 25.8.24. Ger., 15.7.24).—The sewage is successively passed through a settling tank, an aeration tank, and a sedimentation and separation tank, and from there is returned to the settling tank. H. ROYAL-DAWSON.

Separating tarry substances from water (B.P. 279,465).—See II.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 26, 1929.

L—GENERAL; PLANT; MACHINERY.

Nomography. XIII. O. LIESCHE (Chem. Fabr., 1929, 124—125).—A single nomogram is constructed to effect all conversions from English into metric units. A left- and a right-hand scale give the unit readings in the two systems, and different fixed points on an intermediate parallel scale correspond to the nature of the conversion: feet into metres, gallons into litres, etc. C. IRWIN.

Crystal growth in aqueous solution. II. W. L. McCABE (Ind. Eng. Chem., 1929, 21, 112—119; cf. B., 1929, 153).—Experimental verification of the author's theory of crystal growth is provided. The substances investigated were potassium chloride and copper sulphate pentahydrate. A mass of crystals of known weight and sieve analysis was suspended at constant temperature in a saturated solution for a definite time. The crystals were then collected, weighed, and again sieved. If the small crystals were appreciably more soluble than the larger, the final sieve analysis would be expected to show a larger percentage of the coarse crystals than was originally present. No systematic growth of the large crystals at the expense of the smaller could be observed even after 18 hrs.' contact with a saturated solution.

H. INGLESON.

Apparatus for indicating saturation of an absorption system. A. MACKERT (Chem.-Ztg., 1929, 53, 228).—See B., 1929, 191.

PATENTS.

[Gas-fired] furnaces. R. GUNKA (B.P. 302,714, 21.9.27).—A furnace suitable for heating crucibles or for annealing is formed with inner and outer walls of insulating materials, metallic preheating tanks being placed in the annular space through which the products of combustion are exhausted. The air (or oxygen) and gas are supplied separately under high pressure through regulating cocks coupled together, through the preheating tanks to an annular jet where combustion takes place. The annular jet has a dead space in the centre which causes the flame to close on itself and give a high temperature. B. M. VENABLES.

Heat-treatment furnace. H. P. McCANN (U.S.P. 1,697,129, 1.1.29. Appl., 21.2.25).—The furnace comprises an upper and a lower heat-treatment chamber through which the material to be treated is passed in containers running along rails. Between these two chambers are two further chambers the lower one of which serves as a combustion chamber and the upper as a preheating chamber; the gases from the former pass downwards through the lower heat-treatment chamber, then upwards through the preheating chamber, into the upper heat-treatment chamber. A. R. POWELL.

Revolving ring-hearth oven. L. HONIGMANN and F. BARTLING (U.S.P. 1,697,006, 1.1.29. Appl., 2.6.26. Ger., 2.6.25).—A revolving annular hearth forms the bed of an annular oven, and concentric flues for heating gases are placed below the hearth with a space between accommodating the hearth-driving mechanism; between the lower part of the latter and the hearth are cooling troughs to prevent transmission of heat. Four sealing troughs are provided between the hearth and walls of the annular flues. B. M. VENABLES.

Tunnel kiln. G. W. BOOTH (U.S.P. 1,698,700, 8.1.29. Appl., 28.3.27).—A tunnel kiln is provided with a separate drying chamber. Air is drawn through an air supply passage divided into sections, each connecting with a longitudinal distributing passage in the wall of the drying chamber and having openings into the drying chamber, from which there are a series of openings to a longitudinal, moist-air flue. B. M. VENABLES.

Device for utilising the heat radiated from kilns. F. LUTHER, Assr. to G. POLYSIUS (U.S.P. 1,698,313, 8.1.29. Appl., 4.3.27).—The kiln is surrounded by a heat-absorbing cooling drum, and air is passed through the space between the drum and kiln and then into the kiln. B. M. VENABLES.

Regenerative air heater. W. E. DOWD, JUN., Assr. to POWER SPECIALTY Co. (U.S.P. 1,697,591, 1.1.29. Appl., 24.4.24).—The heat is transferred from one fluid to another in adjacent conduits by means of regenerative material which travels longitudinally in one conduit, across to and longitudinally in the other, and back to the first. B. M. VENABLES.

Heat exchangers. W. E. KOCHS & Co., LTD. From H. FÖGE (B.P. 302,562, 21.7.28).—A heat exchanger is constructed of a number of layers of annular cells, the cells of each layer being connected by hollow ribs which rise towards the centre of the apparatus, where are situated the means of entrance and exit for the fluid media. B. M. VENABLES.

Heat-exchange apparatus. SOC. ANON. DES ÉTABL. DELAUNAY BELLEVILLE (B.P. 284,338, 26.1.28. Fr., 29.1.27).—In a heat exchanger of the type where the outer fluid passes over a nest of tubes longitudinally except at entrance and exit, more space is provided at those points by reducing the diameter of the tubes, either by an actual decrease in size or by flattening. B. M. VENABLES.

Chequer work, particularly intended for heat accumulators. F. KRUPP A.-G. FRIEDRICH-ALFRED-HÜTTE, and H. BANSSEN (B.P. 293,447, 29.5.28. Ger., 8.7.27).—The chequer work is divided into a number of stories with the passages in any storey staggered in relation to those in the next. The intermediate brick-

work is stepped and provided with horizontal passages, the combined area of which is at least as great as that of the vertical passages with which they communicate.

B. M. VENABLES.

Pulverising apparatus. RILEY STOKER CORP., Asses. of O. CRAIG (B.P. 302,330, 16.6.28. U.S., 15.12.27).—A form of construction of an impact pulveriser is described in which the wearing parts are individually small and easily renewed.

B. M. VENABLES.

Pulveriser. P. S. KNITTEL (U.S.P. 1,698,758, 15.1.29. Appl., 21.10.27).—The grinding surface comprises grate bars having different spacings, with a gate outside each zone; the zone of widest spacing has also a cross-wall. The size of the material discharged is regulated by closing or opening the gates.

B. M. VENABLES.

Pulverising device. H. G. LYKKEN (U.S.P. 1,699,849, 22.1.29. Appl., 3.11.24).—The pulverised material is drawn from a pulverising chamber to an expansion chamber; the latter is provided with a screen across it, some distance from the entrance, to equalise the flow.

B. M. VENABLES.

Pulverising systems. INTERNAT. COMBUSTION, LTD., and F. H. ROSENCRANTS (B.P. 302,610, 18.7.27).—In a grinding system with air separation in which the cyclone collector operates under slight vacuum, the dust outlet of the cyclone is provided with a flap-valve, which is surrounded by a casing provided with a rotary valve at the bottom. The cyclone is lined with wearing plates spaced from the wall, the space being filled with cement or powder or allowed to become packed with the powder collected in the cyclone.

B. M. VENABLES.

Pulverising machine. J. TEIPEL, Assr. to AMER. PULVERIZER Co. (U.S.P. 1,696,219, 25.12.28. Appl., 29.4.27).—A disintegrator of the hammer type is provided with grinding plates adjustable by pivoting, the adjustment being effected by an eccentric shaft parallel to the axis and forming the abutment of the grinding plate concerned.

B. M. VENABLES.

Pulverising machine. P. S. KNITTEL (U.S.P. 1,698,268, 8.1.29. Appl., 21.5.27).—The pulveriser comprises rotary means co-acting with a concave grinding grate, and a number of pivoted fingers form a yielding surface in continuation of the same curve as the grate.

B. M. VENABLES.

Pulverising machinery. GEN. ELECTRIC Co., LTD., and B. POCHOBRADSKY (B.P. 304,865, 15.11.27).—A pulveriser, preferably of the impact type, has a stream of primary air drawn through it, and the larger particles are separated from the stream of air either by simple settling from the vertically-flowing stream or in combination with deflectors or baffles. After separation of the coarse material (which may be returned to the pulveriser) an additional stream of secondary air is admitted which causes the ground material to remain definitely in suspension, and the whole may be passed through an extracting fan to a volute exhaust passage.

B. M. VENABLES.

Crushing machine. P. L. CROWE (U.S.P. 1,696,387, 25.12.28. Appl., 25.11.25).—The machine comprises

a hopper of which one wall oscillates in relation to the opposite wall, about an elastically supported pivot.

B. M. VENABLES.

Grinding or crushing machines. BURT, BOULTON, & HAYWOOD, LTD., and F. J. E. CHINA (B.P. 303,568, 12.8.27).—In a grinding machine having a slight planetary motion, the gyration of the axis of the rotor is derived from driving means separate from that which produces rotation of the rotor about its own axis. In a construction described, the stator and rotor are conical and an eccentrically rotating roller bears on the inner surface of the rotor, the pressure of the roller being spring-controlled through a toggle gear.

B. M. VENABLES.

Disintegrating machine. B. CUNIFF and N. LEE, Assrs. to COLLOIDAL EQUIPMENT CORP. (U.S.P. 1,697,052, 1.1.29. Appl., 29.10.27).—A pair of opposed rotors are rotated at different velocities by hubs running on a fixed shaft.

B. M. VENABLES.

Grinding mill. S. BJERRE (U.S.P. 1,697,750, 1.1.29. Appl., 19.5.25. Denm., 6.6.24).—One of a pair of grinding members is rotated by a hollow shaft, which also oscillates a feed device. The other grinding member is attached to a solid shaft, not rotating, but capable of angular displacement by the action of the friction of the grinding discs balanced against a resilient restraining means. When an overload occurs the friction will increase and the displacement will reduce the feed until the overload has worked off.

B. M. VENABLES.

Combined grizzly and ball mill. A. F. LEVITT (U.S.P. 1,697,531, 1.1.29. Appl., 11.7.27).—A rotating drum is provided with lengthwise bars placed round the periphery and transversely dividing the grinding media into a number of groups, forming grizzlies for the material being ground.

B. M. VENABLES.

Attrition mill. J. G. BRYANT (U.S.P. 1,698,838, 15.1.29. Appl., 22.3.26).—The mill comprises a frame with vertical end-walls provided with aligned bearings supporting a shaft driving a grinding element which co-operates with another grinding element fixed to the frame.

L. A. COLES.

Granulation of pulverised materials. O. LELLEP (B.P. 292,987, 2.4.28. Ger., 2.4.27).—Granules are formed from powder by adding water in the form of droplets to the powder while in motion. After the formation of wet granules, further powder may be added to stiffen them, and screens may be used to separate dust, which is returned for re-treatment.

B. M. VENABLES.

Grading of material. D. D. DUNKIN (U.S.P. 1,699,522, 22.1.29. Appl., 27.9.27).—A conveyor belt takes a triangular course. Powdered material is supplied to the belt on its upward course at a moderate inclination; the largest particles will roll down and be collected; the remaining material passes over the apex of the triangle and down a slope of greater inclination, which will cause moderate-sized particles to roll off. The finest material adhering to the belt is removed, preferably by washing, from the underside of the third horizontal pass.

B. M. VENABLES.

Separation of intermixed divided materials. K. DAVIS, ASST. to PEALE-DAVIS CO. (U.S.P. 1,695,369, 18.11.28. Appl., 8.12.21).—The material slides over an annular, sloping, stationary table, is stratified by currents of air passing through the table, and the various strata are caught in different receiving chutes.

B. M. VENABLES.

Dryer. C. F. MORRISON, ASST. to MORRISON-MERTINS, INC. (U.S.P. 1,696,172, 18.11.28. Appl., 2.11.27).—A rotary tumbler is divided into segmental sections with spaces between serving to admit air through adjustable valves.

B. M. VENABLES.

Dryer. O. B. BROWN and H. PARKER, ASSTS. to BROWN CO. (U.S.P. 1,697,454, 1.1.29. Appl., 16.7.25).—The articles to be dried are conveyed through a chamber in the opposite direction to the general flow of drying agent, and means are provided for withdrawing some of the drying agent from and returning it to a number of different levels in the chamber.

B. M. VENABLES.

Drying cylinders of drying machine. W. P. EVANS (B.P. 302,785, 17.11.27).—A drying cylinder heated by fluid is arranged so that the fluid enters and leaves at one end of the drum; the other end of the drum is blank and is provided with resilient means to accommodate end-thrust. Both ends are mounted on anti-friction bearings.

B. M. VENABLES.

Removal of moisture from solid materials. M. JUNG (B.P. 292,488, 18.6.28. Ger., 17.6.27).—Moisture is separated from damp solid materials, or insoluble matter is separated from an emulsion, by passing the material in thin layers by centrifugal force over perforated radial blades, through which a current of gas or air is passed. The gas currents are produced by a fan in a separate chamber, and vanes are provided to prevent turbulence.

B. M. VENABLES.

Aeration process for drying, carbonisation, and oxidation plants, and apparatus therefor. R. GLINKA (B.P. 303,572, 10.10.27).—The material is arranged in annular tray-like supports, and air is driven by one or more fans within the annuli radially over the trays and returns to the suction zone of the fan and may be heated during circulation.

B. M. VENABLES.

Production of dry powders from solutions, emulsions, suspensions, etc. N. J. NIELSEN (B.P. 303,576, 10.10.27).—A drying gaseous medium carrying finely-divided particles is passed through two or more cyclones in series. One form of apparatus comprises two tower-like structures; the upper part of the first comprises a drying chamber, the lower parts of the two towers comprise cyclones in series, and the upper part of the second is a screen chamber. A third tower may be provided containing humidifying means.

B. M. VENABLES.

Mixing apparatus. T. W. BARBER (B.P. 302,745, 30.9.27).—One fluid is forced from an inner passage through a number of small radial holes into the other fluid flowing in an outer passage. The inner passage takes the form of a cylinder and is provided with a trunk piston which is spring or gravity-controlled so as to cover the jets when no liquid is flowing, and to uncover only a sufficient number of jets to pass the liquid which is forced in.

B. M. VENABLES.

Mixing machine. O. R. READ, ASST. to READ MACHINERY CO., INC. (U.S.P. 1,695,345, 18.12.28. Appl., 3.6.27).—Means are described for raising and lowering the non-rotating bowl of a mixing machine.

B. M. VENABLES.

Mixing machines. G. STEELE and S. D. COWLISHAW (STEELE & COWLISHAW) (B.P. 302,556, 21.6.28).—A barrel and an interior bladed shaft are rotated in opposite directions from a common countershaft through spur-gearing and chain or belt, respectively.

B. M. VENABLES.

Multi-stage mixer. I. G. FARBERNIND, A.-G. (B.P. 302,141, 30.4.28. Ger., 10.12.27).—A multi-stage turbine or centrifugal pump has the rotors for the several stages mounted on one shaft either concentrically or in line. Between each stage a receiver for the liquid is placed; there may be short-circuiting pipes for liquid and/or gas between the receivers, and they may be provided with heating jackets.

B. M. VENABLES.

Tangential separator. M. G. MARTLING (U.S.P. 1,698,101, 8.1.29. Appl., 18.10.27).—A conical spiral conduit decreases in cross-section towards its exit end; its outer wall is perforated at intervals, affording communication to a number of pockets containing fluid at rest.

B. M. VENABLES.

Centrifugal-type dust separators. C. H. CRAGO and A. E. HAMILTON (B.P. 304,952, 26.3.28).—A centrifugal fan provided with collecting means for dust in its periphery has air drawn through it axially by means of an extractor or circulating fan. Two centrifugal fans may be placed one at either inlet of the extractor, or several centrifugals may be in series with one extractor. The centrifugals are surrounded by a closed dust chamber. The size of the dust-collecting slots is variable, and regulators are provided to short-circuit a portion of the air from the circulating fan back to the centrifugal.

B. M. VENABLES.

Centrifugal-separator feeding device. V. J. GILMORE, ASST. to DE LAVAL SEPARATOR CO. (U.S.P. 1,694,468, 11.12.28. Appl., 19.5.27).—A supply can is arranged to swing in a horizontal plane, and is provided with an outlet cock of which the plug is so shaped and placed at such an angle that when open the outlet of the cock projects below the top of the feed cup of the centrifuge, but when closed is turned up out of the way so that the can can be swung back for a new charge.

B. M. VENABLES.

Centrifugal extractor. F. BALZER, ASST. to TROY LAUNDRY MACHINERY CO., INC. (U.S.P. 1,694,614, 11.12.28. Appl., 13.2.25).—A centrifugal bowl is driven by a motor through a centrifugal clutch, and is braked by reversing the motor.

B. M. VENABLES.

Apparatus for separating fluids from solids. H. NEWALL, and J. CROSFIELD & SONS, LTD. (B.P. 303,649, 5.1.29).—The material is passed between rollers, one of which is surrounded by a perforated or reticulated loose sleeve, or the roller itself may be constructed of wires or bars on a skeleton frame.

B. M. VENABLES.

Filters. H. A. VALLEZ (B.P. 303,707, 23.4.28).—A filter, preferably of the pressure type, comprises a number of leaves arranged longitudinally around a hollow, rotating shaft, each leaf being provided with an

individual filtrate discharge pipe, cut-off valve, and sight glass. The individual pipes are all attached to a rotating plate forming one side of a bus chamber which contains a valve suitable for simultaneously closing all the filtrate pipes. Provision is also made for air to be admitted to the discharge pipes one at a time when the bus valve is closed, and thence to the leaf which happens to be lowest so that the cake may be blown off.

B. M. VENABLES.

[Sand] filters. BAMAG-MEQUIN A.-G. (B.P. 304,967, 30.4.28. Ger., 20.2.28).—Each effluent orifice of a rapid sand filter is utilised during flushing as a water-operated air injector.

B. M. VENABLES.

Filters particularly applicable for the treatment of oil and petrol. G. CATTANEO (B.P. 287,932, 28.3.28. Italy, 30.3.27).—A number of concentric filter elements mutually support one another and are open alternately at the top and bottom for the inlet and outlet of fluid.

B. M. VENABLES.

Spraying of liquids. J. FORD (B.P. 302,817, 11.1.28).—The liquid enters from below a number of "box-like members" (stand pipes) with open ends, and sprays out through orifices round the lower circumferences. The orifices may be tapered larger outwards to avoid choking by liquids like sewage.

B. M. VENABLES.

Preparation of emulsions of liquids or solutions. B. REDLICH (B.P. 302,761, 20.10.27).—A pure, colloidal, precipitated, inorganic, powdery substance of high absorptive capacity (*e.g.*, dried silicic acid) is used as an aid to emulsification; the solid aid may afterwards be almost completely precipitated by settlement, centrifugal action, etc.

B. M. VENABLES.

Emulsifying apparatus. G. L. FISH, Assr. to R. SALISBURY (U.S.P. 1,694,589, 11.12.28. Appl., 25.11.25).—The several liquids to be emulsified are measured and mixed with a gas ("expansible fluid"), and the mixture is subjected to a high pressure and then expanded. The pressure of the mixture regulates the supply of liquids and gas.

B. M. VENABLES.

Vapour extractor. M. H. KOTZEBUE (U.S.P. 1,695,192, 11.12.28. Appl., 28.5.27).—A tower is provided with a number of trays, some having bubbling units. A pair of closely spaced trays have pipes leading therethrough; the lower parts of the pipes are provided with vanes causing whirling, the upper parts are plain, and between the two the pipes are interrupted so that communication is afforded to the dead space between the trays, whereby some of the whirling matter is taken from the gases moving upward through the pipes.

B. M. VENABLES.

Vacuum [evaporating] apparatus. G. B. ELLIS. From E. MERCK (B.P. 303,661, 21.1.28).—The wall of the vacuum chamber is used as a condensing surface by providing it with a cooling jacket. Within the chamber the goods to be dried may be surrounded by hot-water conduits. The joint of the casing is sealed by hot wax poured into a trough embracing the joint, and, if necessary, the wax is kept solid by a cooling pipe in the trough. [Stat. ref.]

B. M. VENABLES.

Piston pumps for corrosive liquids. A. FERRARIS (B.P. 289,018, 9.3.28. Italy, 20.4.27).—In a pump

where a body of liquid such as oil is interposed between the piston and the liquid to be pumped, the piston is not of the trunk type, but is provided with a comparatively thin piston rod which works in a small stuffing box in the cylinder head. There are no piston rings, the piston being merely a good fit in the cylinder. A spring-loaded valve is provided in the cylinder head admitting air when necessary to the space between the piston and head, and a similar valve allows oil which has worked above the piston to be driven back through the piston by the cushion of air above it, on the up-stroke. There is no loss of oil, the only stuffing box is small, and leakage of air through it is immediately replaced.

B. M. VENABLES.

Air separator. J. W. DREISBACH (U.S.P. 1,698,361, 8.1.29. Appl., 22.9.27).—The material enters a casing through an annular side inlet and passes downwards through the space between the outer wall and a hub-shaped baffle. The coarse material falls on a rotary disc at the bottom provided with scrapers to discharge the coarse, forming a false bottom adapted to be sealed by the coarse material at its periphery. Rotary, impact, fan blades are placed in the path of the fines, which are exhausted through a pipe depending within the apparatus.

B. M. VENABLES.

[High-pressure] valves. H. P. DEAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 302,834, 17.2.28).—The valve seat forms the sealing piece between the two parts of the body of the valve, forming ring contacts therewith, and the area enclosed within the contact circle is not more than twice the exposed area of the valve stem. The valve head is loose, and is readily inspected without disturbing the packing round the valve stem, by dividing the body of the valve and removing the detachable seat.

B. M. VENABLES.

Metallic thermostat. L. K. MARSHALL, Assr. to SPENCER THERMOSTAT CO. (U.S.P. 1,700,173, 29.1.29. Appl., 10.1.27. Renewed, 28.6.27).—A thermostat comprises a strip of monel metal and a strip of nickel steel welded together longitudinally by a strip of phosphor bronze.

A. R. POWELL.

Centrifuge. L. ALTPETER (U.S.P. 1,695,990, 18.12.28. Appl., 26.1.27. Ger., 30.1.26).—See B.P. 284,832; B., 1928, 320.

Acid-proof and other tanks, pipes, linings, walls, etc. H. W. FENDER, and PRODORITE, LTD. (B.P. 306,562, 22.8.27, 5.9.27, and 21.6.28).

Means for indicating quantities required for correct operation of continuous processes. G. W. BOST, and ELECTROFLO METERS CO., LTD. (B.P. 306,185, 29.8.27).

II.—FUEL; GAS; TAR; MINERAL OILS.

Assay of coal for carbonisation purposes. II. J. G. KING, C. TASKER, and L. J. EDGCOMBE (Dept. Sci. Ind. Res., Fuel Res., Tech. Paper No. 21, 1929, 34 pp.).—Further details are given of the considerations and experiments which led to the original design of the Gray-King assay (B., 1921, 205 A). The variation of the yields of coke, tar, liquor, and gas, with rate of heating, final temperature, and weight of charge, are

tabulated. The standard conditions adopted are such as to give approximately the maximum tar yield obtainable from a coal by external heating. The assay method has proved of value in the classification of coals, in the detailed examination of coal seams (cf. Fuel Res. Board Survey Papers), and as a standard for judging the tar-producing efficiency of any plant (cf. Reports of tests by the Director of Fuel Res. on low-temperature carbonisation plants). Strongly swelling coals can be satisfactorily assayed if they are first mixed with a suitable proportion of powdered electrode carbon. The proportion necessary to produce a mixture which does not swell on carbonising gives a rough measure of the "caking power" of the coal. It is not easy to correlate the yields of gas from the assay with those from the retort, two corresponding gases differing markedly in composition. In order to obtain a larger quantity of tar for examination, an assay apparatus has been designed with a capacity of 500 g. of coal. Details of both assay apparatus and notes on their manipulation are given.

A. B. MANNING.

Low-temperature carbonisation of coal. S. W. PARR (Ind. Eng. Chem., 1929, 21, 164–168).—A review of general principles of low-temperature carbonisation processes emphasising the importance of the period of heating at about 300–350° and of the final temperatures of 500° or 750°. The advantages of mid-temperature carbonisation are discussed, and operating data for a year's operation under these conditions are given in brief.

R. H. GRIFFITH.

Low-temperature carbonisation of coal from the South Muscovite basin. A. CHAKHNO (Chim. et Ind., 1929, 21, 32–34).—Two coals, known locally as "kournoi" and "boghead," were examined in a small apparatus consisting essentially of a Fischer retort holding 10–22 g. of air-dried coal, and heating was effected at such a rate that the temperature rose a few degrees per minute up to 500°. The first coal gave 7.34% of primary tar, 64.3% of semi-coke, and 8.2% of gas of high calorific value. The boghead yielded 38.47% of tar, 44.9% of coke, and 10.1% of gas; the coke was very friable and of high ash content, and the aqueous liquor produced contained only small amounts of ammonia.

R. H. GRIFFITH.

Rapid method for the determination of nitrogen in coal. J. W. WHITAKER (Fuel, 1929, 8, 145).—0.1 g. of the finely-powdered sample is weighed out into a 100 c.c. conical flask and covered with 15 c.c. of concentrated sulphuric acid. 1 g. of potassium permanganate is added very slowly (0.1 g./min.), and the mixture shaken well after each addition. The flask and contents are heated, at first gently, until the liquid boils and the solution clears. The solution is transferred to a larger flask, diluted with water, made alkaline, and steam-distilled in the usual manner. The ammonia in the distillate is determined by Nesslerising.

A. B. MANNING.

Clean coke and its value. R. A. MOTT (Fuel, 1929, 8, 123–132).—From the available figures relating to the effect of the ash content of the coke on the operation of the blast furnace it is shown that the saving in coke consumption per ton of pig for a reduction of 1%

in ash content is about 0.4 cwt., whilst the total value of the 1% reduction to the blast-furnace operator amounts to at least 6d. per ton of coke. By suitable choice of plant slurry can be efficiently cleaned, with, for example, a 10% reduction in ash content, for a cost of about 4d. per ton. By coking cleaned slurry the output and smoothness of operation of the coke oven are improved, the mechanical strength of the coke is increased, and the cost of transport of the coke is decreased.

A. B. MANNING.

Some forms of carbon and their reactivity. R. V. WHEELER (Chaleur et Ind., 1928; Fuel, 1929, 8, 121–122).—Either the glistening or the dull variety of carbon can be produced by the thermal decomposition of methane or of ethylene, depending on the character of the heated surface over which the gas is passed, the rate of passage, and the temperature. There is little difference between the reactivities of the two varieties as measured by the rate of reduction of carbon dioxide at 1000°, though both are much less reactive than either natural or artificial graphite. It seems inappropriate, therefore, to characterise as "graphitic" the skin of carbon formed on the surface or in the pores of coke by the thermal decomposition of hydrocarbons.

A. B. MANNING.

Formation and origin of acetic acid and of other volatile organic components of pyroligneous liquor.

C. PADOVANI and J. BURRAI (Chim. et Ind., 1929, 21, 20–31, and Annali Chim. Appl., 1929, 19, 3–23).—The production of acetic acid from wood-like substances may be achieved by hydrolysis or by destructive distillation, and a study has been made of the behaviour of a Libyan esparto under these conditions; the material contained 45.52% of cellulose, 21.58% of lignin, 9.6% of moisture, and small amounts of ash and fats. On distillation it gave 4.42% of acetic acid, 0.0269% of methyl alcohol, and 29.78% of charcoal. Hydrolysis was then carried out on 30 g. with about 200 g. of cold alkali solutions of varying concentration, under 5 mm. pressure. Part of the filtrate was evaporated to small bulk, and the volatile acids in it were removed by distillation in the presence of phosphoric acid and titrated with 0.1N-caustic soda. Treatment for 22 hrs. with 1–7.5% alkali resulted in a loss in weight of 23.7–46.64% and yielded 3.32–4.91% of acetic acid, the maximum production of acid occurring with 2.5% alkali. Extraction with water at 170° for 2½ hrs. produced almost the same results, and the use of alkali at 170° increased the yield of acid to 8.6%. On destructive distillation of the residues from treatment with cold alkali it was found that the amount of acid thus obtainable was about one fourth of that originally separated; the total yield was always higher than that obtained from the esparto grass by distillation only. The formation of acetic acid thus occurs first by saponification of acetyl groups from non-cellulosic substances, while lignin is simultaneously attacked, and finally the cellulose is involved in the reaction. Hydrolysis with 2.5% sulphuric acid gave higher yields than with alkali; this appears to be due to attack of cellulose as well as lignin. The relation of these results to those of previous workers is discussed.

R. H. GRIFFITH.

Improvement in method of benzol recovery from coke-oven gas. M. MINOT (*Chim. et Ind.*, 1929, 21, 252).—It is proposed to remove impurities, including water, from the circulating oil by centrifuging, and, using clean, water-free oil, to distil the benzolised oil at 105–110° instead of at 120°. Loss of wash-oil in the crude benzol, and polymerisation of the wash-oil, will be greatly reduced.

C. IRWIN.

Thermal characteristics and heat balance of a large oil-gas generator. R. D. PIKE and G. H. WEST (*Ind. Eng. Chem.*, 1929, 21, 104–109).—A 5-day test was carried out on a Jones oil-gas generator working on a cycle of 10 min. make, during which oil and steam are injected, 5 min. blowing with air to remove carbon, and 5 min. heating with air and oil used simultaneously. The air was measured by a Venturi meter checked by Pitot tubes; oil was determined by meter calibrated against storage records, and steam consumption was calculated from the moisture content of the waste gas and from boiler-room records. Temperatures were measured with bare iron-constantan couples protected by a screen to lessen radiation. Full operating data are shown, of which the most important are as follows: oil used 56.7 gals. per cycle, temperature of waste gas 784–797°, temperature of made gas 800°, tar yield 2.5 lb. per 1000 cub. ft. Calculations are given for gas made, oil and steam used, waste gas produced, and air required at different stages of the cycle, and balances are based on the figures obtained. The weight balance is divided into two periods and expressed in terms of elements and of actual materials; it is shown that in the gas-making period 48.69 lb. of oil and 38.12 lb. of steam produce 1000 cub. ft. of gas, 13.58 lb. of lampblack, and 24.62 lb. of condensed steam. In the heating and blowing period 7.25 lb. of oil and 198.95 lb. of air are required per 1000 cub. ft. of gas. The heat balance for the complete cycle shows 95.13% of the input to be due to oil and 4.5% to steam; 53% is recovered in the purified gas, 18.5% in lampblack, and 3.28% as tar. The greatest loss is in the sensible heat of the waste gases, which accounts for 7.53%, whilst a total of 6.29% has not been traced. The overall efficiency of the plant could be raised by using tar and lampblack as boiler fuel.

R. H. GRIFFITH.

Organic sulphur content of coke-oven gas from coals of widely varying sulphur content. O. P. BRYSCH and J. F. BYRNE (*Proc. Amer. Gas Assoc.*, 1927, 1463–1471).—In five of seven samples the organic sulphur content of the gas was approximately proportional to the percentage of sulphur in the coal. Pocahontas coal accelerates the evolution of organic sulphur from the coals rich in sulphur. The distribution of sulphur in the distillation products of coal mixtures was determined.

CHEMICAL ABSTRACTS.

Removal of organic sulphur from gas. N. F. PRINCE (*Proc. Amer. Gas Assoc.*, 1927, 1441–1452).—In laboratory experiments gas was scrubbed with aqueous aniline; aniline and aqueous sodium hydroxide; aniline, aqueous sodium hydroxide, and sulphur; gas-oil; aniline, sodium hydroxide, sulphur, gas oil, and iodine.

CHEMICAL ABSTRACTS.

Effect of indene on the determination of naph-

thalene [in gas] with picric acid. W. H. FULWEILER, C. W. JORDAN, and A. L. WARD (*Proc. Amer. Gas Assoc.*, 1927, 1418–1421).—Brown and Berger's method with pure naphthalene gives results 8% too high; the error due to indene is smaller than was found by these authors. In the concentrations present in towns' gas indene is not precipitated by picric acid. In presence of a large amount of naphthalene picrate unsaturated hydrocarbons are adsorbed or may react. The bridge method for the determination of naphthalene (more than 2 grains per 100 cub. ft.) is not affected by the presence of up to 50 grains of indene per 100 cub. ft.

CHEMICAL ABSTRACTS.

Determination of naphthalene [in gas] by picric acid. A. R. POWELL (*Proc. Amer. Gas Assoc.*, 1927, 1421–1430).—The sample (20–25 cub. ft.) is passed in 4–5 hrs. through 5% sulphuric acid and a trap into 0.05*N*-picric acid (four bottles); the sample line, sulphuric acid, trap, and first picric acid bottle are kept warm. The picric acid solution is diluted to 500 c.c., filtered, and an aliquot part titrated with 0.1*N*-sodium hydroxide. If sulphur dioxide is present it must be removed in dilute alkali solution; hydrogen sulphide and carbon dioxide do not interfere when methyl-red is used as indicator.

CHEMICAL ABSTRACTS.

Determination of naphthalene [in gas]. K. N. CUNDALL (*Gas Age Rec.*, 1928, 62, 393–394, 402).—The naphthalene is removed as picrate, which is washed with 0.2% picric acid solution, dried with suction, and treated with excess of an aqueous solution containing potassium iodide (376 g./litre) and iodate (48 g./litre), the iodine liberated being titrated with thiosulphate. The picric acid retained in the precipitate and filter paper is determined in a blank experiment.

CHEMICAL ABSTRACTS.

Evaluation of gas oils. T. A. MIGHILL (*Proc. Amer. Gas Assoc.*, 1927, 1454–1463).—The characteristic efficiencies of pyrogenic decomposition at any fixed temperature of hydrocarbon series, where the efficiency is the amount of heat of combustion added permanently to the gas from the thermal decomposition of a unit volume of gas oil divided by the heat of combustion of a unit volume of the oil, have been determined as follows: paraffins (arbitrary) 100, naphthenes 70, aromatics 50, unsaturateds 25%. Methods for the determination of the three last-named series of hydrocarbons are described.

CHEMICAL ABSTRACTS.

Determination of hydrogen and methane in the Orsat apparatus. H. A. BAHR (*Arch. Eisenhüttenw.*, 1928–9, 2, 495–502; *Stahl u. Eisen*, 1929, 49, 285–286).—For the determination of hydrogen and methane the Orsat apparatus should be modified by using as electrodes in the glowing-wire pipette two tubes with expanded upper ends filled with mercury covered with a saturated sodium chloride solution, and by using a saturated sodium chloride solution saturated with the products of combustion as the confining liquid instead of mercury. In case the gases contain ethane, hydrogen should be determined by passing the gas mixture over copper oxide at 300° before carrying out the usual combustion of methane in a quartz tube heated to redness.

A. R. POWELL.

Examination of alcohol motor fuels. K. R. DIETRICH and H. JEGLINSKI (Chem.-Ztg., 1929, 53, 177—178, 198—199).—The most usual diluents for alcohol fuels are benzine, benzene, or ether; the alcohols are removed by treatment with water. The residue after this washing is benzene if it has d^{15} 0.87—0.88, whilst if it is lower a more detailed inspection is necessary, and this may conveniently take the form of the Dietrich or Formánek colour test (cf. B., 1928, 435). The presence of alcohols is more certainly detected by the use of calcium chloride solution or by the addition of a dye which is soluble only in alcohols; ethyl alcohol is identified by the formation of ethyl benzoate, and methyl alcohol by oxidation and application of a modified Schiff's reagent. Ether or acetone is separated from a large volume by careful fractionation, and if acetone is present the aqueous extract of the distillate will give a red or violet coloration on the addition of sodium nitroprusside. Determinations are carried out along the same lines, and a critical review of the methods available is given. R. H. GRIFFITH.

Determination of paraffin hydrocarbons in commercial benzol and motor spirits. R. HELLING-GÖTTER (Chem.-Ztg., 1929, 53, 79).—For determining small amounts of benzine in benzol the usual methods of removing aromatic substances must be modified. By working with mixtures of known composition it was found that fuming sulphuric acid (4—5% SO_3) was the best and gave satisfactory results with mixtures containing 0.5% of paraffins. The motor spirit (25 c.c.) is violently shaken with 50 c.c. of the acid; if the temperature rises to 40—50° the shaking is stopped and the whole is cooled before allowing the sulphonation to continue. When no further warming occurs the process is complete and sulphuric acid is added to bring the contents of the vessel back to the zero mark so that the volume of the residue can be read directly. The residue should have $n_D^{15} < 1.4415$, otherwise sulphonation is incomplete. R. H. GRIFFITH.

Apparatus for benzol determination by the active carbon process. H. TRAMM (Chem. Fabr., 1929, 113—114).—A robust and reliable apparatus is described for use with solid absorbents to remove vapours such as benzol from gases in a quantitative and rapid way. It consists of a boiler, steam superheater, saturator, oven, and condenser built into a single unit with an aluminium cover. The saturator is made of brass and is filled through the lower end; it stands in a small oven which is electrically heated and controlled by an external rheostat. The boiler and superheater are also electrically heated, and the joint between charcoal tube and steam supply can be rapidly broken to allow the connexion of a stream of hot air. At the end of an experiment all moisture can be driven off in this way and the charcoal prepared for a further determination. The advantages of the apparatus are its compactness, the small amount of handling or adjustment required, and ease of exact control.

R. H. GRIFFITH.

Auto-ignition temperatures of flammable liquids. N. J. THOMPSON (Ind. Eng. Chem., 1929, 21, 134—139).—In preliminary experiments two forms of apparatus

were used; (a) made of copper, (b) a glass flask heated in a molten metal bath. In both cases temperatures were measured by means of thermocouples at the part of the apparatus nearest to the source of heat, and it was found that the copper vessel could be more evenly heated than that of glass. In making the test the fuel was dropped in quantities of about 0.01 c.c. on to the surface at such a temperature that ignition readily occurred, and the temperature was then allowed to fall slowly until firing just ceased to take place; in this way the lowest point was determined at which ignition took place under any conditions. The copper block apparatus gave unreliable results owing to reduction of oxide, and observations made with a chromium-plated copper vessel were not considered satisfactory, although they were more consistent. Results with steel apparatus were similar to those with the chromium-copper surface. The effect of size and shape of the cavity was studied with glass flasks, but the small differences found could be accounted for by lack of uniformity in the quality of the glass; similar experiments with steel surfaces showed that change of shape was not important, but that the size of the opening to the air affected the results, lower readings being obtained with a smaller hole. Suggestions are made for the adoption of a standard method and apparatus for testing ignition points. The effect of addition of benzol to gasoline is to raise the ignition point of the mixture. R. H. GRIFFITH.

Concentration of acetic acid. GUINOT.—See III.
Flash points of solvents and plasticisers. MÜHLENDAHL.—See XIII.
Automobile exhaust gas in streets and shops. BLOOMFIELD and ISBELL.—See XXIII.

PATENTS.

Acceleration of flowing characteristics of coal. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 299,718, 12.9.27. U.S., 25.10.26. Cf. B.P. 277,660; B., 1929, 160).—The mobility of a stream of powdered coal which is being pumped from a source of supply to a zone of combustion is greatly increased by heating the coal to a temperature at which vapours are evolved from the particles (e.g., about 200°) and maintaining it at that temperature during transit.

A. B. MANNING.

Distillation of carbonaceous material. D. McN. RAMSAY (B.P. 305,714, 11.11.27).—Carbonaceous material is distilled at temperatures up to 550° in a current of superheated steam under reduced pressures. A water-sealed feed elevator enclosed in a barometric duct supplies the material to a conical retort which is provided with a central screw for regulating the vertical movement of the charge. The carbonised residue is discharged through a barometric duct to another water-sealed elevator. The volatile products of distillation pass through a condenser to a closed separator connected to a gas pump.

A. B. MANNING.

Distilling carbonaceous materials. F. PUENING (U.S.P. 1,698,347—9, 8.1.29. Appl., [A, B] 6.11.22, [C] 23.2.23. Renewed [A] 14.1.28).—(A) Coking receptacles are continuously circulated alternately through a heating and a coking region. After passage through the heating region the receptacles are charged with the

material, which is coked by the heat of the receptacles as they pass through the coking region. The coked material is then discharged and the cycle repeated. (b) Apparatus for distilling carbonaceous materials comprises a rotary carrier on which are mounted a number of heating wall supports. Heating walls are suspended from these supports which are movable relatively to one another. The supports are supplied with cooling channels. A vertically-movable pusher provided with side spreaders and an intermediate ram plate is so arranged that the former engage the heating wall supports in advance of the entry of the ram plate between adjacent heating walls in order to spread the walls apart on the carrier during the pushing operation. (c) Apparatus for the distillation of solid carbonaceous materials comprises a distillation chamber, a number of solid, heat-transfer bodies, means for heating the latter outside the distillation chamber, and means for introducing them into the chamber in such amount as to effect the distillation. Provision is also made for separating the carbonised material from the solid heating bodies.

A. B. MANNING.

Working of explosion turbines with coal gas. H. HOLZWARTH (B.P. 294,978, 2.6.28. Ger., 3.8.27).—Coal dust is introduced periodically by the aid of vaporising air into the combustion chamber of an intermittently acting internal-combustion turbine and ignited by means of an auxiliary gaseous fuel which is likewise periodically and indirectly or directly introduced through a gas and air mixing valve mounted in front of the inlet valve.

H. S. GARLICK.

Producing a combustible charge for use in internal-combustion engines. D. BALACHOWSKY, P. CAIRE, and M. LEVY (B.P. 302,717, 28.3.28. Fr., 21.12.27).—A mixture of air and a hydrocarbon, *e.g.*, petrol, is subjected to the action of a heated catalyst before being mixed with secondary air and admitted to the cylinder. The catalyst is formed of one of the metals which are known catalytically to decompose hydrocarbons. The temperature of the catalysed mixture is controlled by passing it through a cooler, or, if the exhaust forms the source of heat, by cooling the exhaust before its passage round the catalyst.

A. B. MANNING.

Manufacture of coke. F. PUENING (U.S.P. 1,699,448, 15.1.29. Appl., 29.5.22).—Material to be coked is charged into the spaces in a retort between parallel members therein which have been preheated to a coking temperature, and the walls of the retort are moved closer together to compress the contents. The vapours generated during coking are removed from the retort, and when coking is complete the walls are moved apart for removal of the coke.

L. A. COLES.

Production and regeneration of activated carbon. **Production of activated carbon.** N. V. ALGEM. NORIT MAATSCHAPPIJ (B.P. 279,104 and 291,725, 14.10.27. Ger., 16.10.26).—(A) The carbon to be activated is heated to incandescence by passing an electric current through the wet material, the steam produced serving as the activating agent. The material is passed continuously through one or more chambers containing suitably arranged heating electrodes. The process may be

carried out in the presence of other activating gases, *e.g.*, oxygen, chlorine, sulphur dioxide, etc., or the material may be impregnated with an electrolyte, *e.g.*, sodium chloride. Uniform heating of finely-powdered carbon is attained by mixing with it a more coarsely granular material, *e.g.*, coke, which acts as a heating resistance. (b) Highly adsorptive carbon is produced by combining the processes of activation by gas treatment, by impregnation with a non-alkaline chemical, *e.g.*, phosphoric acid, chlorides, bisulphates, peroxides, etc., and by heating to incandescence without previously washing. The heating may be effected as in (A).

A. B. MANNING.

Manufacture of liquid and other hydrocarbons and derivatives thereof by destructive hydrogenation of carbonaceous materials. I. G. FARBEN-IND. A.-G. (B.P. 275,664, 8.8.27. Ger., 7.8.26).—The materials are hydrogenated at raised temperatures and at pressures above 50 atm. in the presence of catalysts consisting of (a) carbides, *e.g.*, silicon carbide, in the absence of nitrogen or its compounds, or (b) active carbon together with an element or compound thereof of the 5th group of the periodic system, *e.g.*, alderwood charcoal heated at 800° and soaked with phosphoric acid.

H. S. GARLICK.

Manufacture of liquid and other hydrocarbons and derivatives thereof by destructive hydrogenation of carbonaceous materials. J. Y. JOHNSON. From I. G. FARBEN-IND. A.-G. (B.P. 295,947—9, 20.8.28).—(A) Carbonaceous materials are treated with a large excess of hydrogen at elevated temperatures and under pressures of at least 50 atm. in the presence of catalysts comprising metals of the 5th and 7th periodic group, or their compounds, together with aluminium or its compounds. Other catalysts for the same purpose are (b) uranium or its compounds, together with elements of the second periodic group or their compounds, or (c) together with aluminium or its compounds. A. B. MANNING.

Manufacture of liquid and other hydrocarbons and derivatives thereof by destructive hydrogenation of carbonaceous materials. I. G. FARBEN-IND. A.-G. (B.P. 299,020—1, 8.8.27. Ger., 7.8.26).—(A) The materials are treated with a large excess of hydrogen, or gases containing or giving off hydrogen, at elevated temperatures and under high pressures, in the presence of catalysts containing boron, phosphorus, arsenic, selenium, or tellurium, particularly in conjunction with metals of the 6th periodic group. These catalysts may be formed, for example, by impregnating alderwood charcoal with phosphoric acid, calcium, molybdenum, or tungsten phosphates, titanium or iron borides, etc. (b) Catalysts for the same purpose may contain halogens in the free or combined state, *e.g.*, calcium fluoride, molybdenum trioxide, to which has been added 10% of aluminium chloride etc. Both series of catalysts are immune to sulphur poisoning.

A. B. MANNING.

Treatment of solid carbonaceous material for the recovery of oils. H. A. HUMPHREY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 305,744, 30.11.27).—The finely-divided material is distilled in a liquid medium, being allowed to sink through an oil of high b.p. contained

in a tower, wherein it encounters progressively higher temperatures as it falls. Residual solid material and oil are removed at the base of the tower, gases and vapours at the top. The tower is preferably provided with suitable baffles for increasing the time of contact of the material with the heating medium. To ensure uniform flow of the solid material the oil may be caused to surge up and down periodically; a U-shaped tower may be used and the pressure alternately released and allowed to build up at the head of each column, or a piston may be reciprocated at the base of the U. Steam, hydrogen, or other gases may be passed up through the column; the steam and hydrogen may be preheated and then serve to supply the heat required for the process. The process may be carried out under any desired pressure from atmospheric up to those used in the destructive hydrogenation of carbonaceous materials.

A. B. MANNING.

Operation of gas producers. C. CARPENTER, and SOUTH METROPOLITAN GAS CO. (B.P. 305,764, 16.12.27).—The waste of fuel during the clinkering of a gas producer is reduced by replacing the air just before the clinkering period by a blast of a gas which is inert or which reacts endothermically with the fuel, so that the temperature of the latter is lowered. The blast of inert gas may be maintained during the clinkering period. At the same time, if desired, a current of such gas may be passed through the space above the fuel bed.

A. B. MANNING.

Production of ammonium sulphate [from coal gas]. SOC. ANON. DES FOURS À COKE SEMET-SOLVAY & PIETTE (B.P. 292,995, 29.5.28. Belg., 29.6.27. Addn. to B.P. 262,320; B., 1927, 107).—The prior process is applied to the production of ammonium sulphate from combustion gases from ovens and furnaces, or other gases containing carbon dioxide. Before removal of the acid constituents the gases are cooled and freed from dust.

A. B. MANNING.

Extraction of ammonia from gases, more particularly from coke-oven gas. GES. F. LINDE'S EISMACHINEN A.-G. (B.P. 281,288, 21.11.27. Ger., 27.11.26).—The gas, from which the tar and most of the water have been removed by cooling to about 40°, is compressed and, after again cooling to 50° and separating the naphthalene in liquid form, is further cooled to 15° and then washed with the dilute ammonia water, which condenses on compression. The cooling is continued to about 0° and the gas then further washed with a small quantity of pure water. The ammonia, carbon dioxide, and hydrogen sulphide are driven off from the concentrated solutions of ammonia which are obtained in this process, by utilising the heat produced in the compression of the gas. The cooling during washing is effected by means of the cold, dry, expanded gas from which the ammonia has been removed. If desired, the gas before expansion may be further cooled to -20° in order to separate the benzene (cf. B.P. 275,633; B., 1929, 160).

A. B. MANNING.

Methods of obtaining bodies of the o-di[hydr]oxybenzene series, and treatment of ammonia liquor and like liquors. H. W. ROBINSON and D. W. PARKES (B.P. 305,494, 5.8. and 23.12.27, 17.2. and 5.6.28).—Ammonia liquor, freed from organic impurities and

sulphides and carbonates, e.g., by distillation, or by precipitation with a mineral acid or with bleaching powder and lime, is then treated with a reagent, e.g., lead chloride or acetate, which will precipitate the pyrocatechol, and is finally treated with activated carbon for the removal of tar acids. The precipitate is treated with hydrochloric acid and the pyrocatechol recovered from the solution by distillation or extraction.

A. B. MANNING.

Treatment of coke-oven gas, coal gas, or similar gases, particularly for the purpose of obtaining acetylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,008, 13.10.27).—The efficiency of the production of acetylene from such gases by the action of an electric arc is greatly increased if the carbon monoxide is first removed from the gas, e.g., by catalytic conversion into methane, or by interaction with steam and removal of the resulting carbon dioxide. The waste heat in the gases issuing from the electric arc is used to preheat the initial gas in order to effect this conversion.

A. B. MANNING.

Purifying gases from sulphuretted hydrogen by decomposing the latter into hydrogen and sulphur. F. FISCHER (B.P. 289,885, 30.4.28. Ger., 5.5.27).—The gases are washed with an alkaline solution of potassium ferricyanide, and the spent solution, after separation of the sulphur produced, is regenerated by oxidation in the anode space of an electrolytic cell. The alkali produced in the cathode space is added to the solution, whilst the hydrogen evolved during electrolysis is added to the purified gas.

A. B. MANNING.

Removal of sulphur compounds from gas mixtures. SILICA GEL CORP., Assees. of E. B. MILLER and G. C. CONNOLLY (B.P. 280,947, 18.11.27. U.S., 19.11.26).—Hydrogen sulphide is removed from gases by passing them, admixed with the amount of air necessary for the reaction $2\text{H}_2\text{S} + \text{O}_2 = 2\text{H}_2\text{O} + 2\text{S}$, over a highly porous adsorbent solid impregnated with a metallic oxide, e.g., silica gel, impregnated with about 2.5% of ferric oxide. The oxidation is preferably carried out at 185–210°; the gases may be passed through two beds of the material in series, the first being at a slightly higher temperature than the second, or the finely-pulverised material may be fed into a stream of the gas through which it falls in counter-flow. The material is reactivated by being heated at about 500° in a current of air. Organic sulphur compounds may be removed from the gas by treatment with the same catalyst in two stages, first at 450°, whereby the compounds are converted into hydrogen sulphide, which is then removed as described above.

A. B. MANNING.

Purification of combustible gas. F. SIEMENS A.-G. (B.P. 276,347, 18.8.27. Austr., 19.8.26).—The gas is passed over at least two catalysts in series in order to oxidise the sulphur compounds to sulphur dioxide or trioxide, and hydrolyse the cyanogen compounds to ammonia and oxides of carbon. Metals of the iron group, particularly nickel and cobalt, or metals such as copper, manganese, lead, zinc, bismuth, or antimony, or their oxides or salts, are used as catalysts. The reaction temperature used lies preferably between 300° and 400°. If necessary, air and water vapour are added

to the gases. If desired, the gas may finally be passed over a nickel catalyst to hydrogenate the unsaturated hydrocarbons.

A. B. MANNING.

Manufacture of [bituminous] emulsions and emulsifiers for producing the same. E. C. R. MARKS. From ROL LISTER & CIE. (B.P. 305,742, 26.11.27).—A mixture of resin and maize starch, with or without a fatty material, is heated with dilute sodium hydroxide solution at 50–96° until the mixture becomes homogeneous. The resultant liquid is suitable for emulsifying tar, petroleum, asphalt, etc.

A. R. POWELL.

Bituminous emulsions. J. A. MONTGOMERIE (B.P. 305,716, 11.11.27).—The wetting, penetrating, and spreading power of aqueous emulsions of bitumen is increased by the addition of a small quantity, *e.g.*, 0.15% of the weight of bitumen, of a salt of lauric acid.

A. B. MANNING.

Pressure distillation of hydrocarbon oils. F. M. ROGERS and E. J. SHAEFFER, ASSRS. to STANDARD OIL CO. (U.S.P. 1,697,573, 1.1.29. Appl., 17.11.22. Can., 26.5.24).—Hydrocarbon oil, b.p. 230–400°, is heated under 5–6 atm. and the vapours are passed into a dephlegmator at the upper end of which gasoline distillate of approximately the characteristics of the desired distillate is introduced. The phlegms are returned to the still. The pressure may be relieved between the still and dephlegmator or between dephlegmator and condenser if desired.

R. BRIGHTMAN.

Continuous process for the refining [cracking] of oils. E. T. HESSLE (U.S.P. 1,696,913, 1.1.29. Appl., 31.3.26).—Hydrocarbon oil, *e.g.*, gas oil, is passed through a heating coil at 200–350°/3–8 atm. and discharged into the reaction vessel (at 250–400°) through a valve-regulated nozzle in opposition to a similar nozzle discharging residual or tail gases from the process. Hydrogen, illuminating gas, water-gas, or other gas containing hydrogen is used at the commencement of the process. The greyish-yellow oil fog thus produced is forced through a molten decomposition catalyst consisting of a tin-antimony alloy (<10% Sb) at 250–240°. The decomposition vapours containing hydrogen are passed successively through a desulphurising tower, containing, *e.g.*, iron and manganese oxides, iron and steel turnings, and a catalytic tower packed with iron, copper, and nickel turnings, respectively, the condensate in each tower being discharged to run-down tanks. From the final condenser uncondensed gases are led to a compressor, *e.g.*, at 15 atm.; the uncondensed tail gas is returned to the reaction vessel through a preheater.

R. BRIGHTMAN.

Increasing the viscosity and improving the lubricating properties of oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,955, 12.9.27).—The addition of less than 1% of montan wax bleached by oxidation (cf. B.P. 289,621; B., 1928, 473) to mineral and fatty oils considerably increases their viscosity; addition of at least 4% gives a product having the consistency of petroleum jelly.

H. S. GARLICK.

Means for hardening paraffins, waxes, ozokerite, stearine, tallow, etc. J. Y. JOHNSON. From I. G.

FARBENIND. A.-G. (B.P. 306,053, 15.9.27).—The addition to natural fats and waxes of up to 10% of montan wax bleached by oxidation gives a considerable increase in their m.p.

H. S. GARLICK.

Vertical coking oven. J. LÜTZ (U.S.P. 1,702,699, 19.2.29. Appl., 21.8.21. Ger., 21.6.16).—See G.P. 307,987; B., 1918, 760 A.

Coking retort oven. J. BECKER, ASSR. to KOPPERS Co. (Re-issue 17,224, 26.2.29, of U.S.P. 1,556,749, 13.10.25).—See B., 1925, 950.

Purification of coke-oven gases etc. G. CLAUDE, ASSR. to LAZOTE, INC. (U.S.P. 1,702,683, 19.2.29. Appl., 12.3.23. Fr., 30.3.22).—See B.P. 195,598; B., 1924, 123.

Distillation and depolymerisation of liquid or liquefiable hydrocarbons. A. A. F. M. SEIGLE (U.S.P. 1,702,275, 19.2.29. Appl., 19.12.22. Fr., 22.12.21).—See B.P. 190,723; B., 1924, 459.

Determination of the amount of carbonic acid contained in flue gases by the thermal conductivity method. SIEMENS & HALSKE A.-G. (B.P. 279,478, 20.10.27. Ger., 25.10.26).—See U.S.P. 1,701,181; B., 1929, 290.

Device for washing coal or other minerals. PRÉPARATION INDUSTRIELLE DES COMBUSTIBLES SOC. ANON. (B.P. 287,884, 28.3.28. Fr., 28.3.27).

[Boiler] furnaces adapted for burning pulverised solid and liquid fuel. TODD DRY DOCK ENGINEERING & REPAIR CORP. (B.P. 295,352, 28.6.28. U.S., 10.8.27).

[Protective lintel for doorways of] producer and like furnaces. DRAKES, LTD., and J. W. DRAKE (B.P. 305,695, 9.11.27).

Coal-oil burners. J. B. MORROW (B.P. 306,628, 2.12.27).

Carbonisation plant (B.P. 303,572). Filters for oil and petrol (B.P. 287,932).—See I. Aromatic hydrocarbons (B.P. 281,298).—See III.

III.—ORGANIC INTERMEDIATES.

Extraction and concentration of acetic acid from dilute aqueous solutions. H. GUINOT (Chim. et Ind., 1929, 21, 243–251).—In the manufacture of cellulose acetate it is necessary to recover the acetic acid from a solution of 15–45% concentration contaminated with organic matter and sodium sulphate. Direct distillation involves an excessive steam consumption, and the impurities cause trouble. Various extraction methods with light solvents have been proposed, but the volume of liquid to be evaporated is reduced by the Suida process employing cresol, from which the acetic acid is distilled. A preliminary concentration is necessary. The process here described employs solvents such as butyl acetate of b.p. close to that of acetic acid. Curves are given showing the concentration of acetic acid in the solvent relative to that in the aqueous solution, which is independent of the volume. For such extraction columns are unsuitable, and a series of agitating and settling tanks working in counter-current is suggested. A graphic method is developed for calculating the number of units necessary for a given extraction. A semi-

works-scale plant on these lines consists of 10 units treating 70 litres per hr. of 35% acetic acid. The aqueous solution is elevated by ejection with nitrogen to reduce the corrosion of copper. The extracted water contains a little of the solvent. This is easily removed, as a constant-b.p. mixture, by evaporation. The separation of the solvent and the acetic acid is effected by the use of a petroleum distillate of b.p. 114°, which forms with acetic acid a homogeneous mixture containing 45% of acid and of b.p. 110°. This is contained in a plate column into the middle part of which the acetic extract is injected. The acetic acid-paraffin mixture is broken into its components by the least trace of water, and if distillation were carried out directly a distillate consisting of an upper layer of paraffin and a lower layer of 75% acetic acid would be obtained. The still is, however, worked so that a constant-b.p. mixture of water and paraffin (b.p. 86°) is produced at the top, and a little lower down a liquid is extracted which separates into paraffin and 90–97% acetic acid. The acetic acid is passed through a small subsidiary column and is recovered in the pure state (m.p. 16.5°). The upper (paraffin) layer is returned to the column. The effluent from the bottom consists of the extracting liquid, amyl acetate, and the like. The steam consumption for this process amounts to 3.5–4.0 kg. of steam per kg. of acid concentrated from 30% with a 97–98% yield. The method may be applied to pyroligneous acid if the latter is first freed from tar with butylresol containing a little toluene.

C. IRWIN.

Pyroligneous liquor. PADOVANI and BURRAI.—See II. Flash points of solvents etc. MÜHLENDAHL.—See XIII.

PATENTS.

Manufacture of oxidation products of hydrocarbons. F. J. CARMAN (U.S.P. 1,697,105, 1.1.29. Appl., 6.8.23).—A mixture of, *e.g.*, 4 pts. by vol. of methane, 1 pt. of oxygen, and 1 pt. of methyl chloride, or its equivalent in methane or available chlorine, is passed over barium chloride or other non-volatile bivalent halide as catalyst at about 480°. About 8–10% of the methyl chloride is oxidised to formaldehyde, which is removed by cooling and scrubbing with water. The residual gas is dried, the carbon monoxide oxidised in presence of a catalyst, and, after absorption of carbon dioxide, is returned to the mixing chamber, where fresh methane, oxygen, and chlorine are admitted to restore the original proportions by volume. Hydrogen chloride liberated by oxidation of the methyl chloride is mainly oxidised to chlorine, from which methyl chloride is regenerated by reaction with the excess of methane.

R. BRIGHTMAN.

Manufacture of methyl alcohol and [catalytic] agents for use therein. BRIT. CELANESE, LTD., W. BADER, and S. J. GREEN (B.P. 300,142, 2.5.27).—Zinc oxide catalyst for use in the synthesis of methyl alcohol from mixtures of hydrogen and oxides of carbon is prepared by precipitating zinc carbonate gel in highly colloidal and gelatinous form, *e.g.*, in 1% solution and in presence of a protective colloid. The precipitate is washed from electrolytes, mixed with a jelly of a pro-

TECTIVE colloid, if such colloid was not previously added, and dried at 60° and finally at 250–400°.

R. BRIGHTMAN.

Manufacture of ethyl alcohol gels. H. OHLE and J. OTHMAN-NEUSCHELLER (B.P. 281,662, 29.11.27. Ger., 30.11.26).—The hydrogen sulphates of diacetone-glucose, -galactose, or -lævulose, or of acetoneglycerol are used as congealing agents. An aqueous solution of a soluble (potassium) salt of one of these (particularly α -diacetone-lævulose) at 100° is mixed with about 50 vols. of ethyl alcohol; on keeping for 15 min. the whole sets to a gel.

C. HOLLINS.

Manufacture of acetaldehyde and acetic acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,855, 2.11.27).—Acetylene is passed with hydrogen and oxidising gases (air), the latter being added in stages, over a catalyst having both oxidising and hydrating properties. For acetaldehyde, salts of mercury or vanadium, *e.g.*, mercuric phosphate, at 120° are used; for acetic acid, salts (especially vanadates) of tin, silver, or mercury at 200–250° are employed.

C. HOLLINS.

Manufacture of acetal. SOC. ANON. DES DISTILLERIES DES DEUX SÈVRES (B.P. 283,112, 29.12.27. Belg., 3.1.27).—Alcohol and acetaldehyde are condensed in presence of a catalyst and the mixture is neutralised, *e.g.*, with sodium hydrogen carbonate. Unchanged acetaldehyde and part of the alcohol are removed by distillation. The residue of acetal, water, and alcohol is treated with an auxiliary liquid insoluble in water, *e.g.*, benzene, and the ternary azeotropic mixture is distilled off, leaving acetal.

R. BRIGHTMAN.

Manufacture of isopropyl acetate. C. ARNOLD. From STANDARD OIL DEVELOPMENT Co. (B.P. 300,418, 24.12.27).—Commercial isopropyl alcohol, containing, *e.g.*, 10% of water and a little butyl alcohol, is distilled with 100–200% excess of glacial acetic acid and a catalyst, *e.g.*, 0.2–3.0% of sulphuric acid at 75–5° (still-head temperature), the isopropyl acetate which comes over being separated from the aqueous layer. Yields of 65–80% are obtained before the concentration of acetic acid falls to 65% and distillation is stopped.

R. BRIGHTMAN.

Production of butyrene [dipropyl ketone]. F. GERMAIN, Assee. of H. DOLTER (B.P. 277,975, 21.9.27. Ger., 23.9.26).—Calcium butyrate is heated directly by hot inert gases in a rotating drum containing heavy balls.

C. HOLLINS.

Production of hydroxyalkylamines [“triethanolamine” etc.]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,563, 24.8.27).—Improved yields of mono- or tri-(β -hydroxyethyl)amines, *e.g.*, “triethanolamine,” are obtained from ethylene oxide added slowly to aqueous ammonia at 10° and 25–30°, respectively, the temperature being carefully regulated. Propylene oxide and ammonia at 5–10° similarly give β -hydroxypropylamine.

C. HOLLINS.

Production of sulphonic acids [of unsaturated aliphatic or alicyclic hydrocarbons]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,052, 14.9.27).—Olefines and their derivatives and unsaturated hydroaromatic compounds yield true C-sulphonic acids when treated with chlorosulphonic acid or oleum in presence

of ether (or other substance capable of forming an additive product with the sulphonating agent), or with alkyl chlorosulphonates. The products are chloro- or hydroxy-sulphonic acids according as water or alkali is used in working up. With hydroxylated unsaturated aliphatic acids excess of the sulphonating agent is necessary. Examples are oleic acid, tetrahydrobenzene, rubber, and balata.

C. HOLLINS.

Manufacture of aromatic hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 281,298, 23.11.27. Ger., 24.11.26).—Heavy middle oil, d 0.900–0.910, obtained, e.g., by hydrogenation of coal under pressure, or distilling mineral oils, is sprayed at 200 atm. into a vessel at 580° in presence of 20–30 vols. excess of hydrogen. The vessel may be partly heated by a spiral, e.g., of chrome-nickel B, which serves as catalyst, or an intimate mixture of oxides of chromium, molybdenum, or manganese may be used. The mixture leaving the reaction chamber is cooled under pressure, giving a liquid containing 60–85% of benzene, 5–15% of naphthalene and homologues of benzene, and traces of anthracene. The residual gases are stripped of gaseous hydrocarbons such as methane by washing with, e.g., benzene, and are re-circulated. By increasing the partial pressure of the oil a liquid containing 30–40% of benzene, 40% of naphthalene, and 5% of anthracene is obtained. Similarly, paraffin wax in presence of chromium oxide catalyst and 25 vols. of hydrogen affords a liquid containing 60% of benzenes. The apparatus is preferably made from Krupp's V2A or WT2 steel.

R. BRIGHTMAN.

Manufacture of condensation products from crude cresol and aliphatic ketones. SCHERING-KAHLBAUM A.-G. (B.P. 279,856, 25.10.27. Ger., 26.10.26. Addn. to B.P. 273,684; B., 1929, 236).—Crude cresol is substituted for *m*- or *p*-cresol in the process of the prior patent.

C. HOLLINS.

Manufacture of alkylisoalkylenephenols [isopropenylcresols] and alkylated coumarans. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), and H. JORDAN (B.P. 306,051, 15.8.27. Addn. to B.P. 273,686; B., 1929, 236).—The condensation product from acetone and *m*- or *p*-cresol is heated in presence of a catalyst, e.g., 0.1% of a metal phenoxide preferably derived from the phenol producible by the reaction, or a nickel catalyst and/or silica gel or other porous material.

C. HOLLINS.

Manufacture of decomposition compounds from condensation products of *m*- and *p*-cresols with acetone. SCHERING-KAHLBAUM A.-G. (B.P. 279,857, 25.10.27. Ger., 26.10.26).—In the process of B.P. 306,051 (preceding) the ether-like condensation products from *m*- and *p*-cresols with acetone may be used as starting-materials.

C. HOLLINS.

Manufacture of *o*-aminoarylmercaptans [*o*-aminothiophenols]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 306,590, 23.11.27).—2-Amino-benz- or -naphtha-thiazoles are heated with concentrated aqueous or alcoholic sodium or potassium hydroxide. 6-Chloro-2-amino-4-methylbenzthiazole yields 5-chloro-2-amino-*m*-thiocresol; 5-ethoxy-2-aminothiophenol, 1-amino-4-ethoxy- β -thionaphthol, and 1-amino-4-methoxy- β -thio-

naphthol are similarly obtained. The thiols are converted directly into thioglycollic acids; the lactams of 1-amino-4-ethoxy- and -methoxy- β -naphthylthioglycollic acids have m.p. 227–228° and 226–227°, respectively.

C. HOLLINS.

Manufacture of diacidyl derivatives of naphthalene and acenaphthene. I. G. FARBENIND. A.-G. (B.P. 291,347, 18.11.27. Addn. to B.P. 279,506; B., 1928, 440).—The products of the prior patent are obtainable from the hydrocarbons by the action of excess of the acid chloride.

C. HOLLINS.

Manufacture of *o*-cyanoarylthioglycollic acids and intermediate products. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 306,575 and 306,607, 18.11.27).—(A) *o*-Thiocyanoarylamines carrying a *p*-substituent are diazotised and converted by Sandmeyer's method into *o*-thiocyanonitriles, from which *o*-cyanothioglycollic acids are obtained by successive treatment with alkali and chloroacetic acid. The following compounds are described: 5-chloro-3-thiocyano-*o*-tolunitrile, m.p. 86°, the 3-thiol derivative, m.p. 86°, and the 3-thioglycollic acid, m.p. 116°; 4-ethoxy-2-thiocyanobenzonitrile, m.p. 112–115°, the 2-thiol derivative and 2-thioglycollic acid; 1-thiocyano- β -naphthonitrile, m.p. 135–136°, the 1-thiol derivative, and 2-cyano- α -naphthylthioglycollic acid, m.p. 137–138°. (B) The same end-products are obtained by first converting the *o*-thiocyanoarylamines into *o*-aminoarylthioglycollic acids and then applying the Sandmeyer reaction. 5-Chloro-2-amino-*m*-tolylthioglycollic acid (lactam, m.p. 186°), 5-ethoxy-2-aminophenylthioglycollic acid, 2-amino- α -naphthylthioglycollic acid, and the thiols from which they are derived, are described.

C. HOLLINS.

New pyrazolones and dyes therefrom. IMPERIAL CHEM. INDUSTRIES, LTD., and M. MENDOZA (B.P. 300,321, 19.8.27).—New pyrazolones are obtained from the diaryl sulphones and sulphides of B.P. 297,855 and 299,501 (B., 1928, 887, 921) by the usual methods, and when coupled with diazo components, including diazo-azo and tetrazo compounds, afford azo dyes for wool or chrome printing on cotton. The methylpyrazolone derived from 2-amino-4-sulphophenyl 2-hydroxy-3-carboxy-1-naphthyl sulphone is coupled with sulphanilic acid (yellow shades on wool, slightly greened on chroming or when chrome-printed on cotton), naphthionic acid (orange), *p*-nitroaniline (yellow-brown), and 1-amino-2-naphthol-4-sulphonic acid (scarlet). The corresponding carboxy-pyrazolone is coupled with sulphanilic acid (brown), naphthionic acid, and *p*-nitroaniline (deeper shades). Similar pyrazolones, and azo dyes therefrom, are prepared from 5-chloro- and 5-methyl derivatives of 2'-amino-2-hydroxy-4'-sulpho-3-carboxydiphenyl sulphone.

R. BRIGHTMAN.

Manufacture of metallic compounds of ethylene-thiocarbamide. G. T. MORGAN and F. H. BURSTALL (B.P. 300,330, 25.8.27).—Complex salts readily soluble in water are obtained by treating a cadmium, lead, palladium, copper, or gold salt with ethylenethiocarbamide as previously described (A., 1928, 278).

R. BRIGHTMAN.

Preparations convertible into dispersions and their manufacture. J. R. GEIGY A.-G. (B.P. 272,896,

8.6.27. Ger., 18.6.26).—Water-insoluble organic dyes and intermediates, other than vat dyes or lakes, are ground with soap or saponin at ordinary temperatures until the finely-powdered mixture yields a dispersion in water. R. BRIGHTMAN.

Concentration of acetic acid. H. SUIDA (U.S.P. 1,703,020, 19.2.29. Appl., 6.7.26. Austr., 13.7.25).—See B.P. 255,047; B., 1927, 316.

Manufacture of *o*-halogenated aromatic quaternary ammonium compounds. G. KALISCHER and K. KELLER, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,703,150, 26.2.29. Appl., 8.7.26. Ger., 15.7.25).—See B.P. 286,984; B., 1928, 361.

Manufacture of alkylsorosindulinesulphonic acids. P. LÄUGER, ASSR. to J. R. GEIGY SOC. ANON. (U.S.P. 1,703,772, 26.2.29. Appl., 16.12.27. Ger., 27.12.26).—See B.P. 282,803; B., 1928, 922.

Dihydroxybenzene compounds from ammonia liquors (B.P. 305,494).—See II.

IV.—DYESTUFFS.

PATENTS.

Manufacture of new [azo direct] dyes. SOC. CHEM. IND. IN BASLE (B.P. 274,130, 11.7.27. Switz., 10.7.26).—Direct, cotton dyes with good levelling and discharging properties are obtained by phosgenation (etc.) of aminoazo dyes formed by coupling a diazotised 1 : 8-aminonaphtholsulphonic acid with a *para*-coupling amine of the benzene series, or by phosgenating (etc.) an equimolecular mixture of such an aminoazo dye and any other amino-azo or -polyazo dye. Alternatively, a suitable amine may be used in place of one of the aminoazo dyes and afterwards converted into an azo dye. The 1 : 8-aminonaphtholsulphonic acids are diazotised in the form of their *o*-*p*-toluenesulphonates, the *o*-acyl group being afterwards removed by hydrolysis. Examples are : H-acid \rightarrow cresidine, phosgenated (blue-red); H-acid \rightarrow cresidine, phosgenated, with *p*-phenylenediaminesulphonic acid \rightarrow γ -acid, coupled acid (blue-red); H-acid \rightarrow cresidine, phosgenated, with *p*-phenylenediamine \rightarrow salicylic acid (orange-red, increased light-fastness when coppered). R. BRIGHTMAN.

Manufacture of [mordant] azo dyes. DURAND & HUGUENIN A.-G. (B.P. 283,482, 5.1.28. Ger., 10.1.27).—Derivatives of 4-aminoazobenzene, containing in the amine nucleus a carboxyl or sulphonic group and in the other nucleus a salicylic acid grouping, are diazotised and stirred with alkali or boiled with acid to destroy the diazo group. The products are chrome-printing browns. Examples are : *p*-nitroaniline-*o*-sulphonic acid \rightarrow salicylic or *m*-cresotic acid, reduced; anthranilic acid \rightarrow salicylic acid, nitrated and reduced. C. HOLLINS.

Manufacture of complex metal compounds of *o*-hydroxyazo dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,732, 21.3.28).—The chromium or copper compounds of azo dyes obtained by coupling a diazotised *o*-aminophenol, containing nitro- and/or chloro-substituents, with nitro-, halogeno-, or alkyl derivatives of *m*-phenylenediamine are useful dyes for leather. Examples are : 4-nitro-*o*-amino-

phenol-6-sulphonic acid \rightarrow 4-chloro-*m*-phenylenediamine or *m*-tolylene-diamine. [Stat. ref.]

C. HOLLINS.

Manufacture of [azo] dyes containing chromium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,425, 11.1.28).—Azo dyes obtained by coupling *o*-hydroxydiazocompounds with an acetoacetic ester or arylamide are heated in aqueous solution under pressure in the absence of a water-soluble organic solvent with a chromium compound in proportion to provide at least one chromium atom for each chromable group in the dye molecule; e.g., the dye from 4-nitro-2-aminophenol-6-sulphonic acid and acetoacetanilide with chromium oxide and aqueous formic acid at 130° for 2 hrs. yields a complex chromium compound dyeing wool in yellow shades of good fastness to washing, milling, and light.

R. BRIGHTMAN.

Manufacture of [azo] dyes. SOC. CHEM. IND. IN BASLE (B.P. 282,782, 24.12.27. Switz., 24.12.26).—*o*-Nitrodiazocompounds, e.g., *o*-nitrodiazobenzene, are coupled with 3-methyl-5-pyrazolone giving yellow dyes for acetate silk of excellent fastness to light.

R. BRIGHTMAN.

Dye preparations [for acetate silk] and their application. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (B.P. 300,299, 9.8.27).—Dry solid preparations are obtained by emulsifying the dye, e.g., azo dyes, indophenols, anthraquinone derivatives, having affinity for acetate silk, with water in presence of saponin or a condensation product of formaldehyde and a naphthalenesulphonic acid. Sulphite-cellulose lye, freed from calcium and fermented, is added as protective colloid, and the dispersion is evaporated to dryness.

R. BRIGHTMAN.

Production of new coloured compounds and of valuable colorations and impressions on cellulose esters or ethers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,804, 22.9.27).—Naphthazarin is treated at 50–60° in presence of zinc dust or boric acid with an aliphatic amine. Methylamine yields 2 : 8-di-(methylamino)-5-hydroxy-1 : 4-naphthaquinone, m.p. 248–252°, and 2 : 5 : 8-tri(methylamino)-1 : 4-naphthaquinone, m.p. 224–226°, separable by means of chlorobenzene. The triamino-compound gives blue-green, or with *p*-nitroaniline \rightarrow β -hydroxyethyl-*m*-toluidine and 1 : 4-aminohydroxyanthraquinone black shades on acetate silk.

C. HOLLINS.

Dyes [anthraquinoneacridones] and dyeing. R. S. BARNES, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 306,573, 17.11.27).—Arylaminoanthraquinone-*o*-carboxylic acids are converted by pyridine-sulphuric anhydride (etc.) and copper powder in pyridine into leuco-esters which, when treated in substance or on the fibre with acid oxidising agents (acid ferric chloride), give rise to anthraquinoneacridones. The leuco-esters may be applied to animal or vegetable fibres, artificial silk, etc. by dyeing, padding, or printing. Examples are : 1- β -naphthylamino-, 1-anilino-, and 1-*p*-chloroanilino-anthraquinone-2-carboxylic acids. C. HOLLINS.

Manufacture of dyes of the anthracene series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,407, 9.12.27).— α - or β -Hydroxyanthracene deriva-

tives are condensed with 2:3-negatively-substituted α -naphthaquinones, at least one of the negative substituents being a halogen atom. Thus 2:3-dichloro- or dibromo- α -naphthaquinone gives with α -anthranol and β -anthranol halogen-free red vat dyes. 1:5-Dihydroxyanthracene (rufol) similarly gives a violet vat dye, and the 1:8-isomeride (chrysazol) a salmon-red. α -Anthranol and nitro-2:3-dichloro- α -naphthaquinone or 2-chloro-3-acetoxy- α -naphthaquinone give violet vat dyes.

R. BRIGHTMAN.

Pyranthrone dyes. BRIT. ALIZARINE CO., LTD., and P. BEGHIN (B.P. 306,434, 17.11.27).—Brown vat dyes are obtained by heating pyranthrone or its dichloro- or dibromo-derivatives with sulphuryl chloride in nitrobenzene (etc.) first at 90–140° and then at 140–205°. The dyeings are treated with hypochlorite. C. HOLLINS.

Pyrazolone dyes (B.P. 300,321). Dispersions (B.P. 272,896).—See III. **Chemotherapeutical compositions** (B.P. 283,565).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Apparatus for autographic records of the strength and elongation of textile fibres and yarns. S. G. BARKER and N. TUNSTALL (Trans. Faraday Soc., 1929, 25, 103–108).—The friction error is eliminated and the inertia error minimised, whilst the advantage of giving a load-elongation diagram for a fibre or yarn referred to a system of rectangular co-ordinates is retained. The connexion between the test piece and the spiral spring used to measure the tension in it is kept sensibly at rest by causing the other ends of the test piece and of the spiral spring to move outwards at appropriate speeds. Typical records showing the behaviour of wool fibres under various conditions are given.

O. J. WALKER.

Electrical conduction in textiles. II. Alternating current conduction in cotton and silk. E. J. MURPHY (J. Physical Chem., 1929, 33, 200–215; cf. B., 1929, 201).—Data on the alternating current (A.-C.) conduction of cotton and silk of the kind previously investigated are recorded and discussed. The equivalent parallel capacity and conductance as functions of humidity, the amount of electrolytic material present, and frequency have been determined. The A.-C. capacity of cotton increases rapidly with an increase in humidity, especially for high values of the latter. In such cases the capacity is much reduced when the amount of electrolytic material in the cotton is decreased. The capacity of cotton at high humidity is considered to be due chiefly to the electrolytic polarisation capacity of the water paths through which direct current (D.-C.) conduction takes place. The A.-C. and D.-C. conductivities approach identity as humidity increases and become practically equal at humidities above 80–85%. Below this percentage the A.-C. conductivity is greater than that of the D.-C. value, the difference increasing as the humidity becomes less. The A.-C. conductivity not due to that of the D.-C. is affected by the amount of electrolytic material present to approximately the same extent as is the D.-C. conductivity. The rate of increase of the former with frequency decreases with a rise in humidity and is practically independent of frequency at

high humidities. In A.-C. conduction a textile acts as an electrolytic cell in parallel with a dielectric, the water-paths forming the electrolyte, and at high humidities, capacity and conductivity are due chiefly to the electrolyte, whilst at low humidities the dielectric plays the chief part; at intermediate humidities a combination of both factors determines the behaviour of the textile.

L. S. THEOBALD.

X-Ray investigation of the structure of cellulose. E. A. HAUSER (Ind. Eng. Chem., 1929, 21, 124–125).—An attempt is made to correlate known facts, such as the identity period of the crystallised phase in the direction of the fibre axis, with the constitution of cellobiose etc., and the cellulose structure model obtained permits an interpretation in both the rhombic and monoclinic systems, and satisfactorily explains the chemical and physical properties of cellulose. The results achieved in attempting to illuminate the structure of fibrous materials are explained by stating that nature makes preferential use of long, main valency chains bundled up by micellar or cohesion forces into little parcels, and that, according to the differences in chemical constitution and in prevailing conditions, these chains are straight or in a helix orientation, various influences causing changes in the length of the chains and width of the parcels. X-Ray research has possibilities as an accurate determinant of the degree of mercerisation, esterification, and nitration of cellulose. (Cf. B., 1929, 333.)

B. P. RIDGE.

Importance of hydrogen-ion concentration in sulphite-cellulose cooking. E. HÄGGLUND (Papier-Fabr., 1929, 27, 165).—The change in the lignin content of a sodium bisulphite cellulose (known to contain completely sulphonated lignin) after heat treatment with phosphoric acid–potassium phosphate solutions of different p_H under standard conditions has been investigated. The rate of dissolution of the solid lignosulphonic acid is found to depend largely on the hydrogen-ion concentration of the liquid; the lower the p_H the greater is the percentage of lignin dissolved. The results confirm the theory that the decomposition of lignin by acid sulphites proceeds in two stages, viz., the formation of solid lignosulphonic acid and then its dissolution. It is the velocity of the latter reaction which depends to such a great extent on the p_H value of the liquid.

B. P. RIDGE.

Electro-soda process for regeneration of black or brown liquors in soda-pulp manufacture with avoidance of trouble due to odours. F. WALLENBERGER (Papier-Fabr., 1929, 27, 81–83).—During the evaporation of the liquors in the usual regeneration process the sodium sulphides present combine with the organic matter with the formation of highly objectionable mercaptans and mercaptides which are volatile and escape with the fumes, thus polluting the atmosphere. Further, large quantities of waste deposit accumulate from the amount of lime used. These disadvantages may be avoided by treating the sulphide liquor with hydrochloric acid in closed vessels, withdrawing the liberated hydrogen sulphide, and absorbing it in sodium hydroxide solution. The latter is obtained by electrolysis of the sodium chloride solution (produced by the above action of the hydrochloric acid) after removal of the

precipitated organic substances, lignin, etc. The chlorine and hydrogen liberated during the electrolysis are recombined to form hydrochloric acid, whilst the washed and dried lignin material is used as fuel. The cycle of operations is thus complete, and there are no by-products. A disadvantage of the process is that the initial cost of the soda-pulp plant inclusive of the regenerating plant is 20–25% higher than that of plant which operates with the usual recovery process, but on the other hand the running costs are less.

B. P. RIDGE.

Hydrolysis of acetylcellulose during the hydration ageing. I. SAKURADA (J. Soc. Chem. Ind., Japan, 1928, 31, 633–638).—The hydrolysis velocity of mono-, di-, and tri-acetylcellulose can be expressed respectively by the equations $dx_3/dt = k(x_2 - x_3)$; $dx_2/dt = 2k(x_1 - x_2)$; $dx_1/dt = 3k(b - x_1)$, where b is the initial concentration of triacetylcellulose (in g.-mol. of C_6 -unit per unit volume), x_3 , x_2 , and x_1 are the respective amounts of mono-, di-, and tri-acetylcellulose which were transformed.

Y. TOMODA.

Acetylcellulose film and its fibre. I. Relation between the quality of acetylcellulose and its film strength. II. Relation between the conditions of film making and the quality of the resulting film. G. KITA and G. KANNO. III. Spinning of acetylcellulose fibre. G. KITA, T. UEMATSU, and S. MASUDA (J. Soc. Chem. Ind., Japan, 1928, 31, 730–732, 733–738, 739–745).—I. The acetylcellulose obtained by acetylation at 25° gave weaker films than those obtained at lower temperature; it does not always follow that the acetylcellulose of the higher viscosity gives the weaker film.

II. In the making of acetylcellulose films, the optimum pressure prevailing during the evaporation of the solvent is about 70 cm. of mercury, and the optimum concentration of acetylcellulose in acetone solution is about 12%. The influence of diluents, *e.g.*, water, benzene, in the acetone solution on the film quality has been examined.

III. Experimental results of dry-spinning of acetylcellulose are described.

Y. TOMODA.

Cellulose octa-acetate. N. SENDA and Y. UYEDA (J. Cellulose Inst., Tokyo, 1929, 5, 29–32).—The effects of various factors such as time, the amount of acetic anhydride, etc. on the acetolysis of cellulose from a tissue paper of moisture content 5.35% and ash content 0.31% have been studied. In the acetolysis the removal of water- and alcohol-soluble substances from the gelatinous product formed by pouring the acetylated cellulose into water is important. For the same material under controlled conditions of treatment the highest yield of the octa-acetate is given when 20 g. of acetic anhydride are used for treatment of 5 g. of tissue paper in the presence of sulphuric acid; this yield, however, is only 37.5% of the theoretical. For the treatment of different materials under standard conditions the highest yields are given by cotton wadding and tissue paper (27.8% of the theoretical) and the lowest by straw cellulose (9.7%) and sulphite pulp (11.5%).

B. P. RIDGE.

Correction curves for determination of the degree of grinding [of pulp] by the Schopper-

Riegler apparatus. KORN (Papier-Fabr., 1929, 27, 123–125).—For a given degree of grinding read off from the measuring cylinder, if the dry weight of the sample taken differs from 2 g., the correct degree of grinding corresponding with a dry weight of 2 g. may be read off from the curves given.

B. P. RIDGE.

Freeness testing as an aid in pulp evaluation. D. S. DAVIS (Ind. Eng. Chem. [Anal.], 1929, 1, 47–49).—“Freeness” is defined as the rate of drainage of water through pulp, and its measurement by suitable apparatus such as the Williams freeness tester is a simple and accurate procedure. The results obtained are capable of mathematical treatment, and simple exponential equations fit the curves given. It is found that freeness determinations on the same material show variations within 1%, bursting tests within 12%, tensile and stretch tests are very erratic, and fold tests often vary by as much as 50%. The test may be used to distinguish between pulps to be used for any special purpose before complete physical tests are carried out, as a means of localising faults due to beating or other machinery, for comparing the performances of two beaters when the same pulp is being beaten, and for comparing the ease of beating for different pulps when the same beater is used.

B. P. RIDGE.

The “Delthirna” rosin sizing process [for paper]. E. HOCHBERGER (Papier-Fabr., 1929, 27, 83–91, 97–99).—The Delthirna process is not fundamentally new, but the apparatus in which the rosin is brought into solution in the cold by means of sodium hydroxide is new and is described. Comparison is made between this and the usual method of sizing from the point of view of economy and the properties of the paper obtained, as shown by the ink and sodium hydroxide tests, and p_H of the aqueous extract of the paper. Of 66 Delthirna-sized papers, 22 were found better, 5 equally well, and 39 worse sized than comparable samples obtained by the usual method. All book papers, especially strong rag papers, were well sized, in most cases better than by the usual method, whilst those of less than 50 g./m.² were not resistant to writing. The p_H of the aqueous extract of different papers sized by the Delthirna process varies from 4.5 to 5.8, whilst the corresponding values for free rosin-sized papers is approximately constant at 5.5–5.8, indicating non-uniformity of sizing by the former process. This variability is also indicated by the changes in the p_H of the sizing liquid during the sizing operation, no such marked effect being shown for the ordinary process. The nature of the alkali present in the Delthirna size liquid has a considerable effect on the rosin content and other properties of the size; thus, for the same concentration of total alkali, if the latter consists of a mixture of sodium hydroxide and carbonate, the rosin content of the size diminishes rapidly with increasing carbonate content. The effect on the p_H of diluting mixtures containing the alkali as sodium hydroxide and as carbonate, respectively, the conditions governing the flocculation and precipitation of the rosin, as determined experimentally, and the properties of papers sized by the Delthirna and by the ordinary methods under different conditions are described. Comparative sizing experi-

ments show that the free rosin method corresponds with a lower p_H of the liquid, a higher aluminium hydroxide content, and a considerably greater excess of sulphur trioxide than the Delthirna method, and explanations of these results are afforded by different theories of sizing. The effects of the hydrogen-ion concentration of the liquid, the buffering action of different kinds of water, the nature of the electric charges on the colloid complex, the isoelectric point of the material, and of various additions, such as alum, acids, and alkalis, to the size mixtures, are discussed. B. P. RIDGE.

Fundamental principles of the "Delthirna" process. G. STRECKER (Papier-Fabr., 1929, 27, 99—102).—Hochberger's remarks (cf. preceding abstract) are criticised. It is claimed that the use of sulphuric acid in sizing has nothing to do with the Delthirna process, which is merely a process for sizing with completely saponified rosin in a state of molecular division. In many cases sizing is carried out without the addition of sulphuric acid, though in a few cases it is necessary to prevent the formation of harmful complexes of rosin and aluminium hydroxide, when, provided it is present in small quantity, it acts in a preventive manner. In practice, the Delthirna process works satisfactorily, is not wasteful in alum, produces no inferiority in the strength of the paper, and, with careful supervision, effects considerable economy. B. P. RIDGE.

[Fundamental principles of the "Delthirna process."] E. HOCHBERGER (Papier-Fabr., 1929, 27, 102—103).—A reply to Strecker's criticism (cf. preceding abstract). B. P. RIDGE.

Wool oil. HART.—See XII.

PATENTS.

Digestion of fibrous material. T. L. DUNBAR and A. F. RICHTER, Assrs. to CHEMIPULP PROCESS INC. (U.S.P. 1,699,056, 15.1.29. Appl., 12.12.27).—The high pressure generated during the digestion of the material with an acid liquor is utilised to discharge the hot gases and vapours generated during the process into a supply of fresh acid liquor, and gases etc. are subsequently withdrawn by suction from the digester and brought while still hot into contact with a flowing stream of the acid liquor for future use. L. A. COLES.

Manufacture of objects of acid-proof material. I. G. FARBENIND. A.-G. (B.P. 276,697, 30.8.27. Ger., 30.8.26).—The objects are constructed of wide-mesh fabric impregnated with liquid artificial resins subsequently hardened by heat. L. A. COLES.

Acid-sulphite pulp process. R. B. WOLF (U.S.P. 1,699,556, 22.1.29. Appl., 19.6.25).—To avoid losses of sulphur dioxide when using a strong sulphite liquor for shortening the time of digestion, the gases from the digester are discharged at the end of the cooking operation through an unobstructed relief opening, and cooled with water to condense steam and separate fibre; they are then conveyed in surface contact with successive portions of strong sulphite liquor circulated from a storage supply at such a rate that its temperature does not rise above that at which the absorbed gases may be retained in the liquor under atmospheric pressure. F. R. ENNOS.

Manufacture of coloured compositions from cellulose esters and the like. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 304,814, 26.10.27. Addn. to B.P. 247,288; B., 1926, 315).—In the process of the prior patent, water-resistant materials such as resins, paraffins, waxes, etc. are milled into the celluloid with the insoluble colouring matter, giving water-resistant products suitable for lacquers. (Cf. also B.P. 293,485; B., 1928, 668.) C. HOLLINS.

Stencil sheet. S. HORII (U.S.P. 1,698,705, 8.1.29. Appl., 18.5.26).—Impregnation with a mixture of phenol resin, cellulose nitrate, stearin, and castor oil is claimed. A. R. POWELL.

[Laundry] drum-washing machines. P. DIEBOLD (B.P. 303,672, 10.2.28).

Treating [drying] paper-board etc. R. KASTNER and H. SCHMOLKA (B.P. 284,319, 13.1.28).

[Supplying pulp in] manufacture of paper. R. MARX (B.P. 306,625, 1.12.27).

Strawboard waste liquor (U.S.P. 1,699,257).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyeing of cellulose esters and ethers. I. G. FARBENIND. A.-G. (B.P. 279,135, 18.10.27. Ger., 18.10.26).—Unsulphonated *p*-arylamino-diphenylamines, carrying in one terminal nucleus three *o*- and *p*-substituents, two of which are nitro-groups, and a carboxyl group either in the other of the three positions or in the middle nucleus, give brown shades on acetate silk. Examples are: 2:6-dinitro-4'-anilino-diphenylamine-4-carboxylic acid, 2:4-dinitro-4'-anilino-diphenylamine-6-carboxylic acid, 2:4-dinitro-4'-*p*-hydroxyanilino-6-methyldiphenylamine-3'-carboxylic acid, 2:6-dinitro-4'-β-anisidinodiphenylamine-4-carboxylic acid, and methyl 2:6-dinitro-4'-anilino-3'-carboxydiphenylamine-4-carboxylate.

C. HOLLINS.

Dyeing and printing by means of vat dyes [soluble leuco-esters]. DURAND & HUGUENIN SOC. ANON. (B.P. 306,800, 28.11.27. Addn. to B.P. 220,964; B., 1925, 879. Cf. B.P. 281,336; B., 1929, 280).—The acid necessary for development of the colour on steaming is supplied by adding ethyl tartrate to the dye-bath or printing paste. C. HOLLINS.

Waterproofing of textile and other materials. C. J. MORETON, and "PRUFIX," LTD. (B.P. 305,493, 5.8.27).—Textile materials and paper are waterproofed by impregnation with an aqueous emulsion containing gum tragacanth and/or Indian gum (the gum obtained from *Sterculia urens* or *Cochlospermum gossypium*), soap, an animal, vegetable, or mineral wax, and ammonia, and drying; the treated material is then impregnated with an aqueous solution of a metal sulphate or acetate, e.g., titanium sulphate, zinc sulphate, aluminium acetate or sulphate. (Cf. B.P. 262,605; B., 1927, 165.)

A. J. HALL.

Impregnating a continuously moving band of fabric with liquid. A. H. KILNER (B.P. 306,586, 23.11.27).

Dyeing of acetate silk (B.P. 300,299). **Colorations on cellulose esters or ethers** (B.P. 304,804). **Dyeing with anthraquinoneacridones** (B.P. 306,573).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Intensive production of sulphuric acid (*d* 1.70). G. A. PERLEY (Ind. Eng. Chem., 1929, 21, 202—205).—A laboratory plant of the tower type is described in which it is claimed that oxidation of sulphurous acid is effected in the liquid phase. After the Glover tower the gases enter a reaction tower fed with nitric acid, the reaction which occurs being: $3\text{H}_2\text{SO}_3 + 2\text{HNO}_3 \rightarrow 3\text{H}_2\text{SO}_4 + \text{H}_2\text{O} + 2\text{NO}$. The evolved gases containing nitric oxide pass through a series of absorption towers of which the last one is fed with water. There is no Gay Lussac tower nor nitrous vitriol, but the product of the reaction tower is freed from any nitric acid it may contain in the Glover tower. The plant operated on a space of 0.01 cub. ft./lb. of sulphur/24 hrs. A 10% mixture of sulphur dioxide with air was used in these experiments. Analyses of gases and liquids are given. Constructional problems, particularly that of cooling, require investigation on a larger scale. C. IRWIN.

[Platinum] gauze catalyst in ammonia oxidation. G. A. PERLEY and M. W. VARRELL (Ind. Eng. Chem., 1929, 21, 222—223).—Conversion efficiencies were determined for a single gauze, for a pair of gauzes in close contact, and for a pair separated by 0.5 in. and 1.0 in. space, respectively. The gas mixture was preheated to 470°. Little difference was found between the efficiencies of the pairs in close contact and those separated by spaces. It had been anticipated that with high gas flows the space would act as a temperature stabiliser, but if this is so any benefit is neutralised by secondary reactions. C. IRWIN.

Replacing lime by ferric oxide in the manufacture of sodium chromate. F. F. VOLF and L. I. POROV (J. Chem. Ind. Moscow, 1928, 5, 618—625).—Use of lime to prevent fusion in the manufacture of sodium chromate from chromite occasions loss of chromium; economy is effected by replacing the lime by ferric oxide. Larger amounts of sodium carbonate are required, together with finer grinding and longer calcination, 93.2% of the chromium then being extracted. CHEMICAL ABSTRACTS.

Purification of crude barium chloride by sodium chloride. J. E. ADADUROV and K. L. BRODOVICH (J. Chem. Ind. Moscow, 1928, 5, 640—642).—The solubilities of barium chloride in 5—30% sodium chloride solutions at 16—100° have been determined and compared with those in hydrochloric acid solutions of the same concentrations. The solubility in sodium chloride solutions is minimal at 35°; for 25—35% solutions the solubility in sodium chloride solutions is much greater than that in hydrochloric acid solutions. The solubility of lead chloride in sodium chloride solutions increases with increase in the concentration of the solution; the values approximate to those in hydrochloric acid of the same concentration. The formation of the compounds $\text{PbCl}_2 \cdot 2\text{NaCl}$ and $\text{PbCl}_2 \cdot 2\text{HCl}$ is assumed. For the

purification of barium chloride, lead sulphide is removed by vacuum-filtration of a solution or by acidifying at 100° with hydrochloric acid; sodium chloride (250—270 g. per litre) is then added, the solution cooled to 35—40°, and the precipitated barium chloride (90%, of 99% purity) filtered in a vacuum. CHEMICAL ABSTRACTS.

Röntgenographic study of varieties of asbestos from different mines. G. L. CLARK and S. L. VAN ORDEN (Kautschuk, 1929, 5, 28—35).—Chrysotile asbestos (7 samples), amphibole (3 samples), crocidolite, and amosite were submitted to röntgenographic examination in the fresh condition, also after heating to 900°, and after heating with concentrated hydrochloric acid. In the acid treatment the chrysotiles lost more than 50% in weight and X-rays showed a complete disappearance of fibrous structure; the other samples showed less loss in weight and retained part of their fibrous character. On heat-treatment the chrysotiles suffered a high loss in weight, but in some cases their structure was less affected than that of certain of the amphiboles. It was possible to distinguish between all the samples by the degrees of difference in behaviour.

D. F. TWISS.

Cylinders for permanent gases. ANON.—See X. **Red lead.** WOLFF and ZEIDLER; also LÜTY.—See XIII.

PATENTS.

Manufacture of nitric acid. W. R. ORMANDY (B.P. 306,705, 21.2.28).—Oil or other fuel is burnt as a flame submerged in water in the presence of a metallic catalyst capable of forming a number of oxides (*e.g.*, vanadium oxide; 0.04% by wt. of the oil used) injected into the flame or combustion air as a powder or spray.

W. G. CAREY.

Catalyst for the production of hydrocyanic acid. T. EWAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 305,816, 10.2.28).—Hydrogen cyanide is obtained in good yield by passing formamide vapour at 500° over an alumina, zirconia, or thoria catalyst which has been subjected to prolonged heating at above 1000°, preferably to incipient fusion. A. R. POWELL.

Methods of drying gases or gas mixtures for the synthesis of ammonia. GASVERARBEITUNGSGES. M.B.H. (B.P. 293,361, 4.7.28. Ger., 4.7.27).—The gases are dried in the usual way by means of chemicals, mixed with a small quantity of ammonia from the process, and cooled to —25° to —70°, whereby the ammonia liquefies and absorbs the last traces of moisture. A. R. POWELL.

Catalytic or contact materials [for ammonia synthesis]. A. HURTER (B.P. 305,753, 7.12.27).—A solution of zirconium oxychloride is treated with a solution of potassium ferrocyanide, and the washed precipitate is dried at 100°, ground to a fine powder, and heated in hydrogen at 140° for a considerable time, then at 400° in a mixture of hydrogen and nitrogen, first at atmospheric pressure, then at gradually increasing pressure up to 90 atm. The product gives a yield of 12 vol.-% of ammonia at 400° under 90 atm. pressure with a gas velocity of 2—3 m.³/hr./kg. of catalyst. A. R. POWELL.

Obtention of salt by vacuum evaporation. METALLWERKE VORM. J. ADERS A.-G. (B.P. 281,726,

5.12.27. Ger., 4.12.26).—Brine is pumped through a heater into a vacuum evaporating pan through a thin connecting tube in the shape of a U, so that the pressure fall between the heater and the vacuum pan is opposed by the column of liquid in the U-tube even when the pump is stopped. The separated salt is removed from the lower funnel-shaped portion of the pan continuously, while the supernatant brine overflows back to the supply reservoir of the heater, which is heated by means of waste steam or the vapour from the salt pans.

A. R. POWELL.

Treatment of calcium compounds with sulphuric acid or its salts. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 301,210, 5.12.27 and 19.3.28).—In the treatment of phosphate rock or similar calcium compound with sulphuric acid, a granular, readily filterable form of gypsum is obtained by adding a small quantity of metaphosphoric acid to the sulphuric acid before use. The process is also applicable to the production of potassium nitrate from calcium nitrate and potassium sulphate.

A. R. POWELL.

Manufacture of a non-hygroscopic or slightly hygroscopic double salt from calcium nitrate. AZOGENO SOC. ANON. PER LA FABBR. DELL'AMMONIACA SINTETICA E PROD. DERIVATI, C. TONIOLO, and B. TANZI (B.P. 276,350, 18.8.27. It., 19.8.26).—Ammonium sulphate (1 mol.) or phosphate (1 mol.) and neutral calcium nitrate (1 mol.) are allowed to react in the presence of water or steam (2 mols.), whereby a solid non-hygroscopic salt is obtained which is suitable for use as a fertiliser. Alternatively, 2–4 mols. of water or steam may be employed in the reaction and the resulting solution evaporated until the residue contains only 2 mols. of water to 1 mol. of calcium nitrate.

A. R. POWELL.

Production of high-percentage calcium or magnesium cyanamide or mixtures thereof. N. CARO and A. R. FRANK (B.P. 281,610, 30.9.27. Ger., 2.12.26).—Calcium and/or magnesium carbonate is treated with dry ammonia at 700–800° at the ordinary or at increased pressure, the amount of ammonia used being 500–5000 c.c. per c.c. of carbonate; the larger the volume of ammonia used the lower should be the temperature to obtain a good yield.

A. R. POWELL.

Production of alumina, sodium carbonate, and hydrochloric acid. N. J. GAREAU (B.P. 305,599, 3.8.27).—Clay with a low content of magnesia and ferric oxide is formed into briquettes which are calcined at a low red heat to render them porous; they are then charged into the top of a tower the lower part of which is heated, and a mixture of steam, sulphur trioxide, sulphur dioxide (from a later stage of the process), and nitric acid is passed into the bottom of the tower, while a spray of water is passed into the top. The alumina in the clay is thus converted into aluminium sulphate and the silica is dehydrated. The briquettes are leached with dilute sulphuric acid on the countercurrent principle, and the concentrated leach liquors are sprayed into a reverberatory furnace maintained at a dull red heat whereby a basic aluminium sulphate, $\text{Al}_2\text{O}_3 \cdot \text{SO}_3$, is produced with liberation of sulphur trioxide. This sulphate is briquetted with salt and powdered coal, and the dried briquettes

are heated with steam at 300°, whereby hydrogen chloride is evolved and a mixture of sodium sulphate and alumina obtained still in briquette form. This product is heated to redness in a rotary furnace to reduce the sulphate to sulphide, and steam is passed into the mixture to decompose the latter with the formation of sodium aluminate and hydrogen sulphide. The aluminate is leached out of the residue and the solution treated with carbon dioxide from the reducing furnace to obtain alumina and sodium carbonate. A. R. POWELL.

Manufacture of bismuth oxide, bismuth carbonate, or other compounds of bismuth. R. S. CARRERAS (B.P. 298,587, 7.7.27).—By the electrolysis of a dilute solution of sodium chlorate saturated with carbon dioxide, using a bismuth anode and an iron or carbon cathode, bismuth hydroxide containing a small proportion of carbonate is formed as a white, non-adherent anode slime.

A. R. POWELL.

Production of soluble lead reagents [containing lead chloride]. H. W. ROBINSON and D. W. PARKES (B.P. 305,827, 17.2.28).—Solutions particularly adapted for recovering pyrotechnicals from ammoniacal solution as described in B.P. 305,494 (B., 1929, 313) are prepared by heating, e.g., 46 pts. of lead chloride, 38 pts. of crystalline sodium acetate, and 15 pts. of water at about 85°, and then adding an acid, e.g., 0.78 pt. of nitric acid, *d* 1.42.

L. A. COLES.

Production of titanium oxide from titaniferous iron ores. C. R. WHITTENORE (U.S.P. 1,699,173, 15.1.29. Appl., 21.8.24).—The ores are reduced without fusion or sintering to convert iron compounds into iron sponge, and after cooling the product in a non-oxidising atmosphere the iron sponge and titanium compounds are magnetically separated from the remaining constituents. The product is treated with a solvent, and the insoluble residue is digested at 250° with an acid, disintegrated, and lixiviated with cold water to extract titanium salts, which are subsequently converted into the oxide by hydrolysis and calcination.

L. A. COLES.

Production of base-exchanging substances. A. ROSENHEIM (B.P. 279,028, 6.7.27. Ger., 16.10.26).—Artificial or natural silicates are treated at high temperatures and pressures with aqueous solutions of acid, neutral, or alkaline inorganic compounds that will supply the requisite base-exchanging constituent. Two or more successive treatments with different solutions may be necessary; thus granite may be treated first with hydrochloric acid at 200° and 16 atm., then with sodium silicate solution at 150° and 10 atm. [Stat. ref.]

A. R. POWELL.

Recovery of nitrogen from liquors obtained in the production of potassium nitrate by decomposition of potassium chloride with nitric acid. KALI-IND. A.-G., C. T. THORSELL, and A. KRISTENSSON (B.P. 287,133, 21.2.28. Ger., 16.3.27).—By cooling a mixture of potassium chloride solution and nitric acid to –5° about 85% of the potassium nitrate formed in the reaction crystallises out. The mother-liquor is heated with ferrous chloride to reduce the remaining nitrates to nitric oxide, which is boiled off, washed with an alkali to free it from chlorine compounds, mixed with air, and

reconverted into nitric acid. The ferric chloride solution obtained is distilled to remove part of the free hydrochloric acid, then reduced with iron to ferrous chloride for use again in the process. A. R. POWELL.

Producing solid blocks of carbon dioxide ice. A. P. THURSTON. From DRYICE CORP. OF AMERICA (B.P. 298,792, 15.11.27).—Flue gases rich in carbon dioxide are freed from impurities, dried by freezing, and further cooled by means of nitrogen obtained from later stages of the process. The liquid carbon dioxide so obtained is then frozen under pressure of 1500–2000 lb./in.² The nitrogen obtained in liquefying the carbon dioxide is cooled to –130° to –185° and used as a refrigerant for freezing the liquid carbon dioxide. Dense, transparent blocks of carbon dioxide weighing up to 100 lb. may be obtained by the process.

A. R. POWELL.

Production of alkali carbonates. L. BRADLEY and E. P. MCKEEFE, Assrs. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,702,588, 19.2.29. Appl., 5.11.21. Renewed 20.5.27).—See Can. P. 248,096; B., 1926, 321.

Production of metal chlorides free from water and oxides. M. JAEGER, W. MOSCHEL, and R. SUCHY, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,702,301, 19.2.29. Appl., 23.4.27. Ger., 15.1.26).—See B.P. 293,410; B., 1928, 670.

Recovery of heat and water vapour in gaseous reactions. G. L. E. PATART (U.S.P. 1,703,747, 26.2.29. Appl., 14.1.25. Fr., 21.1.24).—See B.P. 228,153; B., 1926, 156.

Pumps for corrosive liquids (B.P. 289,018).—See I. **Ammonium sulphate** (B.P. 292,995). **Ammonia from gases** (B.P. 281,288). **Hydrogen and sulphur from gases** (B.P. 289,885).—See II. **Catalyst for methyl alcohol production** (B.P. 300,142).—See III. **Acid-proof material** (B.P. 276,697).—See V. **Alkali bismuth tartrates** (B.P. 304,956).—See XX.

VIII.—GLASS; CERAMICS.

Alkali content of medicinal glass. B. SCHWENKE (Pharm. Ztg., 1929, 74, 261–263).—The alkali removed from various samples of glass vessels, after $\frac{1}{2}$ and $1\frac{1}{2}$ hrs.' boiling with water, reckoned in mg. of sodium hydroxide per 100 cm.² of surface exposed, is tabulated. Of ten different samples, from which the alkali removed varied from 0 to 5.8 mg., only three are regarded as satisfactory.

S. I. LEVY.

Dehydration of kaolin. H. A. J. PIETERS (Diss., Delft, 1928, 148 pp.).—Kaolin, alumina, and silica are hygroscopic. Kaolin exhibits three heat changes: at 100°, escape of moisture; at 600°, dehydration; at 950°, exothermal decomposition. In the dehydration experiments air saturated with water vapour at different temperatures was passed over kaolin and the temperature determined for each aqueous tension at which the substance lost weight. Curves afford the following values of p : 17 mm. at 430°, 149 mm. at 475°, 355 mm. at 505°, 760 mm. at 540°. The heat of dehydration is computed as 300 g.-cal. per g. Dehydration of more than 13% was obtained for p_{H_2O} 0 at 470°, 17 mm. at 490°, 149 mm. at 510°, 760 mm. at 580°, and 3.6 atm. at 640°.

Dehydration of pholerite is slower, corresponding temperatures for the first three values of p_{H_2O} being 510°, 510°, and 560°. The dehydration accords with that of a solid solution of water content x . Between 3 and 13% H_2O the equilibrium is univariant. Metakaolin (dehydrated kaolin) does not absorb any water at suitable temperatures below 700° for $p = 149$ at 760 mm. When heated with water under pressure (125–150 atm. at 300°), alumina absorbs 16–17% of water, forming a monohydrate; ignition above 1100° destroys the absorptive power. Amorphous silica absorbs 2.7–45.7% of water at 80–130°. Amorphous (Zettlitz) kaolin absorbs up to 14.7% of water at 50–130 atm., the amount depending on the temperature of ignition. The absorptive powers of metakaolin, sillimanite, and a mixture of alumina and silica were measured. Ignition of kaolin above 1100° probably gives sillimanite and crystalline silica. Static determinations of the solution pressure of kaolin and pholerite agreed with the dynamic determinations. Metakaolin contains neither free silica nor alumina. The alumina from metakaolin dissolves almost completely in hydrochloric acid if the ignition temperature is below 900°. Hydrated metakaolin is insoluble in hydrochloric acid. Metakaolin (produced between 600° and 900°) is probably a kaolin anhydride, $Al_2O_3 \cdot 2SiO_2$. Attempts to synthesise kaolin failed.

CHEMICAL ABSTRACTS.

Drugs and coloured glass. EISENBRAND.—See XX.

PATENTS.

Manufacture of unsplinterable glass. J. H. ROBERTSON (B.P. 306,351, 25.6.28).—A colloidal layer consisting of silicic acid or its sodium or potassium salt in a solution of pure aqueous gelatin, gelatin treated with chrome alum, agar-agar, albumin, or casein, is used to cement celluloid to glass.

A. COUSEN.

Compound transparent [glass] sheets. Soc. D'ETUDE DES VERRES ET GLACES DE SURETÉ (B.P. 293,052, 30.6.28. Fr., 1.7.27).—The central layer between the glass sheets is composed either wholly or in part of regenerated cellulose, ethyl and methyl celluloses or other homologous cellulose ethers, or of the polysaccharide, caroubin, all of which tend to bleach on exposure to light.

A. COUSEN.

Laminated sheet glazing material. C. W. BONNIKEN (B.P. 306,423, 16.8.27).—Plate glass unpolished on the sides used as interfaces is reinforced with a formaldehyde-urea condensation product which has the same refractive index as the glass, and thus renders the rough interfaces transparent.

W. G. CAREY.

Strengthened glass sheets or plates. P. H. HEAD (B.P. 306,279, 13.2.28).—The reinforced glass is sealed by cauterising the edge of the interposed layer by means of a heated wire, or by application of nitric acid, caustic soda, or phenol. A subsequent treatment with a solution of diacetone alcohol prevents excessive penetration of the caustic soda solution.

A. COUSEN.

Manufacture of hollow articles of quartz etc. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,117, 26.8.27).—Quartz is melted in a mould by means of a removable rod-shaped resistor, and the latter is then removed and replaced by a pipe through which is

blown a current of air to inflate the mass against the sides of the mould.

A. R. POWELL.

Cellular silica product and method of fabrication. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of H. L. WATSON (B.P. 295,628, 25.7.28. U.S., 15.8.27).—Quartz sand is heated at 1700° for 30 min. to convert it into porous cristobalite or tridymite, cooled, mixed with an organic binder, *e.g.*, shellac, moulded to shape, and heated again, slowly to 400° then rapidly to 1750°, so that the mass softens while still occluding gas. The product is light, porous, and impervious to liquids.

A. R. POWELL.

Composition of matter resistant to high temperature and its manufacture. R. L. FRINK (B.P. 306,216, 29.8.27).—Non-plastic material of the sillimanite or bauxite type etc. is bonded with a cement (oxides and chlorides of the alkaline and rare earths) which sets at ordinary temperatures. The coefficient of expansion of the bond should be approximately equal to that of the main refractory. The moulded or rammed material is then fired at 1200–1500° in order to produce a high-temperature bond. By suitable addition of sawdust, wax, resin, etc., blocks of any desired porosity may be built up. An efficient bonding material for sillimanite is composed of calcined silica 50 pts. by wt., boric acid 30 pts., magnesia 20 pts., and magnesium chloride 5 pts. These materials (except the magnesium chloride) are ground to pass 0.01 in. mesh, and 10% of the mixture is mixed with the sillimanite. The magnesium chloride necessary is dissolved in the water used to wet the mass.

J. A. SUGDEN.

Refractory cement. J. W. MARDEN and H. K. RICHARDSON, Assrs. to WESTINGHOUSE LAMP CO. (U.S.P. 1,695,812, 18.12.28. Appl., 25.6.23).—A cement for lining crucibles or furnaces subjected to very high temperatures comprises a mixture of thoria (previously ignited at a high temperature and ground to an impalpable powder), zirconia, cryolite or potassium thorium fluoride, and powdered clay, together with a flocculating agent such as sodium or ammonium phosphate.

A. R. POWELL.

IX.—BUILDING MATERIALS.

Heat transmission [from buildings]. M. FISHENDEN and A. F. DUFTON (Dept. Sci. Ind. Res., Building Res., Rept. No. 11, 1929, 20 pp.).—The coefficient of conductivity (K) of materials comprising a wall is expressed preferably in terms of the thermal resistance (R), which represents the number of hours required for the transmission of one B.Th.U./ft.², when the temperature difference between the two surfaces is 1° F. Values are given for many different varieties of brick, concrete, and materials used in modern construction. The value of K is empirical, and the value required depends on the situation and exposure of the wall, factors which are discussed in relation to heat transference from warmed rooms. Experiments on a practical scale are in progress to determine heat losses from buildings of different construction and heated by different methods, taking into account variations of meteorological conditions.

C. A. KING.

Heat transfer in the rotary kiln burning Portland cement clinker. R. D. PIKE (Ind. Eng. Chem., 1929, 21, 230–234).—A rotary cement kiln includes two zones—the clinkering and the calcination zones. From temperature readings on a particular kiln it is calculated that the heat communicated per lb. of clinker is 175 B.Th.U. in the clinkering zone and 1196 B.Th.U. in the calcination zone. In the same way the heat transferred in the heating zone and the temperature of the exit gases are calculated. The heat transfer number in the calcination zone is 3.22 B.Th.U. per hr. per 1° F., and in the heating zone is 5.23. Calculations based on formulæ ignoring radiation give incorrect results for these quantities. The figures given are experimental results. Their theoretical calculation involves many variables, such as the rate of rotation of the kiln. At any temperature the transfer number is a linear function of the mass velocity, the part due to radiation being constant. The heat consumed in the clinkering zone is calculated not to exceed 4% of that supplied by the fuel.

C. IRWIN.

Breeze and clinker aggregates [for concrete]. F. M. LEA (Dept. Sci. Ind. Res., Building Res., Tech. Paper No. 7, 1929, 86 pp.).—The behaviour of more than 2500 test blocks indicates that the failure of breeze or clinker concretes is determined by the amount and nature of the combustible matter present. Coals may be graded without any sharp lines of demarcation into those which are not dangerous to varieties which when present in relatively small quantities cause cracking of a concrete. Those of the latter class have a high oxygen content and a rapid rate of adsorption of oxygen, moisture, and methylene-blue, the rate of oxygen adsorption being increased by the presence of free lime. Cracking of concretes is due to dimensional changes on wetting, the dry-wet movement of high-oxygen coals being greatly increased in the presence of a setting cement; the swelling is increased also to a smaller extent by a gradual loose combination of adsorbed oxygen. Decomposition of coals by heating at 500° or above decreases the dangerous properties of any coal, though oxidation of coal at lower temperatures acts in the opposite direction. The presence of sulphur appears to exert only a minor influence; the total permissible sulphur, excluding that present as sulphate, is fixed tentatively at a maximum of 0.75%. The limiting amount of a dangerous coal remains approximately constant whether a normal, rapid-hardening, blast-furnace, or aluminous Portland cement is used, and neither weathering nor any process of steam treatment of the concrete renders a coal less harmful, though steaming may stabilise concrete against the action of sulphur compounds. Methods of testing breeze and clinker for suitability for aggregates are given.

C. A. KING.

Use of blast-furnace slag in concrete. W. KOSFELD (Stahl u. Eisen, 1929, 49, 243–249).—A blast-furnace slag having a composition such that $(\text{CaO} + \text{MgO})/(\text{SiO}_2 + \text{Al}_2\text{O}_3) \approx 1.1\text{--}1.2$ forms a satisfactory ballast for concrete provided that no deleterious impurities are present. The grain size should be such that 40–65% passes a 7 mm. hole screen; of the screened

material 15% should be finer than 1 mm., 25% between 1 and 3 mm., and 20% from 3 to 7 mm. Tables showing the strength of slag concrete and its permeability to water are given, and the question of a standard specification for slag for concrete is discussed. A. R. POWELL.

Mechanism of the setting and hardening of cement. T. MAEDA (Chim. et Ind., 1929, 21, 35—36).—See B., 1928, 672.

PATENTS.

Rotary cement-burning kilns. A. V. JENSEN (B.P. 306,613, 28.11.27).—The inlet end of the kiln is closed except for the entry of the slurry pipe. Close to this end one or more pipes, open at each end, pass across the kiln and through the kiln wall. Flanged openings into such pipes permit the exit of combustion gases to the atmosphere without the loss of slurry. It is convenient to allow the end of the kiln to project beyond the smoke chamber. C. A. KING.

Improving the hydraulic properties of Portland cement. I. G. FARHENIND A.-G. (B.P. 298,943, 21.8.28. Ger., 17.10.27).—The initial and final hardening of Portland cement are increased without influencing the setting period by the addition of up to 5% of calcium sulphide. C. A. KING.

Treatment of gypsum. R. E. HAIRE (U.S.P. 1,702,940, 19.2.29. Appl., 22.5.24).—In the treatment of low-grade gypsum rock, the harder and coarser particles left after the separation of commercially pure gypsum are re-ground in order to separate more gypsum. J. A. SUGDEN.

Manufacture of porous clinker-like materials. E. I. LINDMAN (B.P. 280,567, 10.11.27. Swed., 10.11.26).—Clay or rocks which evolve gases on heating are crushed and graded to pass a 15 mm. and to stop on a 2 mm. hole screen, and the undersize is briquetted or moulded into pieces between these sizes. The graded product is mixed with crushed fuel and burnt in shaft furnaces by means of a current of air passed upwards through the mass. The clinker obtained is especially suitable as a ballast for concrete or for macadam. Moulded bodies may be made from the raw material and fuel together with sand or quartz, and, if required, iron reinforcements; these bodies are burnt in moulds the sides and bottom of which are provided with a heat-insulating material of high m.p. A. R. POWELL.

Production of cellular building material. G. M. THOMSON (B.P. 305,806, 31.1.28).—Plaster of Paris or a quick-setting cement is formed into a uniform paste with a dense foam in such a manner as not to include additional air from the atmosphere. A. R. POWELL.

Wall board. J. F. HAGGERTY, Assr. to NAT. GYPSUM Co. (U.S.P. 1,702,966, 19.2.29. Appl., 13.7.25).—Wood fibres, soaked in water, are incorporated with gypsum; the finished product should contain not more than 2% by wt. of gypsum. H. ROYAL-DAWSON.

Manufacture of emulsions or suspensions [for roads etc.]. G. BAUME, P. CHAMBIGE, and D. BOUTIER (B.P. 280,930, 16.11.27. Fr., 17.11.26. Addn. to B.P. 255,074; B., 1928, 194).—The mixtures described previously contain in addition material capable of combining with ingredients of the mixtures or with

other added substances to form soaps etc. having emulsifying properties, e.g., alkali hydroxides, carbonates, etc. The basic mixture comprises various substances adapted to impart desired properties to the product; filling material may also be added.

L. A. COLES.

Manufacture of plastic compositions. J. F. HAGGERTY, Assr. to NAT. GYPSUM Co. (U.S.P. 1,702,965, 19.2.29. Appl., 27.10.25).—See B.P. 260,557; B., 1927, 724.

Furnace walls and bricks therefor. E. C. R. MARKS. From BERNITZ FURNACE APPLIANCE Co. (B.P. 306,601, 25.11.27).

Production of facings, such as road surfaces and floorings, or other structures, such as pipes, mouldings, etc. [from concrete]. M. DENIAU (B.P. 282,039, 29.11.27. Fr., 8.12.26).

Granulation of pulverised materials (B.P. 292,987).—See I. Bituminous emulsions (B.P. 305,742 and 305,716).—See II. Refractory cement (U.S.P. 1,695,812).—See VIII. Metal cement (U.S.P. 1,699,346).—See X.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Yield point of steel at high temperatures. F. KÖRBER (Stahl u. Eisen, 1929, 49, 273—277).—Determinations of the yield point of boiler plate and cast steels of varying composition show that these metals may be divided into a number of groups according to the amount of alloying element present, and that in every group the ratio of the yield point at any given temperature to the tensile strength at the ordinary temperature is a constant. A. R. POWELL.

Heat-treatment and testing of high-speed tool steel. F. RAPATZ (Stahl u. Eisen, 1929, 49, 250—254).—Drills and cutting tools of high-speed tool steel should be hardened by quenching in oil from 1280—1320° and subsequently tempered at 550—600°. The hardening operation is carried out in an electrically-heated borax bath, as borax alone has no decarburising action on the steel, whereas barium chloride appears to dissolve oxygen at high temperatures, and this causes quite a deep decarburisation to take place. The time required for tempering varies from 0.25 to 2 hrs., according to the composition of the steel; after treatment, the tools are allowed to cool slowly in air. The micro-structure and hardness of some tool steels are briefly discussed. A. R. POWELL.

Laboratory corrosion tests of mild steel, with special reference to ship plate. H. S. RAWDON (Bur. Stand. J. Res., 1929, 2, 431—440).—A series of corrosion tests on mild steels was made by the wet-and-dry and the continuous-immersion methods in sea-salt solutions. The steels varied in copper content from a trace to over 0.6%. No differences in corrosion rate were obtained indicating superior corrosion-resistance of any of the compositions used. The corrosion rate in the wet-and-dry test decreased as the surface film was built up, but was always higher than that for simple immersion. The

results do not confirm the claims made for superior corrosion-resistance of the ship plates of *Leviathan*.

C. J. SMITHELLS.

Determination of silicon in ferrosilicon and in other iron alloys. A. STADELER (Arch. Eisenhüttenw., 1928—9, 2, 425—437; Stahl u. Eisen, 1929, 49, 325—326).—A critical examination has been made of the various methods which have been proposed for the determination of silicon in iron alloys. For alloys soluble in acids, decomposition is best effected by means of hydrochloric acid and bromine, and the filtrate from the main silica, together with the first wash-water, should be evaporated again to recover the last traces of silica; alternatively, the main filtrate alone is evaporated together with the precipitate obtained by adding ammonia to the wash-water and boiling until excess is expelled. For alloys insoluble in acids, decomposition by fusion with sodium peroxide, either alone or admixed with sodium potassium carbonate, gives the best results. In this case two evaporations of the first filtrate and wash-waters are necessary. For alloys containing titanium, nitric and sulphuric acids should be used instead of hydrochloric acid for the evaporation.

A. R. POWELL.

Determination of aluminium in steel. A. T. ETHERIDGE (Analyst, 1929, 54, 141—144).—A satisfactory method consists in removing iron from a chloride solution by extraction with ether, and other interfering metals by electrolysis over a mercury cathode from a sulphuric acid solution, the apparatus and operations being similar to those given for vanadium (B., 1928, 714). Phosphorus pentoxide forms the largest correction.

D. G. HEWER.

Determination of chromium, tungsten, molybdenum, vanadium, nickel, manganese, and cobalt in high-alloy steels. H. MENDE (Chem.-Ztg., 1929, 53, 178—179).—Detailed directions are given for the analysis of alloy steels containing high percentages of metals other than iron. Chromium is oxidised with ammonium persulphate and titrated with permanganate solution. Nickel is precipitated by dimethylglyoxime after oxidation with nitric acid. Cobalt is freed from the bulk of other metals by addition of zinc oxide to a solution in nitric acid, and the filtrate is treated with a solution of nitroso- β -naphthol in the presence of acetic acid; the precipitate is calcined to give cobalt oxide. Tungsten is weighed as its trioxide; molybdenum and vanadium are separated in the form of alkali salts from which molybdenum sulphide is precipitated by hydrogen sulphide, and vanadium is subsequently titrated with permanganate solution. A colorimetric test for molybdenum and vanadium is also available unless high percentages of chromium are also present. The method of Hallbauer and Krüger (B., 1927, 447) is used for manganese determinations. R. H. GRIFFITH.

Ordinary commercial cylinders for the "permanent" gases. Summary of recommendations (revised). ANON. (Dept. Sci. Ind. Res. Gas Cylinders Res. Commee., 1929, 7 pp.).—The following amended recommendations supersede those of 1921. The cylinders should be of steel containing 0.43—0.48% C, but, alternatively, steel of 0.25% C may be used under the

test conditions laid down for non-permanent gases with a test pressure of 3000 lb./in.² A formula for thickness is given. Cylinders should be annealed at 820—850°, and those containing poisonous or inflammable gases must have the valves protected. Tensile, impact, and pressure tests are described, together with a hydraulic stretch-test at 3000 lb./in.² The permanent stretch, as shown by the water-jacket method, should not exceed 10% of the total stretch. This test should be repeated every 2 years, and the cylinder at the same time should be cleaned and examined internally with an electric lamp. The maximum working pressure should be 1800 lb./in.²

C. IRWIN.

Chemical action in relation to fatigue in metals.

B. P. HAIGH (Inst. Chem. Eng., March 20, 1929, 15 pp.).

—The endurance of a test-piece is the number of stress cycles required to cause fracture. The fatigue limit is the limiting stress that can be reversed continually without finally causing fracture. It is not a fixed quantity for a given metal, but depends on the nature of previous treatment, type of stress, etc. Endurance and fatigue limits are affected by chemical action on the surface, even by water or air, and even if no visible corrosion occurs. Practical illustrations are given, and these conclusions were confirmed by laboratory tests on specimens moistened with various reagents. It was proved that this "conjoint action" accelerated fatigue, irrespective of corrosion. Discoloration of the fractured face is characteristic of fracture under conjoint action, and sometimes occurs in ordinary fatigue fractures, presumably owing to chemical action of the atmosphere. Experiments carried out in America on the reduction of the fatigue limit of alloy steels by fresh or salt water are described. Results were found not to vary with the size of the specimen. Solutions were not more active than was water in affecting the fatigue limit; some, e.g., sodium dichromate, are beneficial. Tests at higher temperatures suggest that air in solution in water may be of importance. The coating with grease etc. of a specimen tested in air greatly increases the fatigue limit. The crack commences internally though near the surface, the marginal zone breaking by shearing, though this zone may be very thin. It is held that fatigue cracking is not connected with plastic strain. Fatigue cracks are perpendicular to the line of greatest tension. The theory is put forward and discussed that fatigue cracks are due to fluid tensions being formed locally in the specimen by contractions associated with changes of state. This view renders the manner of chemical influences more readily understood.

C. IRWIN.

Work-softening and a theory of intercrystalline cohesion.

F. HARGREAVES and R. J. HILLS (Inst. Metals, March, 1929. Advance copy. 27 pp. Cf. B., 1928, 755).—The effects of cold-working and annealing on the ball-hardness has been investigated for the cadmium-zinc, aluminium-zinc, tin-silver, tin-copper, and lead-silver eutectic alloys, and also for a lead-tin alloy containing 14% Sn, and a ternary alloy of zinc (90%), aluminium (7%), and copper (3%). The conditions necessary for work-softening are that the alloy shall contain two constituents, both of which undergo

spontaneous annealing after working at room temperature. A theory of intercrystalline cohesion is suggested, according to which the boundary region consists of a definite transition pattern of atoms, which is always the same for the same two relative orientations, but differs with varying orientation. This transition pattern is distinct from the true amorphous metal of the Beilby theory, which may be produced in the boundary after sufficient straining. The theory is applied to a number of phenomena.

W. HUME-ROTHERY.

Structure of copper-tin alloys. W. BRONIEWSKI and B. HACKIEWICZ (Rev. Mét., 1928, 25, 671—684; 1929, 26, 20—28).—A full account of experimental work the results of which have already been published (cf. A., 1928, 1327). A further study of the β - and γ -phases in the copper-tin system has yielded results which can be most satisfactorily explained by the assumption that the dissociation of the β -phase is due to the presence of the compound Cu_5Sn . Alloys containing more copper than corresponds with this formula undergo a transformation at 525° , whereas in those with more tin this occurs at 510° .

A. R. POWELL.

Resistance of zinc to indentation. J. N. FRIEND and W. E. THORNEYCROFT (Inst. Metals, March, 1929. Advance copy. 6 pp.).—A machine by which the effect of varying load, duration of application, and temperature on the deformation of soft metals can be determined consists essentially of a steel bar supported on a fulcrum at one end and carrying a collar on the underside of which is affixed a hard steel cone (45°). The cone rests on a small block of the metal to be tested, which is contained in a marble trough with two glass sides, filled with liquid paraffin and heated electrically. With rolled 99.9% zinc under constant load L , at constant temperature the depth of indentation, D , varied with the time θ according to the equation $D = K\theta^m$, where K represents the depth of indentation when L acts for unit time. K varied with temperature and load, m with temperature only. A break in a smooth curve was indicated at about 160° , interesting in view of the supposed allotropic change of zinc at 160 — 180° .

C. A. KING.

Recrystallisation phenomena of zinc. A. E. VAN ARKEL and J. J. PLOOS VAN AMSTEL (Z. Physik, 1928, 51, 534—544).—Peculiarities in the behaviour of zinc when recrystallisation is brought about by cold-working (cf. Masing, A., 1926, 564) have been shown to be due to large crystal formation, for the production of which the time interval between the rolling and bending of the zinc must be very short. It is shown that the recrystallisation phenomenon in zinc is qualitatively comparable with that of aluminium. The difference arises from the circumstance that cold-working will produce recrystallisation of zinc at room temperatures. G. E. WENTWORTH.

Conjugated influence of velocity of deformation and temperature in cold-hardening process [of aluminium]. A. ROUX and J. COURNOT (Compt. rend., 1929, 188, 631—633).—Aluminium is cold-hardened by instantaneous rupture at 380 — 450° and the metal exists in the hardened state above the normal annealing temperature. For each temperature there is a velocity of

deformation above which simultaneous recrystallisation does not immediately annul the hardening effect of deformation (cf. B., 1925, 994; A., 1927, 1019).

R. BRIGHTMAN.

Age-hardening of some aluminium alloys.

M. L. V. GAYLER and G. D. PRESTON (Inst. Metals, March, 1929. Advance copy. 44 pp.).—Five typical aluminium alloys, containing copper or magnesium silicide or both, were annealed at 500° for one or more days, quenched in cold water, and then aged at normal or higher temperatures, and the ageing process was followed by measurement of Brinell hardness, tensile strength, density, electrical conductivity, and crystal structure as revealed by X-ray analysis. The results of the hardness measurements are in agreement with those of Archer and Jeffries (Trans. Amer. Inst. Min. Met. Eng., 1925, 21, 828), and there is a critical heat-treatment temperature at about 150° , which corresponds to a maximum hardness. Heat-treatment at 200° causes a decrease in density, both for specimens treated immediately after quenching from 500° , and for those aged at room temperature, but the exact form of the curves connecting density with time of heat-treatment is not always the same. With alloys heat-treated directly at 200° , maximum values for electrical resistance occur at the same times as the maxima on the hardness curves, but with alloys aged at room temperature the maximum hardness is reached in 2—3 days, whilst the maximum resistance is reached after 1—3 hrs. The density measurements and X-ray analysis indicate that during ageing, precipitation occurs from solid solution, and that the crystals are in a disturbed state, which may be gradually relieved by ageing at higher temperatures. The precipitation process probably consists first in the rejection of solute atoms, which may then form molecules, and secondly the coagulation of the molecules to form small crystals of CuAl_2 or Mg_2Si , or both, according to the composition of the alloy. The alloys used had the following compositions, the balance being aluminium:—(1) Mg 0.85%, Si 0.5%; (2) Mg 0.51%, Cu 4.0%, Si 0.3%; (3) Cu 4.5%; (4) Mg 0.425%, Cu 2.0%, Si 0.25%; (5) Mg 0.68%, Si 0.4%.

W. HUME-ROTHERY.

Equilibria in the reduction, oxidation, and carburisation of iron. VII. Cobalt in an atmosphere of methane and hydrogen; the solid solution range. R. SCHENCK and H. KLAS (Z. anorg. Chem., 1929, 178, 146—156; cf. A., 1928, 844).—The equilibria in the system methane-hydrogen-cobalt-cobalt carbide have been determined in a similar manner to that used in the examination of the corresponding iron system; the results obtained are very similar. The pearlite point in the cobalt system is at 685° , and the temperature at which the solid solution of carbide in metal can co-exist with carbon is 695° . Cobalt carbide appears to be more stable than iron carbide in an atmosphere of hydrogen and methane, but the amount of carbon which cobalt can hold in solid solution is much less than in the case of iron.

A. R. POWELL.

Silver contents of specimens of ancient and mediæval lead. J. N. FRIEND and W. E. THORNEYCROFT (Inst. Metals, March, 1929. Advance copy.

12 pp.).—Determinations of the silver content of 20 specimens of ancient, Roman, and mediæval lead showed that pre-Roman lead was not desilverised, although considerable variation occurred in their silver content, a variation which occurred in mediæval samples, some of which, however, were really of Roman origin. Spartan votive figurines (700—500 B.C.) contained 0.0568% Ag.

C. A. KING.

Properties of eutectics and eutectoid alloys in binary metallic systems. P. SALDAU (Inst. Metals, March, 1929. Advance copy. 21 pp.).—Discontinuities in the electrical conductivity-temperature curves indicate the following transition points for pure metals: tin, 145° and 162°; lead, between 155° and 160°; zinc, 340° and 200—210°. By annealing alloys for 6 weeks at 185°, the solubility of zinc in tin was shown to lie between 1 and 3 at.-% zinc. The hardness, electrical resistance, and temperature coefficient of resistance have been measured for alloys in the neighbourhood of the eutectic composition in the systems tin-lead, lead-antimony, tin-zinc, gold-zinc, and gold-cadmium, and also for annealed eutectoid steels. At all temperatures the eutectic or eutectoid alloys correspond to a maximum on the resistance and hardness curves, and to a minimum on the temperature coefficient curves. These abnormalities are regarded as due to the finer dispersion of the constituents in the eutectic or eutectoid. Coalescence of the eutectic or eutectoid on annealing only takes place if one of the constituents be present in excess.

W. HUME-ROTHERY.

Alloys of zirconium. II. C. SYKES (Inst. Metals, March, 1929. Advance copy. 11 pp.; cf. Allibone and Sykes, B., 1928, 410).—The specific electrical resistance and its temperature coefficient have been measured for copper-zirconium, nickel-zirconium, and iron-zirconium alloys containing small amounts of zirconium, and the magnetic saturation induction was measured for the alloys with iron and nickel. The addition of zirconium to the above metals has no beneficial effect, the resistance being increased and the saturation induction diminished. Powdered zirconium alloys rather violently with aluminium, and alloys containing up to 70% Zr have been made. They consist of a dark constituent, which in low-percentage alloys crystallises in long needles, embedded in nearly pure aluminium. The hardness increases with the zirconium content, but the alloys have a coarse structure, resulting in low ductility and no appreciable increase in tensile strength, whilst their electrical resistance and corrodibility in sea-water are greater than those of aluminium and its more common alloys. Silver-zirconium alloys containing up to 30% Zr have been prepared, and consist of an angular constituent, probably an intermetallic compound, embedded in a silver-zirconium matrix. Annealing at 800° produces little change in structure, and the solid-solubility is small. The hardness increases rapidly up to 1% Zr, and then more gradually.

W. HUME-ROTHERY.

Analysis of metals and ores by heating in a current of chlorine. R. WASMUHT (Chem. Fabr., 1929, 121—122).—The reaction tube of hard glass or silica has connexions for the supply of pure, dry chlorine

or nitrogen, and at the outlet end is coupled to a sublimation flask for collecting the chloride. A bent bulb tube retains any chloride carried further forward. The exit gas then passes a sulphuric acid seal and enters a fume-pipe beneath which stands a basin of ammonia. The furnace is in three units for complete temperature control. The nitrogen used for sweeping out the apparatus is freed from oxygen by sodium hyposulphite solution.

C. IRWIN.

Action of air-carbon tetrachloride vapour mixtures on certain natural and artificial sulphides, selenides, and a telluride. K. BRADDOCK-ROGERS (Chem. News, 1929, 138, 147—152).—A series of natural and artificial sulphides, selenides, and a telluride (altaite) were heated to redness in a current of pure dry air containing carbon tetrachloride vapour, and the issuing gases were passed through bromine-hydrochloric acid absorption flasks, then through two flasks containing ammoniacal hydrogen peroxide. In all cases complete decomposition of the substance took place, and all the sulphur, selenium, and tellurium was converted into chlorides, which collected in the absorption vessels. For the determination of these elements in the material tested the residual chlorides in the decomposition tube were always dissolved in acid and added to the contents of the absorption flasks, and accurate results were then obtained. No figures are given showing whether these residues retained any sulphur, selenium, or tellurium.

A. R. POWELL.

Influence of surface conditions on the friction of metals. R. B. DOW (Physical Rev., 1929, [ii], 33, 252—257).—An improved tilt method was used to determine accurately the static coefficient of friction for metallic surfaces under varying surface conditions in different media. The presence and nature of oxide films materially affect the rise of the friction curve; a thin film acting as an abrasive causes the curve to rise, the rate depending on the normal force, until a layer of increased stability is reached. In terms of surface flow this indicates the formation of a vitreous layer. Thick films of certain oxides act as lubricants of great stability. Area of rubbing, shape of surfaces, and dry gases have no influence on μ ; moist inert gases show a capillary effect; moist active gases destroy the surfaces. The value of μ is found to be a linear function of the number of slips of one surface over another.

N. M. BIGH.

Clean coke and its value. MOTT.—See II. Blast-furnace slag in concrete. KOSFELD.—See IX.

PATENTS.

Smelting of ores in reverberatory furnaces. G. W. PRINCE, ASSR. to UNITED VERDE EXTENSION MINING Co. (U.S.P. 1,700,515, 29.1.29. Appl., 28.10.27).—The ore is deposited along the bottom of the furnace from points in the roof some distance from the side walls, and fettling material is blown into the space between the ore heaps and the side walls from the firing end of the furnace so as to protect the walls from slagging.

A. R. POWELL.

Rotary concentrator for ores etc. A. R. BROWN and J. I. YEATS (B.P. 304,508, 12.3.28).—A rotating cylinder is provided with a series of annular baffles,

on the feed side of which concentrate may be removed through openings provided with removable or trap covers.

C. A. KING.

Metallurgical furnace. A. J. BRIGGS, Assr. to ONONDAGA STEEL CO., INC. (U.S.P. 1,697,259, 1.1.29. Appl., 21.4.26).—The furnace comprises a working chamber rotating about a horizontal axis, and stationary electrodes entering from opposite ends of the chamber, their axis being above the axis of the chamber.

B. M. VENABLES.

Open-hearth furnace. F. R. MCGEE (U.S.P. 1,691,982, 20.11.28. Appl., 9.8.26).—An open-hearth furnace is provided with at least one regenerative chamber the lower part of which has a series of transverse walls forming chambers for the collection of dust, whilst the upper part contains a number of parallel longitudinal walls forming a series of flues along which the gas passes; occasional openings in the walls serve to equalise the pressure and rate of flow of the gas throughout the chamber.

A. R. POWELL.

Means for carrying off gases from rotary tubular furnaces for gas-treatment of ores. C. P. DERUCH (B.P. 304,937, 22.2.28).—The gas-discharge outlet from the furnace is connected to the furnace by means of a water-cooled, sleeve-like ring secured to the head of the furnace, the inner surface of which has a sliding contact with the enlarged end of the pipe.

A. R. POWELL.

Sintering machines. Apparatus for roasting or sintering of ores etc. NAT. PROCESSES, LTD., and G. E. STORER (B.P. 306,674 and 306,787, [A] 25.1.28, [B] 21.7.28).—(A) The circular travelling grate and gas box beneath of a rotary roasting furnace are divided into a number of independent segments, each of which may be overturned at the point of discharge. The turning movement may be radial or tangential, suitable gland joints being provided to maintain gas-tightness, and a blow is delivered to the segment to assist in the discharge of the roasted material. (B) Several gas boxes are provided so that rich or poor gas may be drawn and collected as required in separate collectors, these chambers terminating in hoppers for the collection of dust.

C. A. KING.

Manufacture of metal pots for heat-treating and carburising metal objects etc. FLANNERY BOLT CO., Assees. of F. K. LANDGRAF and E. S. FITZSIMMONS (B.P. 306,346, 6.6.28. U.S., 27.3.28).—The pot consists of an inner and outer shell, the outer being adapted to withstand scaling at a high temperature and the inner to withstand the corrosive action of the material to be heated. *E.g.*, caustic soda pots of wrought iron are provided with a welded layer of nickel-chromium-iron alloy outside, or a pot made of nickel-chromium alloy may have an inner lining of wrought iron applied by means of a welding operation.

A. R. POWELL.

Production of iron powder of high purity and small grain size. J. Y. JOHNSON. From I. G. FARBER-IND. A.-G. (B.P. 306,215, 2.12.27).—Iron powder prepared from iron carbonyl and containing about 1.6% C is treated in a thin layer at 500° with hydrogen until no further methane or water is evolved. The mass is

cooled in hydrogen, finely ground, and re-heated in hydrogen at 500–600° for several hours to remove the last traces of carbon. The product has a permeability of 25–32 and very low hysteresis losses; it is, therefore, specially suitable for the manufacture of cores for Pupin coils.

A. R. POWELL.

Desulphurisation of pyrite cinders. I. CAVALLI (U.S.P. 1,703,027, 19.2.29. Appl., 27.9.27. Italy, 7.10.26).—The cinders are treated with alkali hypochlorite solution.

H. ROYAL-DAWSON.

Production of unhardened [white iron] castings in metal moulds. H. DOAT, Assr. to COMP. GÉN. DES CONDUITES D'EAU, SOC. ANON. (U.S.P. 1,699,612, 22.1.29. Appl., 26.10.25. Belg., 4.11.24).—In the production of white-iron castings in centrifugal moulds a quantity of fine-grained ferrosilicon is introduced into the mould prior to casting so that it is held against the sides of the mould by centrifugal force. This lining protects the mould during casting and becomes incorporated with the outer portion of the cast metal, thereby converting this portion of the white iron into grey iron containing flakes of graphite and very little cementite.

A. R. POWELL.

Chilled cast-iron roll. C. I. NIEDRINGHAUS and L. W. MESTA, Assrs. to MESTA MACHINE CO. (U.S.P. 1,702,128, 12.2.29. Appl., 19.5.27).—The alloy comprises iron with 2–4% C, 1.8–4.0% Cr, 0–0.3% P, 0–0.2% S, 0.1–1.5% Mn, and 0.5–2.5% Si, the silicon, carbon, and chromium contents being so proportioned that the chill castings produced from the alloy contain only a small but appreciable proportion of graphite in the outer layers and have a structure changing gradually from the outside to the core, which has a mottled iron structure.

A. R. POWELL.

Treatment of steel. H. J. BIEFANG (U.S.P. 1,699,683, 22.1.29. Appl., 7.5.23).—High-speed steel is heat-treated in a bath comprising 7 pts. of lead and 1 pt. of copper at a temperature between the m.p. of the bath and 1000°, immersion for 20 min. being required for every 0.5 in. of thickness.

A. R. POWELL.

Manufacture of alloy steels. J. L. ANDREN (U.S.P. 1,699,731, 22.1.29. Appl., 19.12.25).—A mixture of iron ore, an ore of the alloying element, and carbon sufficient to reduce the contained oxides and to supply the desired proportion of carbon to the final alloy is heated at a temperature sufficient to effect reduction but not to melt the alloy, and when reduction is complete the temperature is raised until the mass melts.

A. R. POWELL.

Ferrous base article of manufacture [case-hardened molybdenum steel]. W. H. PHILLIPS, Assr. to MOLYBDENUM CORP. OF AMERICA (U.S.P. 1,697,083, 1.1.29. Appl., 13.7.28).—Steel containing up to 4% Mo and 0.1–0.4% Al when case-hardened in nitrogen at 700° has a hard and tough surface which is highly resistant to corrosion.

A. R. POWELL.

Corrosion-resistant ferrous alloys and their manufacture. H. L. MILLER (B.P. 306,320, 13.4.28).—The alloy comprises iron with less than 0.2% C, 0.2–2.0% Cu, up to 0.2% Mo, 0.2–1% Mn, and up to 0.5% Si. It is deoxidised in the ladle by means of

aluminium in such quantities that a trace of this metal remains in the final alloy. A. R. POWELL.

Removing rust, scale, dirt, or grease from iron and steel articles. W. H. COLE (B.P. 306,604, 22.8.27. Cf. B.P. 298,328; B., 1928, 898).—The articles are immersed in a dilute solution of phosphoric acid in an autoclave, and heated for 10 min. to a pressure of about 50 lb./in.² (140°). H. ROYAL-DAWSON.

Removal of copper oxide from copper. H. S. LUKENS and R. P. HEUER (U.S.P. 1,698,443, 8.1.29. Appl., 31.3.24).—Copper is melted below a chloride slag containing alkali phosphates, borates, or silicates which dissolve the oxide from the copper. The slag may be electrolysed while still over the copper to effect reduction of the dissolved copper oxide and so make the process continuous, or, if the slag has absorbed metallic impurities from the copper, it may be withdrawn into a separate furnace for electrolysis to recover a crude copper which is returned to the ordinary refining process. A. R. POWELL.

Solvent treatment of copper silicate ores. ANGLO-AMERICAN CORP. OF S. AFRICA, LTD. (B.P. 301,859, 22.5.28. S. Afr., 7.12.27).—Copper silicate ores are ground wet to pass 200-mesh, the pulp is dewatered to 20% moisture in a pressure filter, and a solution of ammonium carbonate is forced through the mass to leach out the copper. A. R. POWELL.

Manufacture of nickel and nickel alloys. INTERNATIONAL NICKEL CO., ASSEES. OF P. D. MERICA and A. E. KAYES (B.P. 290,647, 24.11.27. U.S., 19.5.27).—Nickel or monel metal is treated in the molten state with nickel oxide until all the carbon is burnt out and the carbon monoxide is expelled by "boiling." The metal is then deoxidised with silicon and finally a little magnesium is added just prior to casting. A. R. POWELL.

Solder. J. SILBERSTEIN, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,699,716, 22.1.29. Appl., 21.5.27).—A lead alloy containing 0.25–5.0% Ag, 0.25–10% Cu, and minor impurities is claimed. A. R. POWELL.

Metal cement. W. R. CHAPIN (U.S.P. 1,699,346, 15.1.29. Appl., 1.9.27).—The cement consists of about 60% of silico-manganese, 10% of ferrosilicon, up to 25% of nickel, and up to 10% of borax glass. The materials are all finely ground before being mixed. H. ROYAL-DAWSON.

Production of metal-powder articles of spherical shape. E. PONSZUS and E. KRAMER, ASSRS. to HARTSTOFF-METALL A.-G. (HAMETAG) (U.S.P. 1,699,205, 15.1.29. Appl., 16.7.26. Ger., 10.10.25).—The edges of flake-shaped particles are gradually turned over in a suitable mill until they form hollow spheres. H. ROYAL-DAWSON.

Indicating the size of the particles in ore pulp flowing in conduits. H. E. T. HAULTAIN (U.S.P. 1,697,867, 8.1.29. Appl., 12.9.22).—A portion of the pulp is continuously by-passed from the conduit through a U-tube provided with a manometer device to each arm so that the sp. gr. of the pulp in the downward stream is compared with that in the upward stream by comparing the manometer readings. Variations in the

average size of the particles are accompanied by changes in sp. gr. of the two streams owing to the classifying action of the U-tube. A. R. POWELL.

Metallurgical furnace. D. CUSHING (U.S.P. 1,703,337, 26.2.29. Appl., 19.7.26).—See B.P. 303,042; B., 1929, 175.

Blast furnace. Protection of blast-furnace jackets. J. P. DOVEL (U.S.P. 1,703,518, 26.2.29. Appl., [A] 28.7.27, [B] 16.3.28).—See B.P. 299,941; B., 1929, 23.

Heat-treatment of metal articles. F. TWYMAN, ASSR. to A. ILLGER, LTD. (U.S.P. 1,702,985, 19.2.29. Appl., 31.3.28. U.K., 27.1.27).—See B.P. 291,483; B., 1928, 575.

Treatment of ores for extracting metallic values therefrom. F. DIETZSCH (U.S.P. 1,703,763, 26.2.29. Appl., 31.10.27. U.K., 8.12.26).—See B.P. 287,207; B., 1928, 373.

Coating cast iron with lead. O. SPENGLER (U.S.P. 1,703,019, 19.2.29. Appl., 2.10.26. Ger., 28.11.25).—See B.P. 260,452; B., 1926, 1018.

[Lead]-tungsten [bearing-metal] alloy. H. FALKENBERG (U.S.P. 1,703,577, 26.2.29. Appl., 2.12.25. U.K., 8.12.24).—See B.P. 247,687; B., 1926, 368.

Pulverising process [for metals]. E. PODSZUS, ASSR. to HARTSTOFF-METALL A.-G. (HAMETAG) (U.S.P. 1,703,634, 26.2.29. Appl., 24.4.25. Ger., 28.4.24).—See B.P. 232,993; B., 1925, 853.

[Stirring device for] production of pig iron. BRADLEY & FOSTER, LTD., and R. P. BETHELL (B.P. 306,670, 16.1.28).

Composite materials containing metal foil. L. RADO (B.P. 306,225, 31.8.27).

Gas-fired furnaces (B.P. 302,714).—See I. Insulated coverings on aluminium (B.P. 287,503).—See XI.

XI.—ELECTROTECHNICS.

Conduction in textiles. MURPHY. Soda-pulp manufacture (WALLENBERGER).—See V. Sulphates in drinking water. FEHN and others.—See XXIII.

PATENTS.

Electrical induction furnace. N. R. DAVIS, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (B.P. 306,707, 23.2.28).—In an electrical induction furnace of the type in which a coil surrounds the charge to be heated and does not embrace an iron core, the coil is provided with tapping points, to any one of which connexion can be made as desired, and the amount of furnace coil connected in circuit is varied during progressive heating of the furnace so that the effective inductance of the furnace coil is maintained substantially constant. J. S. G. THOMAS.

Electrodes for arc-welding etc. WELDRICS (1922), LTD., and A. D. ANDERSON (B.P. 306,785, 17.7.28).—An iron core, wound with a mixture of blue and white asbestos, is coated with a paste consisting of not less than 95% (preferably 98%) of blue asbestos and not more than 5% of sodium silicate. Alternatively, a small proportion of the sodium silicate (about 2%) is replaced by iron oxide or china clay. J. S. G. THOMAS.

Electrolytic apparatus. W. G. ALLAN, Assr. to F. G. CLARK (U.S.P. 1,702,924, 19.2.29. Appl., 10.11.23. Renewed, 23.5.27).—Anode and cathode compartments separated by a porous diaphragm are connected with anolyte and catholyte separating chambers by conduits the heights of which above the level of electrolyte in the cell compartments can be adjusted in order to regulate the total pressures in the anode and cathode compartments. J. S. G. THOMAS.

[Filling for] gas-filled electric incandescence lamps. GEN. ELECTRIC CO., LTD., F. J. G. VAN DEN BOSCH, and N. R. CAMPBELL (B.P. 306,612, 28.11.27).—A gas-filling contains 0.015–0.4% by vol. of hydrogen phosphide, introduced by passing the filling gas over hydrogen phosphide at a temperature near that of boiling oxygen. Flashing is thus prevented. J. S. G. THOMAS.

Production of insulated coverings on wires, bands, plates, or sheets of aluminium or aluminium alloys. SPEZIALFABR. F. ALUMINIUM-SPULEN U. -LEITUNGEN GES.M.B.H. (B.P. 287,503, 19.3.28. Ger., 22.3.27).—The articles to be treated are passed over rollers rotating in a bath containing oxalic, nitric, or chromic acid to produce an oxide coating which is flexible.

A. R. POWELL.

Manufacture of ozone. W. ESMARCH, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,702,254, 19.2.29. Appl., 12.7.27. Ger., 26.8.26).—See B.P. 276,637; B., 1928, 338.

[Spiral tungsten] filaments for electric incandescence optical projection lamps. FALK STADELMANN & CO., LTD., Assees. of NEUE GLÜHLAMPEN GES. M.B.H. (B.P. 301,015, 24.7.28. Ger., 23.11.27).

Acetylene from gases (B.P. 306,008). Purification of gases (B.P. 289,885).—See II. Bismuth compounds (B.P. 298,587).—See VII. Iron powder (B.P. 306,215).—See X.

XII.—FATS; OILS; WAXES.

Component glycerides of cacao butter. C. H. LEA (J.C.S.I., 1929, 48, 41–46 T).—Oxidation in acetone solution with potassium permanganate of a sample of cacao butter (the mixed fatty acids of which contained 24.4% of palmitic, 34.5% of stearic, 39.1% of oleic, and 2.0% of linoleic acids) showed that only 2.5% of fully saturated glycerides was present, whence it follows that the molecular ratio of saturated acids linked with unsaturated acids in the mixed glycerides is 1.4 : 1. This proportion is very similar to that observed by Collin and Hilditch (B., 1928, 791) in the case of the mixed glycerides of coconut and palm-kernel fats. It also follows from the above data that the content of mono-oleo-disaturated glycerides must lie between 73% and 85%, whilst that of dioleo-monosaturated glycerides cannot exceed 24.5%, or that of triolein 12.5%. These conclusions are supported by the results of a study of the acidic products of oxidation. Fractional separation of the latter in the form of lithium salts indicated that more than 70% of the cacao butter was present in the form of monoazelaoglycerides, and fairly pure monoazelaoglycerides, corresponding to 53–54% of the

original fat, were isolated by this procedure. It was also found that the sodium salts of the monoazelaoglycerides were soluble in warm ether, but insoluble in ice-cold ether, and by this means an alternative isolation of almost pure monoazelaoglycerides, in amount corresponding to about 53% of the original fat, was obtained. Fractional crystallisation of the purified monoazelaoglycerides indicated that in the original fat one molecule of oleodistearin was associated with 5–6 molecules of oleopalmitostearin. Oleopalmitostearins are undoubtedly the predominating component of cacao butter, and determine its characteristic physical properties. There is some evidence that small amounts of triolein are present, but the content of this glyceride can hardly exceed 4%. It is believed that the following general estimate of the component glycerides present in cacao butter may be put forward with some confidence: Fully saturated glycerides (mainly mixed palmitostearins), 2.5%; mono-oleo-disaturated glycerides (oleodistearins, probably about 10%; oleopalmitostearins, at least 50–60%), 77%; dioleo-monosaturated glycerides (probably mainly dioleostearins), 16%; triolein (?), 4%.

Component glycerides of a mutton tallow. G. COLLIN, T. P. HILDITCH, and C. H. LEA (J.S.C.I., 1929, 48, 46–50 T).—Oxidation in acetone solution with potassium permanganate of a sample of mutton tallow (the mixed fatty acids of which contained 4.6% of myristic, 24.6% of palmitic, 30.5% of stearic, 36.0% of oleic, and 4.3% of linoleic acids) has shown that the structure of the component glycerides present is widely different from that of cacao butter, although the fatty acid mixture in each case is very similar in type. The mutton tallow contained 26% of fully saturated glycerides, the mixed fatty acids of which were composed of 6.1% of myristic, 50.2% of palmitic, and 43.7% of stearic acids. Fractional crystallisation of the fully saturated glycerides showed that a complex mixture was present; no individual glyceride was isolated in the pure condition, but evidence of tristearin, palmitodistearins, and dipalmitostearins was obtained. In the mixed saturated–unsaturated glycerides of the tallow, 0.9 equivalent of saturated acid was linked with 1 equivalent of unsaturated acid. It follows that there cannot be less than 30%, or more than 52%, of mono-unsaturated–di-saturated glycerides in the original tallow, whilst the amount of di-unsaturated–mono-saturated derivatives cannot exceed 44%, and that of tri-unsaturated glycerides 22%. The mixture of acidic glyceride derivatives, corresponding to the original mixed saturated–unsaturated glycerides, did not prove so amenable to separation as in the case of the similar cacao butter products. The general indications obtained led to the inference that there was probably a fairly large amount of di-unsaturated glycerides present and, correspondingly, a relatively small percentage of triolein, whilst the amount of mono-unsaturated glycerides is probably closer to the lower than to the upper limit given. Quite possibly the composition of the glycerides is not far removed from the following: fully saturated, 26%; mono-unsaturated–disaturated, about 35%; di-unsaturated–mono-saturated, about 35%; tri-unsaturated, about 4%.

Composition of a rape oil of German origin. K. TÄUFEL and C. BAUSCHINGER (Z. Unters. Lebensm., 1928, 56, 253—264).—From an alcoholic solution of the fatty acids from rape oil a fraction was precipitated by lead acetate consisting of all the saturated acids and part of the erucic acid. A second fraction precipitated from the alcoholic filtrate by magnesium acetate consisted of magnesium erucate. In the third fraction consisting of the filtrates from the precipitation of the second fraction and from the recrystallisation of the free erucic acid from alcohol, α -linoleic and α -linolenic acids with smaller quantities of the β -isomerides were identified by bromination methods. Oleic acid was isolated by Grabner's method (B., 1921, 896 A). The composition of the third fraction was calculated from the iodine value, mean mol. wt., and the assumption of 5% content of linolenic acid from the quantity of hexabromostearic acid isolated. The composition of rape oil was found to be: glyceryl residue (C_3H_5) 3.8%, unsaponifiable matter 1.0%, saturated fatty acids 0.8%, erucic acid 43.5%, oleic acid 37.8%, linoleic acid 10.6%, linolenic acid 3.5%. W. J. BOYD.

Glycerides of rape oil. K. TÄUFEL and C. BAUSCHINGER (Z. Unters. Lebensm., 1928, 56, 265—272).—The presence of 1.7% of oleolinolenocerin in rape oil was proved by the bromination method, and the presence of oleodierucin (cf. Amberger, *ibid.*, 1920, 40, 192) and trierucin (cf. Reimer and Will, A., 1887, 233, 1030) was confirmed by the elaidin methods of Tomow (Diss., München, Techn. Hochschule, 1914) and of Heiduschka and Felser (B., 1920, 272 A). W. J. BOYD.

Composition of California walnut oil. G. S. JAMIESON and R. S. MCKINNEY (Oil & Fat Ind., 1929, 6, 21—23).—The crude oil had d_{20}^{25} 0.9235, n_D^{25} 1.4751, acid value 5.11, iodine value (Hanus) 158.5, saponif. value 194.5, acetyl val. 6.09, Reichert-Meissl value 0.11, hexabromide 8.88%, unsaponifiable matter 0.51%, saturated acids 5.34% (corr.), unsaturated acids 89.74% (corr.), iodine value of unsaturated acids 166.7. The high iodine value is notable, otherwise the characteristics are similar to those reported for European walnut oils (iodine value 138—148). No lauric acid was found, and only traces of myristic acid, the composition of the oil being determined as glycerides of oleic acid 17.6%, linoleic acid 72.8%, linolenic acid 3.2%, myristic acid trace, palmitic acid 4.6%, stearic acid 0.9%, arachidic acid trace, unsaponifiable matter 0.5%. E. LEWKOWITSCH.

Apricot-kernel oil as substitute for almond oil. G. KOGAN (Chem. Umschau, 1929, 36, 62—63).—The only appreciable difference between the characteristics of peach-kernel, apricot-kernel, and almond oils is shown by the m.p. of the fatty acids, apricot-kernel fatty acids having f.p. 2—4°, m.p. 6—8°, whilst those from peach-kernel and almond oils give higher values. Apricot-kernel oil is superior to almond oil in that the acid value of the commercial oil (several hundred samples) does not exceed 2. Apricot-kernel oil is suitable for subcutaneous injection, and it is suggested that since peach-kernel oil practically never appears in commerce, the former should be termed *Oleum Armeniaceae*.

E. LEWKOWITSCH.

Thiocyanometry of parsley-seed oil. A. STEGER and J. VAN LOON (Z. Unters. Lebensm., 1928, 56, 365—367).—By means of the "thiocyanometric" iodine value of Kaufmann (B., 1926, 447) more accurate values for the contents of linoleic acid and saturated acids in parsley-seed oil are obtained. The content of the former is found from the difference between the Wijs iodine value and the thiocyanogen-iodine value, only one of the ethylenic linkings in linoleic acid being attacked by thiocyanogen. The value for saturated acids is deduced from the reduction in the thiocyanogen-iodine value due to dilution with saturated acids, this value being shown to be 90 in the absence of these substances. Parsley-seed oil was found to contain unsaponifiable matter 30%, saturated acids 3.0%, petroselinic acid 45%, "9:10"-oleic acid 8.0%, linoleic acid 9.1%, glyceryl residue 2.8%, volatile substances 2%. W. J. BOYD.

Method for wool oil unsaponifiables. L. HART (Oil & Fat Ind., 1929, 6, 29).—A sample of wool oil (sufficient to give 10 c.c. of unsaponifiable oils) suspected of adulteration by mineral oils is saponified with alcoholic potash to separate the bulk of the fatty acids, and the mixture is extracted with ether. The extract is washed free from soap with 10% alcohol and 2% potash solution and the ether is evaporated; the extract is then treated with 40 c.c. of a mixture (1:2) of amyl and ethyl alcohols. On shaking, the wool-grease alcohols dissolve and any hydrocarbon oils separate, and can be examined for sp. gr. etc. The determination may be checked by refluxing 10 c.c. of the unsaponifiable material (after determining the sp. gr.) with twice its weight of acetic anhydride for $\frac{1}{2}$ hr.; the liquid is transferred to a separating funnel, the flask being rinsed with a minimum amount of acetic anhydride. If hydrocarbons are present the hot solution separates into two layers. The upper layer is separated, and washed, and its volume noted. In the absence of mineral oil the hot solution remains homogeneous. E. LEWKOWITSCH.

Polymerisation of linolenic acid and herring oil by heating. T. MAZUME and G. SHOBAYASHI (J. Soc. Chem. Ind., Japan, 1928, 31, 746—748).—On heating the methyl ester of linolenic acid for 6—10 hrs. at 300° in an atmosphere of hydrogen, its mol. wt. was doubled and the iodine value fell to about half of its original value, thus indicating the polymerisation of 2 mols. of the acid. Similar experiments with herring oil showed that the decrease of iodine value does not exactly correspond to the increase of mol. wt.; the authors suggest that in this case an extra-molecular polymerisation had occurred. Y. TOMODA.

Increase in acidity of whale oil during its extraction. S. STENE (Tidsskr. Kjemi Berg. 1929, 9, 9—12, 24—27; cf. Heyerdahl, Norsk Hvalfaugstid., 1925, 14, 19, 113).—In the "trying out" process where steam under pressure is employed, the acidity of the oil obtained first decreases, then remains constant for a period, and finally exhibits a steady increase. The first two stages are influenced by various factors which are inherent in the process and cannot easily be controlled, but an attempt has been made to obtain a mathematical expression representing the third. The reaction velocity

involved in this may be calculated from the formula $\log_e (x_2/x_1)[(100 - x_1)/(100 - x_2)] = K(t_2 - t_1)$, where x_1, x_2 are the percentages of oleic acid at times t_1, t_2 . It is concluded that the increase in acidity is an autocatalytic process (fat + water \rightarrow fatty acid + glycerol, catalysed by hydrogen ion) which takes place in the homogeneous oil phase of the mixture in the extraction vessel. Values of x calculated from the above formula agree satisfactorily with the observed values obtained in a series of experiments. H. F. HARWOOD.

Palmitic acid in fusel oil. KINO.—See XVIII.

PATENTS.

Manufacture of fatty acids. H. WEYLAND and S. DEICHSEL, Assrs. to WINTROP CHEM. CO., INC. (U.S.P. 1,703,129, 26.2.29. Appl., 4.4.25. Ger., 11.4.24).—See G.P. 422,687; B., 1926, 287.

Separating fluids from solids (B.P. 303,649).—See I. **Hardening of waxes** (B.P. 306,053).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

The red lead question. H. WOLFF and G. ZEIDLER (Farben-Ztg., 1929, 34, 1309—1310).—Junk's observations (B., 1929, 103) are adversely criticised on various grounds: e.g., aggregates and not primary particles are measured by the method adopted; bulking value and sedimentation value are not necessarily parallel; the chemical nature of the setting of red lead is not established; the existence of free glycerol in the red lead-linseed oil system has never been demonstrated; the samples of "disperse" red lead used by Junk do not represent the current quality of this type of material.

S. S. WOOLF.

Types of red lead. W. LÜTY (Farben-Ztg., 1929, 34, 1310—1312).—Adverse criticism of Junk's work (B., 1929, 103) is offered for similar reasons to those advanced by Wolff and Zeidler (preceding abstract). Further points developed are the failure to consider particle shape and nature of surface, and the wrong construction placed on "covering power" by Junk.

S. S. WOOLF.

Flash points of solvents and plasticisers. E. VON MÜHLENDahl (Farben-Ztg., 1929, 34, 1427—1429).—Various methods for determining flash points, and empirical relationships by which they may be calculated, are critically discussed. The flash points of various solvents and plasticisers and characteristic binary and ternary mixtures are tabulated, with the names of the observers. Most of these determinations have been made with the Ormandy-Craven apparatus, which is considered by the author to be the most suitable for the purpose.

S. S. WOOLF.

Autoxidation of French turpentine oils. A. A. BOLIN and A. TSCHIRCH (Pine Inst. Amer. Abstr. Chem. Sect., 1928, 2, 86—90).—A review and discussion.

CHEMICAL ABSTRACTS.

PATENTS.

Nitrocellulose solutions. W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 305,960, 12.10.27).—In the manufacture of nitrocellulose lacquers and enamels, methyl, ethyl, *n*- and *iso*-propyl ethers of

lactic esters, especially of ethyl lactate, are used as solvents. *Ethyl α -ethoxypropionate*, b.p. 135.5°, and *n-propyl α -*n*-propoxypropionate*, b.p. 187—188°, are described.

C. HOLLINS.

Manufacture of artificial resins. IMPERIAL CHEM. INDUSTRIES, LTD., R. HILL, and E. E. WALKER (B.P. 305,965, 11.11.27).—Resins of the "glyptal" type having low acid value are obtained by replacing the glycerol (or other polyhydric alcohol) by a mixture of alcohols one or more of which is polyhydric; e.g., phthalic anhydride is condensed with glycerol and glycol, or with glycerol and butyl alcohol, or with glycerol and cyclohexanol; or butyl hydrogen phthalate is condensed with glycerol; or a glycol-phthalic resin is fused with a glycerol-phthalic resin. Monobasic acids (e.g., drying-oil acids) may be used for part of the polybasic acid. C. HOLLINS.

Manufacture of abietic acid esters of polyglycerol. E. M. SYMMES, Assr. to HERCULES POWDER CO. (U.S.P. 1,696,337, 25.12.28. Appl., 12.11.27).—Glycerol is polymerised and the polyglycerol esterified by refluxing with abietic acid in presence of zinc dust, boric anhydride, or other esterification catalyst. Alternatively, the polymerisation may be effected in presence of the abietic acid. E.g., diglycerol, obtained by heating glycerol with zinc dust and sodium acetate (anhydrous) at 270—290°, when heated for 15 hrs. at 290° with 100 pts. of rosin affords, after removal of glycerol and diglycerol, a diglyceryl resinate, m.p. 107° (drop method), 85° (capillary method), acid value 12.

R. BRIGHTMAN.

Production of oil-soluble phenol-aldehyde condensation products. BAKELITE GES.M.B.H. (B.P. 282,414, 14.12.27. Ger., 14.12.26).—Oil-soluble resins of the hardening or non-hardening type are produced by combining under heat phenol-aldehyde condensation products with polyhydric or polycyclic phenols (e.g., α - or β -naphthol, resorcinol, diphenols, dicresols, etc.) or with molecular compounds of these. Hydrated oxide of manganese, lead oxide, resinsates of lead and/or manganese, etc. may be present as catalysts, and an oil, such as linseed oil, and a mild oxidising agent may be added.

S. S. WOOLF.

Manufacture of resin solutions. SOC. D'EXPLOIT. DE BREVETS ET PROCÉDÉS P.N. (B.P. 299,293, 20.1.28. Belg., 22.10.27. Addn. to B.P. 263,393; B., 1927, 147).—The caustic alkali used in the process previously described is replaced by other free inorganic or organic bases or by suitable salts thereof. The solutions obtained may be enriched by allowing them to react repeatedly with the resin after successive additions of base or salt.

S. S. WOOLF.

Acid-proof material (B.P. 276,697). **Cellulose ester compositions** (B.P. 304,814).—See V. **Plastic products** (B.P. 305,968).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber structure research and its bearing on the elastic properties of colloids in general. E. A. HAUSER (Ind. Eng. Chem., 1929, 21, 249—251).—Recent physico-chemical research on rubber structure is reviewed. Rubber is an isocolloid representing a solid

solution having the structure of β -rubber (*i.e.*, gel-rubber) swollen by the liquid α -rubber (sol-rubber), these two constituents being interconvertible by various means. Just as cellulose, by X-ray investigation, has been shown to comprise bundles of long chains linked together by main-valency linkings, the chains consisting of simple glucose rings joined in a diagonal helix, so rubber is regarded as containing main-valency isoprene chains disposed spirally, the helix becoming un wound when the rubber is stretched. Celluloid is analogous to rubber in giving "amorphous" scattering of X-rays in the unstretched condition, because under these conditions the nitrocellulose is swollen by the added camphor; when stretched, the camphor is expelled from the fibre structure and the original nitrocellulose structure is revealed. (Cf. B., 1929, 318.)

D. F. TWISS.

Absorption of oxygen by rubber. G. T. KOHMAN (J. Physical Chem., 1929, 33, 226—243).—The influence of various factors on the absorption at 80° has been studied in an attempt to correlate natural ageing and oxygen absorption. Absorption is autocatalytic, especially in the early stages, and the amount absorbed is generally greater than the theoretical amount required for saturation of the double linkings. A constant rate of absorption is finally reached, and two separate reactions, *viz.*, a decomposition and an addition, appear to be involved. The rate of absorption at 80° depends on the surface area when this is small, but is practically independent when the surface area is increased to 40 cm.², in which case diffusion is considered to be great enough to keep the particles of rubber saturated with oxygen. The changes which occur when oxygen is absorbed are similar to those which occur during vulcanisation with sulphur. The process of curing rubber increases the rate and the amount of oxygen absorbed when compared with the same phenomena for the raw material; the mechanisms involved also appear to be different and the character of the absorption curve changes. Absorption increases with an increase in the extent of curing. Anti-ageing materials act as negative catalysts and the extent of their effect varies with their concentration and nature. In one case (0.5% of anti-ageing material) absorption was accelerated. Ozonised oxygen is absorbed at first more rapidly than oxygen, but the rate of absorption quickly decreases and less oxygen is finally absorbed. Protection from the further action of pure oxygen appears to be conferred; the reaction is not autocatalytic as in the case with oxygen and rubber. The deterioration in tensile strength is considered to be proportional to the quantity of oxygen absorbed provided that well-cured rubber possesses sufficient strength to be of practical value. The absorption of 0.5% of oxygen (by wt.) decreases the tensile strength by 50%. When heated in nitrogen or carbon dioxide, under-cured rubber shows an initial increase in tensile strength due to a curing action, followed by a slow decrease which is of no significance compared with that suffered in oxygen. The present work indicates that natural ageing is essentially the result of absorption of oxygen from the atmosphere by rubber.

L. S. THEOBALD.

Nature of vulcanisation. IV. H. P. STEVENS and W. H. STEVENS (J.S.C.I., 1929, 48, 55—59 r).—The

limiting figure for combined sulphur is discussed, and it is shown that where ultra-accelerators are used it is possible to prepare vulcanised rubber containing more combined sulphur than that required by the formula C_5H_8S . It is thought that the excess may result from substitution of sulphur for hydrogen in the rubber molecule. These compounds were all prepared by vulcanisation at or below 100°. It was further found, in confirmation of Whitby's work (cf. B., 1926, 682), that the presence of rubber protein was without influence on the final coefficient. Vulcanisation in the presence of a common solvent for rubber and sulphur was also found to yield coefficients in excess of those for the formula C_5H_8S , although owing to the dilution of the reactants by the solvent the conditions were not so favourable for exhaustive vulcanisation. The brown-coloured material associated with the acetone extract of the vulcanite was found to be due to decomposition products of non-rubber matter and not to rubber sulphides. It is concluded that under suitable conditions vulcanisation will proceed further than the stage called for by the formula C_5H_8S , but that such excess vulcanisation is due to the substitution of sulphur for hydrogen in the rubber molecule.

Nature of vulcanisation. W. H. STEVENS (J.S.C.I., 1929, 48, 60—62 r).—Some of the recent work on the problem of vulcanisation is summarised. In particular attention is drawn to the theory of vulcanisation in which the distinctive properties of vulcanised rubber are regarded as brought about by a dispersion of rubber sulphide with raw rubber as the continuous phase. The action of accelerators is discussed from this viewpoint. The author considers that the shape of the vulcanisation coefficient-time curves can be largely explained on a solubility basis without introducing an auto-catalytic effect, and further compares accelerator action with the effect of a common solvent. The action of soaps formed from the rubber "resins" is considered to be similar.

Degree of devulcanisation and the evaluation of regenerated rubber. H. LOEWEN (Kautschuk, 1929, 5, 61—62; cf. Lindmayer, B., 1929, 181).—Although examination of reclaimed rubber by extraction with chloroform may yield useful results, the theoretical views expressed by Lindmayer as to the mechanism of "devulcanisation" were unsupported by experimental evidence and are contrary to existing knowledge.

D. F. TWISS.

PATENTS.

Manufacture of rubber compositions. GOODYEAR TIRE & RUBBER Co., Assees. of L. B. SEBRELL (B.P. 286,288, 11.1.28. U.S., 3.3.27).—Rubber compositions are vulcanised in presence of the cold reaction products of an aliphatic aldehyde and an aromatic amine, *e.g.*, anilinobutylideneaniline or anilinoxyldieneaniline.

R. BRIGHTMAN.

Production of plastic products from india-rubber. F. C. DYCHE-TEAGUE (B.P. 305,968, 13.9.27).—Rubber which has been thoroughly depolymerised by milling is dissolved in a suitable solvent, *e.g.*, carbon tetrachloride or benzene, and then chlorinated; additional depolymerisation, prior to chlorination, may be effected by subjecting the solution to actinic light. Distillation

removes solvent and hydrogen chloride, and the reaction product, which may contain 50–60% Cl, forms fluid solutions which provide alkali- and acid-resistant lacquers or paints. D. F. TWISS.

Production of goods made from aqueous dispersions of rubber and similar materials. DUNLOP RUBBER Co., LTD., and R. C. DAVIES (B.P. 306,621, 1.12.27).—Finely-divided fillers such as gas-black are incorporated in aqueous dispersions of rubber with the aid of a solution of cellulose xanthate as a dispersion medium; additional compounding ingredients may also be introduced, and the mixtures may be concentrated before application to such manufacturing processes as moulding, spraying, spreading, or electrophoretic deposition. D. F. TWISS.

XV.—LEATHER; GLUE.

Determination of acidity in tanning liquids. G. A. BRAVO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1929, 7, 45–52).—For determining the total acidity in tanning liquors, the use of 4–5 c.c. of 0.1% aqueous eosin, which changes colour between p_H 0 and 3, or about 2 c.c. of 1% alcoholic dichlorofluorescein, which changes between p_H 4 and 6.6, per 100 c.c. of the liquid is recommended. The titration is carried out in the light from a Wood's lamp, which is placed as near as possible to, and in any case not more than 30–40 cm. from, the surface of the liquid, and is slightly inclined so as to illuminate the whole surface almost vertically. A wide shallow titration vessel of non-fluorescing glass is used, and comparison with a second quantity of the tanning liquid renders it easy to determine exactly when the titrated solution begins to exhibit permanent fluorescence; the reverse change is not quite so easily seen. Difficulties in the method still remain to be overcome.

T. H. POPE.

Hoof meal. STERLING.—See XVI.

PATENT.

[Manufacture of] imitation suede leather [by coating fabric with cellulose acetate etc.]. C. E. SIMPSON (B.P. 306,693, 11.2.28).

XVI.—AGRICULTURE.

Nature of acidity in mineral soils. S. Goy, P. MÜLLER, and O. ROOS (Z. Pflanz. Düng., 1929, 13A, 66–91).—The acid condition of mineral soils is characterised by the following factors. (1) Total acidity determined by electrometric titration to p_H 7.7. This comprises the exchange acidity, determined by titrating a soil suspension in potassium chloride to p_H 5.5, and the hydrolytic acidity. (2) The existing p_H of the soil in aqueous suspension. (3) The p_H of the soil in potassium chloride solution. (4) Buffer value for the total acidity expressed as mg. of sodium hydroxide necessary to neutralise 1 mg. of hydrogen ion. (5) Buffer value for the exchange acidity (to p_H 5.0). (6) Buffer value for the hydrolytic or residual acidity (p_H 5.0–7.7).

A. G. POLLARD.

Acid content and lime requirement of cultivated soils. J. HASENBÄUMER, R. BALKS, and M. BACH (Z. Pflanz. Düng., 1929, 13A, 93–118).—Comparison

is made of well-known methods for ascertaining the lime requirement of soils. The lime contents of a number of soils as determined by Hissink, Gehring, and by Bobko were in close relationship. The extent of the exchange and hydrolytic acidities of soils were generally in agreement with their lime contents. The degree of saturation by the Gehring method was a satisfactory measure of the lime condition of soils and was in agreement with observed data of the replaceable calcium and the exchange and hydrolytic acidities. In soils of similar composition and humus content there is a close relationship between the reaction and hydrolytic and exchange acidities. The interdependence of soil reaction, exchange acidity, lime content, and plant growth is discussed. A. G. POLLARD.

Degree of saturation of soils with lime and potash. A. GEHRING (Z. Pflanz. Düng., 1929, 13A, 1–17).—The amounts of replaceable calcium in soils as determined by the methods of Hissink and of Gedroiz are slightly higher when smaller soil samples are used. The Gehring-Wehrmann method (B., 1927, 728) for determining the saturation capacity of soils for lime also indicates higher values when 12.5 g. of soil are used in place of the usual 25 g. The treatment with lime water and carbon dioxide and subsequent leaching out with sodium chloride brings into solution much calcium chloride which reduces the solubility of calcium carbonate in the leaching solution. This results in a false value for the calcium content in the second litre of leaching solution (Hissink). Where much chalk is present in soils, more than the customary 1 litre of sodium chloride solution is necessary completely to remove the adsorbed calcium. A modified process for the determination of the saturation capacity of soils for lime is described. The significance of Neubauer values and determinations of the degree of saturation of soils with potash in relation to the results of fertiliser trials is discussed. A. G. POLLARD.

Significance of the degree of saturation of soils with potash in evaluating their potash requirement. A. GEHRING and O. WEHRMANN (Z. Pflanz. Düng., 1929, 13A, 18–28).—Relationships of general application between fertiliser requirement of soils, crop yields, and the degree of saturation of soil with lime or potash are obscure. The solubility of soil potassium (Neubauer) varies with the degree of saturation with calcium, there being a minimum value at 40% saturation. Plant experiments do not, however, indicate a direct relationship between the degree of saturation of the soil with calcium and the potassium assimilation by the crop. Better agreement is found between the crop yield and the assimilable potassium (Neubauer) expressed as a percentage of the replaceable potassium. The latter is designated "relative Neubauer value."

A. G. POLLARD.

Activity of silicic acid [in soils]. O. LEMMERMANN (Z. Pflanz. Düng., 1929, 13A, 28–39).—A critical survey of literature concerning the relationship between silica and phosphates in soil. A. G. POLLARD.

Iodine in soil and plants. R. KÖHLER (Z. angew. Chem., 1929, 42, 192–197).—Determinations of the iodine content of a variety of soils revealed no definite

relationship with other factors such as p_H value or humus content. Peats appear to be richest in iodine, the proportion being independent of the distance from the sea. The amount of iodine taken up by plants is increased by addition of potassium iodide to the soil.

F. R. ENNOS.

Determination of small quantities of nitrogen in plant materials. J. T. SULLIVAN and L. E. HORAT (J. Assoc. Off. Agric. Chem., 1929, 12, 133—135).—Modifications of the standard Kjeldahl method suitable for plant materials very poor in nitrogen are described.

S. I. LEVY.

Growth and nutrient intake of the cotton plant. S. A. KUDRIN (Z. Pflanz. Düng., 1929, 13A, 91—93).—The chemical composition of the cotton plant at different stages of its growth was examined. Nitrogen and phosphorus accumulate mainly in the flower and seed, which in the later stages of development contain two thirds of the total amounts of these elements in the plant. Calcium is chiefly distributed in the leaves and magnesium fairly uniformly throughout the plant. The greatest intake of nutrients occurs during bud and flower formation, when the proportion of calcium and nitrogen absorbed increases in proportion to magnesium and phosphorus. The nutrient requirement of the cotton plant is high.

A. G. POLLARD.

Effect of commercial fertilisers on the performance of apple trees. J. R. COOPER (Ark. Agric. Exp. Sta. Bull., 1928, No. 227, 1—61).—Phosphorus increased the number of fruit spurs, the setting and yield of fruit, and the growth of orchard cover crops. Nitrogen increased terminal and trunk tree growth, the number of fruit spurs set, and total crop. During the first two years only, sodium nitrate was more effective than ammonium sulphate. Gains in growth or yield were not recorded with potassium. The quality was not affected by any fertilisers.

CHEMICAL ABSTRACTS.

Minimum potassium level required by tomato plants grown in water cultures. E. S. JOHNSTON and D. R. HOAGLAND (Soil Sci., 1929, 27, 89—109).—Water-culture experiments are described in which a constant flow of nutrient solution was maintained throughout the growth period. Optimum growth of tomatoes occurred with 5 p.p.m. of potassium in the nutrient flowing at an average rate of 8 c.c./min. per plant. With smaller concentrations the intake of potassium by the plants decreased, and there was a tendency for larger proportions of magnesium, calcium, and phosphate to be absorbed. Potash deficiency was reflected in the plant by characteristic leaf-spotting. Evidence is obtained that variations in light intensity affect crop yield to a similarly proportional extent whether the nutrient has a high or low concentration of potassium.

A. G. POLLARD.

Field experiments with fertilisers for sugar cane. H. H. DODDS, P. FOWLIE, and D. McRAE (J. S. African Chem. Inst., 1929, 12, 33—36).—The addition of phosphorus, nitrogen, or potassium in suitable forms to a soil consisting of a heavy clay loam which had been under sugar cane for many years resulted in each case in a larger yield of cane and sucrose per acre, although no increase in the concentration of sucrose in the cane

was apparent. Fertilisation with phosphate produced a large rise in the phosphorus content of the sugar juice; nitrates were found to give better results than ammoniacal or amido-compounds as sources of nitrogen.

F. R. ENNOS.

Determination of hoof meal. W. F. STERLING (J. Assoc. Off. Agric. Chem., 1929, 12, 129—132).—A method of determining keratinous material is based on the resistance to attack by pepsin. The dried and ground sample is shaken with carbon tetrachloride; bone sinks to the bottom, the suspension is washed off, filtered, and the insoluble portion is digested with acidulated pepsin at 37—40° for 48 hrs. The residue separated by aid of a centrifuge is dried and weighed; the result is multiplied by 1.54 to give the proportion of hoof (and horn) meal present. The dried residue should be examined microscopically to detect and estimate any vegetable tissue present.

S. I. LEVY.

Cannery waste. HOLMES and FINK.—See XIX.

PATENTS.

Treatment of cotton plants. W. EGGERT, JUN. (U.S.P. 1,698,539, 8.1.29. Appl., 31.12.25. Cf. U.S.P. 1,686,964; B., 1928, 938).—The plants are treated during the blossoming time with a fermented solution of cotton-plant leaves and blossoms containing molasses and extract of sassafras leaves.

A. R. POWELL.

Calcium nitrate compounds (B.P. 276,350).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Technical method of using the mercury arc to obtain data at wave-length 560 μ in the spectrophotometric analysis of sugar products. H. H. PETERS and F. P. PHELPS (Bur. Stand. J. Res., 1929, 2, 335—342).—Quantitative spectrophotometric analysis of absorption and transmission of technical sugar products at wave-length 560 μ is of importance (cf. Bur. Stand. Tech. Paper No. 338), but no suitable source of monochromatic light is available. From data on 40 representative cane sugar products it is shown that the specific absorptive index, $-\log t$, of these products for $\lambda = 560 \mu$ may be calculated to give results within 1% of the observed value by deducting 48% of the difference between the values of $-\log t_{546}$ and $-\log t_{578}$ from $-\log t_{546}$. This permits the use of the mercury vapour lamp with spectral filters as the light source and enables routine measurements to be made without an elaborate spectrophotometer.

C. J. SMITHELLS.

Titration of sugars. N. SCHOORL (Chem. Weekblad, 1929, 26, 130—134).—The influence of the time of boiling and of other factors on the reduction of cupric salts by various sugars has been examined, and tables are given from which the amounts of sugars present under standard conditions, using Luff's method (B., 1899, 78), with 10 min. boiling, may be read off directly from the volumes of thiosulphate used.

S. I. LEVY.

Verification of the 100° point of the Ventzke sugar scale. Introduction. C. A. BROWNE (J. Assoc. Off. Agric. Chem., 1929, 12, 106—107). I. R. T. BALCH and H. G. HILL (*Ibid.*, 108—115). II. F. W. ZERBAN, C. A. GAMBLE, and G. H. HARDIN (*Ibid.*, 115—120).—

An account of the discrepant determinations which led up to the re-examination now described.

I. The polarisation in a tube of 200 mm. length at 20° of solutions containing 26 g. of pure sugar in 100 c.c. was determined on 20 samples, for each of which 100 readings were made, as $99.907^\circ \pm 0.003^\circ$. As a normal solution, one containing 26.026 g. per 100 c.c. is suggested; from the same number of determinations the average value 100.003° was found. This suggested normal solution contains 23.724% of sucrose and has $d_{20}^{20} 1.09965$.

II. A further set of 20 determinations gave an average corrected value for a solution of 26 g. of pure sugar per 100 c.c. of $99.912^\circ \pm 0.004^\circ$. For the whole series described in both parts of the set of papers, the average value is 99.904° .
S. I. LEVY.

Manufacture of starch and glucose. J. A. McLACHLAN (J.S. African Chem. Inst., 1929, 12, 3—15).—A discussion of some of the problems involved.

F. R. ENNOS.

Wolff's method for the determination of starch by means of the interferometer. B. ELEMA (Z. angew. Chem., 1929, 42, 199—201).—The relatively low values for the starch content of plant materials obtained by this method (cf. B., 1924, 567) are possibly due to the high blank, since it is shown that on grinding the material with sand and water as much as 8—16% of its starch passes into solution, whilst the disintegrated starch grains are attacked by diastase at ordinary temperatures. Where possible the grinding with sand in the blank experiment should be avoided.
F. R. ENNOS.

Field experiments with sugar cane. DODDS and others.—See XVI. **Glycerol by fermentation.** TOMODA.—See XVIII. **Starch value of frozen potatoes.** LAMPE.—See XIX.

PATENT.

Oxidation of aldoses. A. STOLL and W. KUSSMAUL, Assrs. to CHEM. WORKS, FORMERLY SANDOZ (U.S.P. 1,703,755, 26.2.29. Appl., 18.6.28. Ger., 2.7.27).—See B.P. 293,322; B., 1929, 33.

XVIII.—FERMENTATION INDUSTRIES.

Lactic acid in the examination of wines, and Möslinger's method for its determination. J. SCHINDLER and V. HULAČ (Chem. Listy, 1929, 23, 73—77).—Tartaric, malic, and other crystalline acids present in wine may during maturation undergo conversion into lactic acid, whereby the acidity of the wine is diminished; this process is advantageous to northern wines, but not to southern wines, the acidity of which is normally low. The lactic acid content of wines, which may attain 5 g. per litre, should be taken into account in estimating the quality of wine on the basis of the crystalline acid content and of the content of extractive substances; the formation of 1 g. of lactic acid corresponds to the loss of 0.57 g. of extractives. Möslinger's method for the determination of lactic acid as modified by Baragiola (B., 1914, 762) gives results somewhat higher than does Kunz's method (Z. Unters. Nahr. Genussm., 1901, 4, 673); the former method is, however, more convenient for ordinary use. R. TRUSZKOWSKI.

Extractives of brandy. W. PARTRIDGE (Analyst, 1929, 54, 154—155).—The total solids in brandy vary

somewhat, but 1% appears to be too high for modern products. For 26 samples analysed during 3 years the maximum figure was 0.71%.
D. G. HEWER.

Production of glycerol by fermentation. VI. Influence of sugar concentration on the yield. Y. TOMODA (J. Soc. Chem. Ind., Japan, 1928, 31, 601—613; cf. A., 1928, 365, 923).—At a given total sulphite concentration, the yield of glycerol is proportional to the sugar concentration in the initial mash, whilst at a given sugar concentration it is proportional to the square root of the total sulphite concentration. Thus the amount of glycerol produced in a given volume of the mash can be expressed by $w = (a + Kx)\sqrt{y}$, where w is the amount of glycerol produced (g./100 c.c.), x is the concentration of sugar in the mash, y the concentration of total sulphite in the mash (as NaHSO_3 , g./100 c.c.), and a and K are constants ($a = 0.42$, $K = 0.11$). It is found that the presence of the acetaldehyde-bisulphite compound in the fermenting mash favours the formation of glycerol, and when the fermenting medium is made alkaline by the addition of bisulphite, the formation of glycerol becomes very marked. In the presence of an appreciable amount of the combined sulphite (even in the absence of free sulphite) in the fermenting medium, no less than 17% of the fermented sugar was converted into glycerol. It is suggested that the acetaldehyde-bisulphite compound becomes slightly dissociated; the acetaldehyde set free is converted into butylene-glycol etc., and the remaining sulphite tends to combine with more aldehyde, with the production of more glycerol from the sugar present.
S. OKA.

Palmitic acid as a constituent of fusel oil. K. KINO (J. Soc. Chem. Ind., Japan, 1928, 31, 749).—When fusel oil obtained from kaolian was treated with caustic potash, nearly pure crystals of potassium palmitate were obtained.
Y. TOMODA.

Animal feeding and brewery by-products. C. HEIGHAM (J. Inst. Brew., 1929, 35, 104—108).—Wet grains containing 5.5% of digestive protein and 18.4 units of starch equivalent are cheaper than several other succulent feeding stuffs, but have the disadvantages of being bulky and of souring quickly. The dried grains escape these limitations, but have a high percentage of fibre, rendering their digestion difficult and making their price, judged in units of starch equivalent, rather high. Malt culms as a source of protein (19.9%) are useful, but their high fibre content and expense compare badly with coconut cake. A content of 41.6% of protein with 67.2 units of starch equivalent renders dried yeast valuable, but the supply is small and uncertain.

C. RANKEN.

Starch value of frozen potatoes. LAMPE. **Pickling of frozen potatoes.** GLAUBITZ.—See XIX.

PATENTS.

Production of absolute alcohol. E. I. CLAPP, Assr. to U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,702,495, 19.2.29. Appl. 18.5.25).—See B.P. 283,701; B., 1928, 241.

Milk-serum for baking of bread etc. (B.P. 306,374). **Pectin from apples** (U.S.P. 1,698,985).—See XIX.

XIX.—FOODS.

Detection of egg-yolk in margarine. J. ANGERHAUSEN and G. SCHULZE (Pharm. Zentr., 1929, 70, 133—137).—If the yolk used has been dried at 100°, it is rendered insoluble in 2% salt solution; it then becomes necessary to test for the yellow colouring matter in the insoluble material, by extracting with alcohol-ether. The yellow solution is evaporated, the residue taken up in 50% alcohol, and warmed with 1% sulphuric acid; the colour remains unaltered if egg-yolk has been added, but becomes red if artificial colouring materials have been used. S. I. LEVY.

Pickling of frozen potatoes. M. GLAUBITZ (Z. Spiritusind., 1929, 52, 84).—The potatoes, which make good fodder on account of their high sugar content, should preferably be steamed before "souring." The steamed potatoes are tightly packed with a minimum of air space into water-tight pits constructed of concrete or clay. Boards are placed over a layer of straw or chaff on the top of the packed potatoes, and the whole is covered with clay or earth. The formation of butyric acid bacteria or fungi is prevented by the natural development of lactic acid bacteria. It is customary, however, to ensure this development by sprinkling the packed and still warm potatoes with a pure culture of lactic acid bacteria which reproduces at 50—55°. If the preliminary steaming is omitted, the potatoes are washed, cut into very small pieces, and packed into the pit in a solid mass without air spaces. Since the potatoes are cold the lactic acid culture used above is unsuitable, and a mixture of two varieties which develops at 30° takes its place. C. RANKEN.

Determination of the starch value of frozen potatoes. B. LAMPE (Z. Spiritusind., 1929, 52, 92—93).—The determination of starch in frozen potatoes must be carried out immediately after the thawing of the potatoes, since the starch content during storage after thawing shows a gradual increase which may amount to as much as 5%. In addition, the starch value depends on the degree of freezing which the potatoes have experienced. If they have been frozen so hard that they cannot be cut with a knife, the correct starch content is obtained by deducting 2% from the determined value, whilst a correction of 1% is sufficient in those cases where the potatoes have not reached the "glass-hard" stage. In measuring the quantity of potatoes required to give a fixed starch value in the mash, allowance must also be made for the smaller volume occupied by the thawed potatoes compared with the volume of those in the normal state. C. RANKEN.

Comparison of the Monier-Williams and the A.O.A.C. methods for the determination of sulphurous acid in food products. J. FITELSON (J. Assoc. Off. Agric. Chem., 1929, 12, 120—129).—The method of Monier-Williams (Rep. Public Health and Medical Subjects, 1927, No. 43) is found to be more accurate and reliable than the official method employed in America. The preservation of onions in brine reduces the proportion of volatile sulphur compounds, and does not produce sulphurous acid. S. I. LEVY.

Thermophilic and thermoduric micro-organisms with special reference to species isolated from

milk. V. Description of spore-forming types. P. S. PRICKETT (N.Y. Agric. Exp. Sta., Tech. Bull, 147, Oct., 1928, 58 pp.)

Brewery by-products as feeding stuffs. HEIGHAM.—See XVIII. **Coagulation of cannery waste waters.** HOLMES and FINK.—See XXIII.

PATENTS.

Preservation of butter. E. GFELLER (B.P. 297,457, 30.8.28. Switz., 22.9.27).—The butter is heated and energetically agitated until it solidifies again by cooling, whereby the butter fat is prevented from separating from the watery constituents. [Stat. ref.]

W. J. BOYD.

Producing a milk-serum preparation for the improvement of bread, cakes, and biscuits and as an aid in the baking thereof. N. M. KRONBERG (B.P. 306,374, 29.6.28).—Skimmed milk, which has been heated to 115—120°, and subsequently cooled to and retained at 30—40°, is inoculated with pure lactic acid cultures. After the incorporation of an ammonium salt and saccharine matter with the mass, vegetable fat and a substance rich in diastase, such as *Triticum amylaceum*, are added 12 hrs. later, and the mixture is allowed to reach a maximum acidity of 1% at 25—30°. The product is dried at 36—40° for 3 or 4 days and ground to powder. C. RANKEN.

Preservation of liquid eggs, egg yolk, or egg white. A. DONNER (B.P. 306,013, 10.11.27).—Acetic or lactic acid, or a mixture of these, with or without an essential oil, is added to the liquid egg material.

W. J. BOYD.

Fruit product. [Preparation of pectin from apples.] W. A. BENDER, Assr. to CERTO CORP. (U.S.P. 1,698,985, 15.1.29. Appl., 20.7.27).—The pulp is extracted with water to remove sugars, and the residue is digested with lactic acid to obtain a weak pectin solution, which is clarified and freed from tannin by agitation with powdered casein. The filtered liquor is subjected to diastatic fermentation to remove starch, and the pectin precipitated by addition of alcohol.

A. R. POWELL.

Freezing or cooling fish and other articles of food. H. L. RAASCHOU-NIELSEN (B.P. 306,754, 16.4.28).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Keeping chloroform for anaesthesia. I. G. OBERHARD (Pharm. Ztg., 1929, 74, 260—261).—Addition of small quantities of non-volatile acids is found completely to inhibit the decomposition of chloroform, even when freely exposed, with shaking, to light and air for long periods. As the most suitable preservative, 0.1% of pure, powdered, anhydrous citric acid is recommended.

S. I. LEVY.

Iodoform content of iodoform gauze. E. SCHULEX and G. VASTAGH (Pharm. Zentr., 1929, 70, 117—119).—The iodoform content is generally independent of the medium used for fixation (glycerin, paraffin, glycerin and resin), but is very unevenly distributed throughout the gauze, and is considerably lower than it should be, by reason of losses during the drying operation. Standard gauzes should therefore be impregnated with 25% more than the final requirement of iodoform. The

iodoform content varies little on keeping, even in warm weather.

S. I. LEVY.

Bio-assay of commercial pituitary powders. W. T. McCLOSKEY and J. C. MUNCH (J. Assoc. Off. Agric. Chem., 1929, 12, 135—136).—The standard method of the U.S. Pharmacopœia for testing liquor pituitarii is suggested as a test for powdered preparations.

S. I. LEVY.

Protection of drugs susceptible to light by coloured glass. III. J. EISENBRAND (Pharm. Ztg., 1929, 74, 263; cf. von Halban and Eisenbrand, A., 1927, 1017).—A table is given for the proportion of incident light between wave-lengths 650 and 313 μ . transmitted by glasses of different origin and colour.

S. I. LEVY.

Pine-needle extract. BOHRISCH (Pharm. Ztg., 1929, 74, 294—299).—Whilst adulteration of the extract with sulphite lye, which was formerly commonly practised, is easily detected, the presence of refined sulphite lye, which appears to contain neither free nor combined sulphur dioxide and very little mineral constituent, cannot be detected by the usual tests. The claims made by Peyer and Diepenbrock (Apoth.-Ztg., 1926, No. 94/95) that their capillary test gives quantitative results in the determination of adulteration by refined sulphite lye have been examined; whilst this method is very valuable, accurate quantitative results can hardly be expected, in view of the variable compositions both of pine-needle extracts and refined sulphite lye. The formation of foam is frequently independent of the composition. An exhaustive specification for inclusion in a pharmacopœia is suggested.

S. I. LEVY.

Mechanism of formation of essential oil in the resin of conifers. XII. Formation of the essential oil of *Pinus silvestris*. G. V. PIGULEVSKI and Z. M. ZAIKINA (J. Russ. Phys. Chem. Soc., 1928, 60, 1417—1425).—The amounts and properties of the essential oil and resin present in the needles and young twigs of *Pinus silvestris* during different periods of its annual growth were determined. The growth of the needles reaches a maximum about the middle of August, but the amount of essential oil present does not increase after the beginning of July. The oil from the needles has $\alpha_D -15.44^\circ$ to $+7.52^\circ$; that from the twigs has $\alpha_D -15.3^\circ$ to -45.68° . The amount of resin increases continuously, and is formed from the essential oil. The composition of both the oil and the resin is very complex, consisting of free acids, esters, and ethers.

M. ZVEGINTZOV.

Essential oil from seeds of *Smyrnum perfoliatum*. L. V. I. NILOV (J. Russ. Phys. Chem. Soc., 1928, 60, 1579—1584).—The essential oil on distillation yields a liquid fraction (deep violet) and a crystalline solid. The liquid when distilled *in vacuo* yielded various fractions: (a) b.p. 160—165°/760 mm., had $d_{20}^{25} 0.8767$, $n_D^{25} 1.4810$, and consisted mainly of the unidentified hydrocarbon, $C_{10}H_{16}$; (b) b.p. 135—140°/20 mm., had $d_{20}^{25} 0.9167$, $n_D^{25} 1.5061$, and contained the sesquiterpene $C_{15}H_{24}$; (c) b.p. 140—148°/8 mm., was a complex mixture of substances; (d) b.p. 160—170°/8 mm. was deep blue turning to dark violet on keeping. The crystalline solid on recrystallisation gave a com-

pound, $C_{15}H_{22}O$, m.p. 56° , b.p. 143—144°/7 mm., $d_{20}^{25} 0.9797$, $n_D^{25} 1.5100$, which contained neither a hydroxyl nor a carboxyl group. On reduction, a liquid hydrocarbon, b.p. 135—140°/12 mm., $d_{20}^{25} 0.9339$ —0.9321, $n_D^{25} 1.4870$, was obtained.

M. ZVEGINTZOV.

Composition of the essential oil from *Seseli dychotomum*. V. I. NILOV (J. Russ. Phys. Chem. Soc., 1928, 60, 1575—1577).—When the oil, $d_{20}^{25} 0.8754$, $\alpha_D -5.5^\circ$, $n_D 1.4775$, was distilled at 20 mm., 94% came over below 75° . This distillate on fractionation at atmospheric pressure boiled between 157° and 175° . The presence of α -pinene in the first fraction was indicated by the formation of a nitrosochloride (b.p. 103°) which on treatment with sodium ethoxide yielded a nitrosoterpene (m.p. 131°). This was confirmed by oxidation of the oil with potassium permanganate, when pinonic acid, m.p. 103 — 104° (semicarbazone, m.p. 203 — 204°), was obtained. The higher fractions on oxidation gave nopinic acid, m.p. 126° , indicating the presence of β -pinene.

M. ZVEGINTZOV.

Medicinal glass. SCHWENKE.—See VIII.

PATENTS.

Manufacture of alkali bismuth tartrates, especially intended for the treatment of syphilis. R. W. JAMES. From D. E. SEARLE & Co. (B.P. 304,956, 2.4.28).—Bismuth hydroxide, tartaric acid, water, and sodium or potassium hydroxide are shaken at 14 — 15° for 6—7 days, the product is filtered, and half the volume of 95% alcohol added to the filtrate. A sodium (or potassium) tetrabismuthyl tartrate, $C_4H_2O_6(BiO)_4NaOH$ (or KOH), is precipitated. Aqueous solutions are stable at 70° .

C. HOLLINS.

Manufacture of thiosemicarbazones of arseno-phenol-aldehydes or -ketones. I. G. FARBENIND. A.-G. (B.P. 294,263, 21.7.28. Ger., 21.7.27).—The sodium salts of the thiosemicarbazones of hydroxy-aldehydo- and hydroxyacyl-arsenobenzenes are much more stable in aqueous solution than the salts of the parent hydroxy-aldehydes and -ketones. The sodium salt of 2:2'-dihydroxy-4:4'-dialdehydearsenobenzene-dithiosemicarbazone is prepared by reducing the thiosemicarbazone of 3-hydroxybenzaldehyde-4-arsinic acid, neutralising the separated product with sodium hydroxide, and precipitating with alcohol.

C. HOLLINS.

Manufacture of chemotherapeutical compositions. CHEM. FABR. VORM. SANDOZ (B.P. 283,565, 8.11.27. Switz., 15.1.27).—The trypanocidal action of dyes of the type of trypan-blue, trypan-red, and chloramine-blue (Colour Index, Nos. 447, 438, 406) is enhanced by mixing the dyes with bile acids or their salts (sodium cholate). The products cure also nuttalliosis in domestic animals, whilst the pure dyes have no effect.

C. HOLLINS.

Cultivation of the micro-organisms parasitically vegetating in human and animal bodies and preparation of immunising sera therefrom. W. SCHMIDT, and W. SCHMIDT SERUMWERK A.-G. (B.P. 304,155, 11.7.27).—Parasitic micro-organisms which are promoters of malignant tumours are cultivated in symbiosis with lower plants (*Mucor racemosus*, *M. mucedo*) by using a nutrient medium containing not more

than 6% of sugar, preferably commercial glucose, *e.g.*, peptone-sugar broth, or pea-sugar-agar broth, at 10–18°. At these temperatures some of the other micro-organisms present in the starting-material fail to develop, and others are starved by the consumption of the available nutrient material by the *Mucor*. The starting-material of human origin is often with advantage transferred first, with the *Mucor*, to a lower animal, so as to destroy the unwanted micro-organisms before culture on the broth; the removal from the animal must be timed so that the *Mucor* has not been also destroyed. The symbiotic culture may be freed from its lower-plant host by injection into an animal, and pure cultures of the parasitic organism obtained. These may be grown on the broth at 25–37°, or may be introduced into the peritoneal cavity of an animal in a perforated glass tube closed with collodion, which allows the animal juices to penetrate into the tube but excludes symphocytes and phagocytes; on removal of the tube its contents are found to have increased in virulence. Inoculation of animals with the mixed or pure cultures so obtained leads to the production of anti-bodies, and valuable sera for immunisation of human beings against the parasitic micro-organisms promoting malignant tumours may thus be prepared. C. HOLLINS.

Production of cancer toxin and antitoxin. T. J. GLOVER (B.P. 300,144, 6.5.27).—Culture medium, prepared by mixing equal quantities of boiled, strained, and filtered mixtures of sunflower seed, Iceland moss, and Irish moss in tap-water and adding 1½% of Fairchild's old peptone, is inoculated with fresh cancerous tissue. The mixture is heated, the p_H adjusted to 6.4–6.6, and, after heating in an autoclave for 1½ hrs. at 15 lb./in.², the whole is treated with about half its volume of hormone-gelatin broth and 0.5–1.0% of dextrose, the p_H being again adjusted to 6.4–6.6. The medium is sterilised in tubes at full steam on three successive days to the same final p_H reaction, and is further cultivated, *e.g.*, in fresh sterile horse or rabbit serum at 37° for 7–10 days, to obtain cancer toxin of maximum potency. The toxin is filtered through a fine Berkefeld filter, treated with 0.5% of phenol to kill organisms passing through the filter and kept at a low temperature until it ceases to lose potency. The toxin is then standardised and used to inoculate an animal, *e.g.*, young healthy horse, giving a negative glands test, the blood being drawn off and the cancer anti-toxin serum separated when the antitoxin serum has been found by test to reach the desired potency, *e.g.*, 6–9 months. R. BRIGHTMAN.

Manufacture of hormones having a heart-stimulating action, or of extracts containing such hormones. I. G. FARBENIND. A.-G. (B.P. 279,445 and Addn. B.P. 306,606 and 306,608, 17.10.27. Ger., [A] 20.10.26).—Comminuted heart of cattle or pigs is extracted (A) with water at 15°, or (B) with a water-insoluble organic solvent (ether), the aqueous layer being used, or (C) with a water-miscible organic solvent (acetone) which is removed, after precipitation of ballast substances, and the lipoids extracted from the aqueous residue by means of a water-insoluble solvent (methylene chloride). For (B) and (C) the mash should be made

neutral or slightly alkaline. For (C) dried heart and water may be used. In each case the aqueous solution, after being freed from ballast substances and lipoids, contains alcohol-insoluble, thermo-stable hormones useful in cardiac diseases. C. HOLLINS.

Preparation of monocyclic ketones and their alkyl derivatives having more than nine ring members. L. RUZICKA, ASSR. to M. NAEF & Co. (U.S.P. 1,702,842–1,702,852, 19.2.29. Appl., [A–K] 19.11.26. Switz., [A–J] 15.12.25, [K] 16.6.24).—See B.P. 235,540 and 263,153; B., 1925, 738; 1928, 36.

Manufacture of aromatic acid esters of α' -dimethyl- γ -hydroxypiperidine- β -carboxylic acid esters. F. STOLZ and W. KROHS, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,703,121, 26.2.29. Appl., 25.4.27. Ger., 17.5.26).—See B.P. 271,467; B., 1927, 669.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Sensitivity of photographic emulsions. III. S. O. RAWLING (Phot. J., 1929, 69, 83–93; cf. B., 1927, 125).—The rapid change in sensitivity of emulsions accompanying a change in p_H during the digestion period after washing has been investigated over a p_H range 8.5–5.0, and it is shown that this rapid change is a characteristic of the gelatin and is independent of whether the silver halide grains consist of pure silver bromide or of silver bromide with 4% of silver iodide, or whether or not ammonia ripening was used before washing. In addition to this rapid and almost fully reversible change in sensitivity there appears to be a slow change which depends on the p_H of the emulsion during digestion. In general, sensitivity increases more rapidly with digestion at high p_H than at low, and cases were found in which the sensitivity actually decreased with digestion at low p_H . Further examples of emulsions are recorded in which, at the p_H values stated above, fog, shape of characteristic curve, and rate of development are independent of p_H during digestion. Sensitisation of the emulsion with thiosinamine before washing had no effect on the magnitude of the change in sensitivity with change in p_H . J. W. GLASSETT.

Development of images with fine grain. A. and L. LUMIERE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1928, 15, 258–263).—Development after fixation undoubtedly gives the finest grain, but a very long time of development and a much increased exposure are necessary. The developer which, though very slow (time of development 1 hr.), gave the best results with ordinary developments consisted of water 1000 c.c., *p*-phenylenediamine 10 g., sodium sulphite (anhyd.) 60 g., and borax 50 g. J. W. GLASSETT.

Evolution of the latent image. G. JAUSERAN (Compt. rend., 1929, 188, 783–785).—Density determinations on plates developed at various intervals after exposure indicate that the evolution of the latent image is very rapid at first, but depends subsequently on the type of plate and on the density under consideration, the differences being least marked when the density is low or approaches that of saturation. The rate of evolution then falls off and a maximum constant density is obtained after a few hours. J. GRANT.

XXII.—EXPLOSIVES; MATCHES.**PATENT.**

Separation and, especially, after-separation of nitroglycerin or nitroglycol and similar explosives from the acids used in their preparation. A. SCHMID and J. MEISSNER (B.P. 299,384, 28.1.28. Ger., 25.10.27. Addn. to B.P. 284,701; B., 1929, 304).—The separator is provided in the front and at the back with a distributor which has perforated plates the holes in which are staggered with respect to those in the succeeding plate and are also smaller and more numerous from plate to plate. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

The problem of automobile exhaust gas in streets and repair shops of large cities. J. J. BLOOMFIELD and H. S. ISBELL (Rep. U.S. Public Health Service, 1928, Reprint No. 1217, 16 pp.).—250 samples of air were obtained from 14 of the largest American cities. The samples were analysed by the iodine pentoxide method, using a liquid-air cooling tube to eliminate gasoline vapour which otherwise would vitiate the results. The average of 141 tests made in city streets at peak hours of traffic showed a contamination of 0.8 pt. of carbon monoxide per 10,000 pts. of air. Of the street samples, 24% had more than 1 pt., and in one location a covered passageway yielded 2 pts. per 10,000 of air. Samples taken inside motor-buses yielded lower concentrations. The figures for street air do not reveal the existence of a health hazard from this source. Of the 102 tests made in 27 garages of considerable size the average carbon monoxide content was 2.1 pts. per 10,000 pts. of air; 59% contained over 1 pt. and 18% over 4 pts. These results show a dangerous condition that should be avoided by not allowing automobile engines to run longer than 30 sec. unless special precautions are taken to remove the exhaust gases.

H. S. GARLICK.

Sodium aluminate as coagulant in chemical treatment of cannery waste waters. J. A. HOLMES and G. J. FINK (Ind. Eng. Chem., 1929, 21, 150—151).—The liquid waste from a tomato cannery, after removal of skins etc., is passed through a 30 × 40 mesh screen (0.014 in. opening) and a solution of lime and sodium aluminate is added at the average rate of 2.5 lb. of lime and 0.4 lb. of sodium aluminate per 1000 gals., the p_H value being maintained above 10.0. After thorough mixing and settlement the effluent obtained is clear and sparkling, and the oxygen-consuming power has been reduced from 2100 p.p.m. to 396 p.p.m. The resultant sludge is pumped daily into adjacent fields, and there dries rapidly without nuisance, and is used as a fertiliser with good results. C. JEPSON.

Factors contributing to quality of public water supplies. H. E. JORDAN (Ind. Eng. Chem., 1929, 21, 152—156).—The first essential in a public water supply is that it must be safe; nothing less is permissible. The supply must be palatable and of good appearance, as these are the criteria by which an ordinary person judges its quality. The removal of hardness and corrosive power is largely a question of economics. C. JEPSON.

Chlorination of water. II. L. W. HAASE (Gas- u. Wasserfach, 1929, 72, 217—222. Cf. B., 1928, 466).—

Since chlorination of crude water results in increased acidity and a reduction in the organic matter in solution as given by the oxygen absorption, the removal of iron is facilitated, as humates are converted into easily precipitated chlorides or carbonates. Even the increased acidity may be an advantage if chlorination is to be followed by coagulation with aluminium sulphate. It may, however, result in pipe corrosion unless the water is further treated with alkali. The chlorination of filtered water usually involves the use of a slight excess followed by dechlorination by treatment with carbon, sodium thiosulphate, or hydrogen sulphide. In cases of carbon filtration the carbon plays a part similar to the organic matter in crude water, and the chlorine is quantitatively transformed into hydrochloric acid. A series of samples of coloured water were chlorinated, and it was found that where the colour was due to humates it was almost entirely removed, and the oxygen absorption was reduced by 20—50%; the water remained alkaline. When the colour is not due to humates, as in effluents, chlorination produces little effect. A further series of tests determined changes in p_H value, hardness, and chlorine content with chlorination, and subsequent carbon treatment, each step marking an increase in acidity. In some cases the increase in chlorine content found far exceeded the chlorine added. C. IRWIN.

Chlorination of coagulated water. J. S. WHITENER (J. Amer. Water Works' Assoc., 1929, 21, 258—261).—Comparison of operating data of the filtration plant at Raleigh, N.C., for periods with and without chlorination of the coagulated water, after allowing for differences due to weather conditions, fluctuation in demand, etc., indicated that a saving of \$144 per million gals. of plant output per annum was effected by the use of chlorine. This was largely due to an improvement in the condition of the filters, which allowed longer runs to be made and, consequently, an increased quantity to be dealt with per run and smaller losses due to wash-water. The *B. coli* index of the filtered water was lower when chlorination was practised, although that for the raw water was higher. At a plant at Southern Pines, N.C., which handles a coloured water, an addition of chlorine along with the alum and lime in the mixing chamber was found greatly to assist in the removal of colour by increasing the size of the floc. The length of the filter runs was also increased and the condition of the filters greatly improved. The addition of chlorine to the coagulated water at this plant produced no beneficial effect. C. JEPSON.

Chlorination of deep well supply for taste and odour removal. M. F. TRICE (J. Amer. Water Works' Assoc., 1929, 21, 255—257).—A slight taste and odour in the water supply of Siler City after two years' operation was intensified a year later to an objectionable extent, although the sanitary quality was said to be excellent. Flushing of the mains failed to remove the trouble, which was eventually traced to the presence in the mains of iron bacteria. The whole of the water-main system was sterilised with chlorine, and, once the chlorine had disappeared, the unpleasant taste and odour of the water improved and all trace of it was gone in a few days. C. JEPSON.

Superchlorination and subsequent dechlorination over carbon of water for municipal supply. E. WATZL (Ind. Eng. Chem., 1929, 21, 156—158).—In order to avoid contamination of potable water owing to fluctuation in the bacterial and organic contents of the raw water it is advisable to add enough chlorine to leave 0.2—0.5 p.p.m. of residual chlorine present after a detention period varying from 10 to 30 min., according to local conditions. Phenolic substances etc. which cause offensive tastes and odours when small amounts of chlorine are present are rendered comparatively harmless by increasing the chlorine dose. On the other hand, high chlorination and the hypochlorous acid thus produced have the effect of breaking down the benzene nucleus of many isocyclic compounds containing one or more hydroxyl groups. These compounds and the residual chlorine may be oxidised or adsorbed by passing through a filter of granular carbon, thereby rendering the water supply tasteless and odourless. C. JEPSON.

Phosphate in boiler-water conditioning. R. E. HALL, H. A. JACKSON, E. W. BUTZLER, J. A. ROBB, E. A. HERTZELL, and G. W. SMITH (J. Amer. Water Works' Assoc., 1929, 21, 79—95).—When aqueous solutions, containing substances the solubilities of which increase with rise in temperature, are concentrated beyond their saturation value these substances deposit as sludge on the heating surfaces and as adherent scale on the cooling surfaces. If the solubility decreases with rise of temperature the reverse is the case. Hence boiler-water conditioning has for one of its objectives so to arrange the chemical equilibria that the water will always have a positive solubility temperature coefficient at the evaporating surface. From the point of view of economy the use of soda ash for this purpose has been considered; for boiler pressures up to 150 lb. its use is advisable, but beyond this its rate of decomposition at the higher temperatures involved is such that excessive amounts must be used to conform with the Amer. Soc. Mech. Eng. Boiler code, and the high hydroxide alkalinity thus produced favours accumulations of undesirable silicates and is inimical to good boiling. For use at high pressures sodium phosphate is recommended, as its stability is satisfactory and the tricalcium salt is less soluble than calcium carbonate. Success therewith depends on its maintenance in the boiler water as free dissolved substance, together with a slight hydroxide alkalinity. The smooth boiling of water depends largely on the nature of the dissolved substances present. In pure water the separation between gaseous and liquid phases is clear and distinct, but increasing concentration of dissolved solids will reduce the size of the bubbles and delay their bursting at the surface, thus causing "foaming," especially when a sudden demand is placed on the boiler. Acid solutions diminish this effect, but alkaline solutions increase it, especially if soap is present even in very small amounts. Suspended matter in a state of fine division is more detrimental than that in a crystalline state, though both increase the tendency towards foaming; tannates and higher alcohols reduce this tendency. C. JEPSON.

Rapid volumetric determination of the sulphate content of drinking water by visual conductometric titration. H. FEHN, G. JANDER, and O. PFUNDT (Z.

angew. Chem., 1929, 42, 158—159).—The water is boiled to convert bicarbonates into carbonates, which are precipitated and collected. The filtrate is treated with an equal volume of alcohol and titrated directly with standard barium chloride or acetate solution using the visual conductometric method of Jander and Pfundt (A., 1926, 700).

A. R. POWELL.

Differentiation of the coli and aerogenes groups of bacteria. A. J. SALLE (J. Amer. Water Works' Assoc., 1929, 21, 71—78).—The differentiation of these groups of organisms and others which ferment lactose with the production of acid or gas or both is of importance in routine water analysis. A solid streaking medium, containing erythrosin, methylene-blue, and bromocresol-purple with a lactose-agar base, which contains enough lactose to supply the carbohydrate needs of *B. coli* but insufficient for *B. aerogenes*, has given good results. Commercial erythrosin is said to be very variable in chemical and physical properties; a special product has therefore been developed for the purpose. The use of dyes, with or without bile in primary enrichment broth, for the purpose of inhibiting the growth of organisms other than those of the colon group and which may give false presumptive tubes gives promising results. Gentian-violet and brilliant-green are generally recommended, but the former—a mixture of dyes—is variable in composition. One of its constituents, crystal-violet, is very actively bactericidal but less toxic to *B. coli* than brilliant-green, and is superior to the latter because of the greater margin of safety which it offers between Gram-positive and Gram-negative organisms. Attempts are also being made to obtain a medium suitable for the enumeration of *B. coli* by direct plating, e.g., the cyanide-citrate medium of Noble (cf. B., 1928, 318).

C. JEPSON.

PATENTS.

Treatment of sewage and industrial [straw-board] waste. J. T. TRAVERS, Assr. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,699,257, 15.1.29. Appl., 27.5.27).—The waste liquor is treated with calcium sulphate and calcium hydroxide, solid material is allowed to settle and is removed, and the residual liquor is treated with a mixture containing monocalcium phosphate.

L. A. COLES.

De-aeration of feed water for boilers. G. & J. WEIR, LTD., and J. SIM (B.P. 306,257, 21.1.28).—The feed water, after being preheated in the condenser of a steam-jet injector, is sprayed into a cylindrical de-aerating chamber maintained under reduced pressure and falls through the chamber into an annular well, from which it passes through ports below water level into a second annular well where it is boiled vigorously by a direct-contact steam heater. The steam generated passes through ports into the main chamber, and on encountering the water spray is condensed and finally discharged over a weir by a pump. Regulation of the water level is effected by means of a float-operated valve.

W. G. CAREY.

Water-softening apparatus. UNITED WATER SOFTENERS, LTD., and H. S. LAWRENCE (B.P. 302,744, 29.9.27).

Spraying of liquids (B.P. 302,817).—See I. Base-exchanging substances (B.P. 279,028).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 10, 1929.

I.—GENERAL; PLANT; MACHINERY.

Waste-heat dryer calculations and charts.

A. E. R. WESTMAN and R. G. MILLS (J. Amer. Ceram. Soc., 1929, 12, 162—174).—Charts are derived which show the effect of atmospheric humidity conditions and air-discharge temperature on the operation of a typical waste-heat dryer. The output of a dryer could be increased by pre-drying the air with silica gel or increasing the outlet air temperature. This would be economically possible if the latent heat of vaporisation of the water were recovered by condensation.

J. A. SUGDEN.

Absorption of solvent vapours. A. ALEŠ (Chem. Listy, 1929, 23, 1—5).—A review of methods used in industry for the recovery of volatile solvents.

R. TRUSZKOWSKI.

Abolition of the Beaumé hydrometer. T. WALLIS (Chem. Fabr., 1929, 135—136).—The proposed abolition of the Beaumé hydrometer involves its replacement by a more satisfactory instrument. It is suggested that in future all hydrometers should record the value of d directly, and should be graduated by immersion in aqueous sulphuric acid from d 1.000 to d 1.841, above this value in zinc chloride solution, and below it in alcohol and petroleum distillate. The standard temperature should be 20°. For other liquids than those used as standards a correction is required on account of the difference in surface tension if an accurate value of d is required.

C. IRWIN.

Use of interferometry in science and technology.

E. BERL and L. RAVIS (Fortschr. Chem. Phys., 1928, 19, No. 7, 1—52; Chem. Zentr., 1928, ii, 1591—1592).

Crystallisation at rest and in motion. K. KIEPER (Chem. App., 1928, 15, 185—186; Chem. Zentr., 1928, ii, 1599).

Determination of drop points. NAUMANN.—See II.

PATENTS.

Concentrating or distilling apparatus. A. MONTUPET (F.P. 629,395, 27.4.26).—The liquids are allowed to circulate in the tubes of a multiple-effect apparatus in which each heating tube is axially disposed within another tube, so that the circulation of the respective fluid is from below to above in the annular space so formed and from above to below in the inner tubes.

W. J. BOYD.

Heat treatment apparatus. J. SCHONBRUNN and G. KELLER, ASSRS. to A.-G. BROWN, BOVERI & Co. (U.S.P. 1,697,144, 1.1.29. Appl., 8.9.27. Ger., 10.9.26).—A furnace chamber is provided with interior means of heating, and with a lid or closure which is provided with

an extension or bell of bent conducting material which maintains the chamber closed to atmosphere when the lid is removed.

B. M. VENABLES.

Treating adsorbent materials. R. E. MANLEY, ASSR. to TEXAS Co. (U.S.P. 1,702,738, 19.2.29. Appl., 26.1.24).—For the removal of adsorbed material from the adsorbent the latter is introduced into a chamber with a vertical helical baffle tapering towards the lower end, and steam is introduced at an intermediate point and air at a lower point tangentially to the baffle, the evolved vapours or liquid being removed from the opposite side at the lower end.

A. R. POWELL.

Manufacture of disperse systems. A. G. BLOXAM. From J. R. GEIGY A.-G. (B.P. 307,000, 1.12.27).—A mixture of sulphite-cellulose lye and colloidal silica is a more effective emulsifying agent than either alone, and may be used in neutral or acid solutions. Fish oil emulsified with this mixture is not precipitated by contact with acid leather.

C. HOLLINS.

Filtering apparatus. A. KÖNIG and G. MÜLLER, ASSRS. to STAATLICHE PORZELLAN MANUF. (U.S.P. 1,704,330, 5.3.29. Appl., 11.1.27. Ger., 19.1.26).—See G.P. 433,376; B., 1927, 189.

Precipitation of solid substances in coarse granular condition from solutions. F. JEREMIASSEN, ASSR. to A./S. KRYSTAL (U.S.P. 1,704,611, 5.3.29. Appl., 10.9.25. Norw., 18.9.24).—See B.P. 240,164; B., 1925, 977.

Catalytic apparatus. SELDEN Co., Assees. of A. O. JAEGER (B.P. 279,819 and 305,636, 11.10.27. U.S., 27.10.26).—See U.S.P. 1,685,672; B., 1929, 229.

Gas and liquid contact apparatus. Sprayer for effecting intimate contact between liquids and gases. W. F. SLATER, ASSR. to KIRKHAM, HULETT, & CHANDLER, LTD. (U.S.P. 1,704,518—9, 5.3.29. Appl., [A, B] 30.4.26. U.K., [A] 25.5.25, [B] 2.6.25).—See B.P. 256,351 and 256,358; B., 1926, 857.

Manufacture of disperse systems. C. A. AGTHE, ASSR. to J. R. GEIGY A.-G. (U.S.P. 1,696,374, 25.12.28. Appl., 22.11.27).—See B.P. 307,000; preceding.

Centrifugal machines. T. BROADBENT & SONS, LTD., and B. L. BROADBENT (B.P. 308,033, 6.2.28).

Furnace wall construction. F. B. DEHN. From M. H. DETRICK Co. (B.P. 307,567, 28.12.27).

Regulating combustion in [boiler] furnaces. SMOOT ENGINEERING CORP., Assees. of C. H. SMOOT (B.P. 282,438 and 300,512, 16.12.27. U.S., 16.12.26).

Apparatus for heating liquids. L. F. THOMPSON (B.P. 305,275, 7.7.28).

[Self-cleansing] apparatus for straining liquids. F. BAILEY and F. H. JACKSON (B.P. 307,984, 21.12.27. Addn. to B.P. 246,551).

Apparatus for pneumatic separation or grading of solid materials [by air blast]. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 307,810, 8.12.27).

Absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE GES.M.B.H. (B.P. 279,898, 29.10.27. Ger., 30.10.26).

Refrigerator evaporators. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. L. HEISLER (B.P. 289,062, 20.4.28. U.S., 22.4.27).

Absorption refrigerating plants. C. PÖHLMANN (B.P. 292,568 and 306,873, 23.8.27).

Refrigerating apparatus. H. R. VAN DEVENTER (B.P. 279,088, 12.10.27. U.S., 12.10.26).

Refrigerating apparatus. GEN. ELECTRIC Co., LTD., and N. E. BARBER (B.P. 307,534, 9.12.27).

Absorption refrigerating systems. ELECTROLUX, LTD., Assees. of [A] C. G. MUTERS, [B] A. LENNING (B.P. 282,065 and 283,473, [A] 7.12.27, [B] 9.1.28. U.S., [A] 8.12.26, [B] 8.1.27).

Absorption refrigerating machines. B. E. D. KILBURN. From SULZER FRÈRES SOC. ANON. (B.P. 282,056, 6.12.27).

Compressed gas cylinders and the like. C. H. BINGHAM, JUN. (B.P. 307,147, 29.12.27).

Apparatus for continuously producing and drying briquettes. N. FLODIN and G. CORNELIUS (B.P. 306,977, 25.10.27).

II.—FUEL; GAS; TAR; MINERAL OILS.

Coals from the Jakutski Mountain region. N. M. KARAVAIEV and I. B. RAPOPORT (Izvestia Teplotch. Inst. [Moscow], 1928, 10, 32—43).—Results of analyses and various other particulars are given concerning these coals. T. H. POPE.

Low-temperature carbonisation of fuels by the Hérens process. C. ROSZAK (Bull. Soc. Ing. Civ. France, 1928, 81, 1064—1104).—The coal, dried and preferably preheated to about 200°, is carried through the distillation chamber by means of a continuous chain grate, and is carbonised principally by the sensible heat of a current of hot gas which is passed down through the coal and the grate, but also by direct radiation from above, the roof of the chamber being externally heated by combustion of part of the gas produced. The process may be used in conjunction with steam-raising plant, the grate then being extended to carry the coke directly into the combustion chamber, wherein also the excess gas may be burnt. To produce a compact coke a heavy endless chain mounted within the chamber rests on the surface of the coal, which is thus compressed as it reaches the softening point.

A. B. MANNING.

Chemical utilisation of coal. C. BERTHELOT (Bull. Soc. Ing. Civ. France, 1928, 81, 1105—1181).—

The methods available for the transformation of coal into motor fuels and fertilisers are outlined and the economic factors involved are discussed in detail.

A. B. MANNING.

Relation between caking and moisture-absorbing power of some Japanese coals. S. IKI (Ind. Eng. Chem., 1929, 21, 239—241).—Dried samples of Japanese bituminous coals of different caking power together with some anthracite and brown coals were kept in an atmosphere of constant humidity and the moisture absorption of each was measured by its increase in weight. The coals were also separated into their α -, β - and γ -portions, the ulmin of the brown coal being extracted. The chief caking constituent of coal, γ , had the lowest moisture-absorbing power, whilst the constituent of greatest absorbing power was ulmin; this may account for the decrease in moisture absorption with increase in caking properties, and may explain the strong affinity for water shown by weathered coals.

C. B. MARSON.

Oxidation of sub-Moscow coal by atmospheric oxygen at different temperatures. N. M. KARAVAIEV and A. K. IVANOV (Izvestia Teplotch. Inst. [Moscow], 1928, 10, 44—50).—Oxidation proceeds differently with the Schtschekinski and Bobrikovski coals of the sub-Moscow basin owing to differences in composition and possibly in the nature of the organic components of the coal. Up to 140° for the former and 80° for the latter the oxidation consists mainly of combination of oxygen; the proportion of carbon monoxide formed increases as the temperature is raised further, the percentage of hydrogen in the coal and, to a considerably less extent, that of hydrogen diminishing and that of oxygen increasing. As a result of the oxidation, the coal loses up to 14% of its heating value. T. H. POPE.

Orsat absorption tube. A. H. MOODY and G. E. STEVENS (Chemist-Analyst, 1928, 17, No. 4, 15).—A new type of absorption tube for the determination of carbon dioxide, oxygen, and carbon monoxide in flue gases is described. CHEMICAL ABSTRACTS.

Composition of tar from low-temperature carbonisation of Utah coal. II. Heavy portions. R. L. BROWN and R. N. POLLOCK (Ind. Eng. Chem., 1929, 21, 234—238; cf. B., 1927, 179; 1928, 393).—The heavy portions of the tar obtained from the carbonisation of Mesa Verde coal at about 700° (by means of superheated steam) which had been collected in air-cooled condensers were analysed. The fractions lighter and heavier than the aqueous distillate consisted of insoluble matter 0.85 and 9.5%, carboxylic acids 2.25 and 2.5%, alkali-soluble (phenolic) 15.0 and 40.0%, tar bases 1.55 and 3.0%, and neutral portions 80 and 45%, respectively. These neutral portions were composed of resin, 18.5 and 20%, wax, 24.1 and 10%, and oil, 57.3 and 69%, respectively. The chief feature observed was the high paraffin wax content of the tar; in the consideration of the economic value of low-temperature carbonisation by-products this is of importance. C. B. MARSON.

Detection of benzene with dracorubin. BERGER (Zentr. Gewerbehyg., 1928, 15, 227—228; Chem. Zentr., 1928, ii, 1594).—The dye "dracorubin," which

is soluble in benzene but not in benzenes, is used in the form of test-paper.

A. A. ELDRIDGE.

Apparatus and methods for separation, identification, and determination of the constituents of petroleum. E. WASHBURN, J. H. BRUNN, and (Miss) M. M. HICKS (Bur. Stand. J. Res., 1929, 2, 467—488).—A rectifying still with a 20-plate column and with means for independently controlling and measuring the temperatures of the plates is described. Distillation was carried out in a stream of inert gas, with or without boiling. The fractions from the still were further separated in all-glass rectifying stills working *in vacuo*. Finally, molecular stills were used in which distillation can be carried out at any temperature at which the vapour pressure at the distilling surface is not lower than the degree of vacuum attainable. Azeotropic mixtures were broken up by fractional crystallisation or melting. A special apparatus for combustion analysis is described in which the oxygen is purified with unusual care and in which no rubber connexions are employed. It is claimed that the accuracy is such as to permit of determining the formula of any hydrocarbon up to C_{100} (i.e., carbon and hydrogen each to $\pm 0.05\%$). The change in iodine value of a petroleum oil resulting from heating the oil at different temperatures up to 370° is much less in an inert gas than in air.

W. E. DOWNEY.

Determination of water in oil fuels. N. I. TSCHERNOSHUKOV (Izvestia Teplotch. Inst. [Moscow], 1928, 10, 11—12).—The methods examined were: (1) Dean and Stark's, (2) Lysenko's, (3) the centrifugal, and (4) Denemark's. The most accurate method is (1), with a maximum error of 0.2%, and the most inaccurate (2), which gave an error of 98% with a sample of mazut. Method (3) mostly gives excellent results, the greatest inaccuracy being 1% for a mazut. Method (4) is simple, and with a mazut free from paraffin and with naphthas is accurate to 1%; with paraffinous mazuts negative results were obtained in some cases.

T. H. POPE.

Cracking. N. A. BUTKOV (Izvestia Teplotch. Inst. [Moscow], 1928, 10, 7—10).—The cracking of various oils has been investigated in a furnace similar to that of Rittman, consisting of a vertical tube-furnace with internal packing heated electrically. The experiments were carried out at constant temperature and pressure, the pressure being developed initially by the introduction of hydrogen, about 1 g. of which was used per 300 c.c. of the prime material. The results obtained from an Embenski solar distillate having $d_{45}^{20} 0.8793$, flash point 126° , "Akzys" oils 2%, initial b.p. 225° , distillate up to 270° 4%, show that, with cracking temperatures and pressures varying between 478° and 0.25 atm. and 530° and 4.7 atm., increase in the pressure effects increase in the yield of benzine (b.p. to 200°) and diminution of the yield of gaseous products; increase in the temperature at constant pressure results in increased formation of gases. Cracking of decalin, b.p. 187° , at 478° and 5 atm. gives 34% of products b.p. up to 180° and 11 litres of gas per 100 c.c. of the starting material; the total liquid products range in b.p. from 60° to above 260° (74.6% of total). Under 4 atm. pressure, tetralin, b.p. 205° , gives at 484° , 533° , and 575° , 3.5%, 21.3%,

and 10.7% of liquid products b.p. up to 200° , and scarcely any, 2.5, and 19.0 litres of gaseous products per 100 c.c. of tetralin respectively. At 533° the hydrocarbons formed range in b.p. from 130° to 200° and contain no appreciable amount of naphthalene. At 575° the decomposition assumes a different character, since the b.p. range of the resulting hydrocarbons becomes 94 — 200° and a large proportion of naphthalene is obtained; thus the decomposition here consists largely of dehydrogenation. At 467° and 5 atm. pinene yields 22.8% of products b.p. 61 — 145° and 7.0 litres of gas per 100 c.c. of pinene, and limonene at 450° and 5 atm. gives 28.9% of products b.p. 56 — 160° and 2.5 litres of gas. The thermodynamics of the cracking process is briefly considered.

T. H. POPE.

Coke formation with fuel oils and lubricating oils. N. I. TSCHERNOSHUKOV (Izvestia Teplotch. Inst. [Moscow], 1928, 10, 28—31; cf. B., 1929, 85).—Experiments made with Emba naphtha (containing a small proportion of resinous substances), which was heated for 3—6 hrs. at 150 — 175° in presence of oxygen at 15 atm., show that the substances occurring in naphtha and capable of yielding asphaltenes diminish on prolonged oxidation, no renewal of these substances taking place. The compositions of asphaltenes obtained from Grozni wax-free naphtha, turbine oil, and light spindle oil have the following percentage compositions: C 85.2, 83.05, 81.88; H 7.4, 7.35, 7.12; O 6.7, 8.79, 9.30; S 0.7, 1.21, 1.70%, these corresponding with the formulae $C_{355}H_{250}O_{22}S$, $C_{172}H_{176}O_{14}S$, and $C_{136}H_{143}O_{12}S$. The acid precipitate of hydroxy-acids, insoluble in light petroleum, formed by oxidising resin-free oil contained C 71.64, H 8.81, O 19.55%, S nil. When colourless Bachinski oil is heated with 3% of sulphur, reaction commences above 200° , and is rapid at 250° , and extremely so at 270° , hydrogen sulphide being liberated and the resulting oil containing 0.35% (at 220°) or 0.21% (at 250°) S. The asphaltenes obtained by oxidising this oil (1) before and (2) after purification with floridin contained: C 71.62, 74.82; H 6.82, 7.02; O 13.48, 12.41; S 8.08, 5.75%. In these, as in the asphaltenes referred to above, the atomic proportions of carbon and hydrogen are approximately equal.

T. H. POPE.

Knock ratings of pure hydrocarbons. S. F. BIRCH and R. STANSFIELD (Nature, 1929, 123, 490—491).—Diamylene is not such a good anti-knock material as Nash and Howes (B., 1929, 272) suggest, and is certainly not better than trimethylethylene. The observation that polymerisation reduces the anti-knock value was also confirmed for methylcyclohexene and its dimeride methylcyclohexylmethylcyclohexene. The behaviour of cyclohexene depends on the history of the sample, the anti-knock value improving on distillation over sodium and falling on exposure to light and air; the effect is due to the formation of a peroxide. Hydrocarbons containing conjugated double linkings have excellent anti-knock properties, but a diolefine in which the double linkings are not conjugated does not possess marked anti-knock properties. Although an increase in the length of a chain reduces anti-knock properties, the reverse is the case for side chains attached to benzene rings.

A. A. ELDRIDGE.

Knock ratings of pure hydrocarbons. A. W. NASH and D. A. HOWES (*Nature*, 1929, 123, 526—527).—Birch and Stansfield's results (preceding abstract) for trimethylethylene and diamylene refer to concentrations by weight, and the authors' results to concentrations by volume; hence strict comparison is impossible. An acid-refined unsaturated spirit has a lower anti-knock value than the original, since quantitative polymerisation of olefines is not attained in practice. Moreover, standardisation of the air : fuel ratio is necessary for comparison. In the olefine series, and possibly also in the aromatic series, ease of oxidation appears to be intimately connected with anti-knock action. A. A. ELDRIDGE.

Properties and analysis of used crank-case oils. D. G. PRIDGON and H. E. TESTER (*J. Inst. Petroleum Tech.*, 1929, 15, 91—99).—The extent of dilution of a used crank-case oil is satisfactorily determined by measuring the distillate obtained by passing slightly superheated steam into the sample maintained by external heating at 150° until distillation of oil ceases. The use of wet steam at 100° without external heating gives low results. Dilutions up to 14.0% are found in the used oil from nine different cars operating on the same original lubricating oil. So many factors affect the viscosity that no relationship can be traced between this property and the dilution of the oils. The bulked distillate from a number of dilution determinations was a yellow liquid, d_{40}^{20} 0.802, which was decidedly unsaturated and consisted largely of heavy fractions of the motor fuel together with cracked products from the decomposition of the oil. Using synthetic mixtures of fresh lubricating oil and up to 15% of such a diluent, it is shown that when a medium-grade oil contains 10% of diluent it has the properties of a spindle oil, and with 15% of diluent those of a light spindle oil. For any particular engine the percentage dilution of the oil reaches an approximately constant and characteristic value, but thereafter the viscosity slowly increases, and adventitious matter and oxidation products accumulate. With certain types of engine the dilution is such that a heavy grade of oil is necessary if the equilibrated diluted oil is to have a safe viscosity. The quantity of adventitious matter is not a function of percentage dilution.

W. S. NORRIS.

Determination of oxidisability of transformer and turbine oils. N. I. TSCHERNOSHUKOV (*Izvestia Teplotekh. Inst. [Moscow]*, 1928, 10, 13—18).—For practical purposes, transformer and turbo-generator oils may be divided into three classes, according as they give rise on oxidation to a precipitate of (1) asphaltenes or (2) hydroxy-acids, or (3) no precipitate but a greatly increased acidity. Of the many methods devised for examining the oils from this point of view, a number of typical examples, including three rapid methods, are described in detail, and the value of their indications is discussed. The results obtained for a large number of oils by the simple and rapid Butkov test (cf. B., 1929, 119) show that this method is of wide applicability as a means of controlling the quality of the oils in question.

T. H. POPE.

Determination of drop points by Ubbelohde's method. E. NAUMANN (*Chem. Fabr.*, 1929, 136—138).

—The determination of the drop point (temperature at which a semi-solid liquefies sufficiently to drop from the constricted end of a tube) by Ubbelohde's method depends for its accuracy on the use of the prescribed dimensions, otherwise capillarity comes into play. The accuracy required is difficult to secure with glass nipples, and these are fragile. Results of comparative tests using brass nipples on vaseline, asphalt, etc. were concordant and agreed with those obtained with accurate glass nipples. The quantity of brass or glass is too small for any error to be caused by variation in heat capacity or conductivity. The end of the tube may be either sharp or rounded without variation in results.

C. IRWIN.

Electrical conductivity of thin oil films. WATSON and MENON.—See XI. **Petroleum oils for plants.** DE ONG.—See XVI. **"Norit" and "Carboraffin."** REICH and VAVRINECK.—See XVII. **Ignition of fire-damp.** SEGAY.—See XXII.

PATENTS.

Hydrogenation of coal. C. H. LANDER, F. S. SINNATT, and J. G. KING (B.P. 306,564, 17.9.27).—Before subjecting a coal to destructive hydrogenation a material proportion of the fusain and/or durain is removed. The durain may be removed by hand-picking, and the fusain by separation of the finer material from the crushed coal. The coal may also be given a preliminary treatment to modify its caking properties without changing its physical state. It may, for example, be subjected to a preliminary hydrogenation or oxidation, or to the action of gases or vapours at atmospheric or other pressures; or coals which have been subjected to different treatments of the kind described may be blended in suitable proportions. A. B. MANNING.

Retort setting for heating fuel with gases or superheated steam. F. B. DEHN. From Soc. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (B.P. 307,250, 16.5.28).—Coal or other fuel is distilled in a retort comprising a number of chambers through which a current of hot gas or steam is passed, and provided with auxiliary gas or steam pipes so arranged that some or all of the chambers may be supplied with gas or steam directly from one or more generators or superheaters, while part of the hot vapours passing out of some of the chambers may be shunted and part only passed into the following chambers. The temperature in each chamber can thereby be adjusted to control the amount and kind of hydrocarbons which are condensed therein and can be collected in oil separators disposed between the chambers. The fuel passes from chamber to chamber counter-current to the steam until distillation is complete, when it is brought into chambers through which cool or low-pressure steam is passed, these chambers serving as heat exchangers.

A. B. MANNING.

Distillation of carbonaceous materials, tar sands, oil-bearing limestone, etc., and apparatus for subjecting the gases and vapours to the action of liquids. H. NIELSEN and B. LAING (B.P. 306,429, 19.10.27).—The oil vapours evolved in the distillation of carbonaceous materials are subjected to a process of fractional condensation in a series of scrubbers, in each

of which the scrubbing medium consists of an oil of the same or lower b.p. than the corresponding fraction which it is desired to condense. Each scrubber comprises a horizontal cylindrical casing containing a pair of shafts on which are mounted a large number of discs, which by their rotation throw up the oil in the form of a spray and so bring about an intimate contact of the gas with the scrubbing medium. A number of perforated baffle-plates at the exit end of the casing serve to prevent entrainment of oil globules by the gas. A part of the oil condensed in each scrubber serves as scrubbing medium for the immediately preceding scrubber.

A. B. MANNING.

Distillation of solid carbonaceous or oil-bearing materials. H. NIELSEN and B. LAING (B.P. 306,654, 23.12.27).—The material is distilled in a current of heated gas and, before its entry into the distillation chamber, is heated to a temperature, between 100° and 250°, dependent on the volume of gas used, but above that at which the heaviest oil fractions present in the vapour are liable to condense. The walls of the retort, the conduits, and the dust extractor are similarly heated to prevent condensation therein. A. B. MANNING.

Low-temperature carbonisation of bituminous and oil-bearing materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,723, 9.3.28).—The material to be carbonised is passed through a sheaf of tubes mounted longitudinally in an inclined cylindrical rotary retort. Hot gases are circulated through the retort, which is lined with heat-insulating material. The heating gases may be introduced and carried away through a centrally disposed guide pipe.

A. B. MANNING.

Manufacture of coal gas. SOUTH METROPOLITAN GAS CO., E. V. EVANS, and H. STANIER (B.P. 306,435, 19.11.27).—The gases and vapours produced by the low-temperature carbonisation of coal are mixed with hydrogen or a gas containing hydrogen, *e.g.*, water-gas, and the mixture is subjected to a high temperature in a cracking chamber. The water-gas may be passed through the retort in which the coal is being carbonised, or may be preheated and added to the volatile products of carbonisation as they are leaving the retort. A gas suitable for town supply is produced, and the tar approximates in character to ordinary high-temperature tar.

A. B. MANNING.

Manufacture of gas from heavy oils. C. CHILOVSKY (B.P. 279,834, 20.10.27. Fr., 27.10.26).—The apparatus for carrying out the process described in B.P. 255,423, 271,899, and 271,907 (B., 1927, 805, 867) is modified in many details. In particular, the gases after leaving the catalyst are caused to pass back along the outer walls of the vaporisation chamber, the air for partial combustion is preheated by being circulated through a jacket surrounding the apparatus, the catalyst is so constructed that the oil cloud will circulate freely through it in straight channels, and the steam which is added to the cloud of oil is generated in a coil heated by the gases leaving the apparatus. Other modifications relate to the use of the process in conjunction with an internal-combustion engine. A. B. MANNING.

Gas producers. O. MISCH (B.P. 306,614, 28.11.27).

—A vertical shaft is divided into three sections, the uppermost section forming an externally-heated distillation retort, the middle section a water-gas generator, and the lowest section a producer-gas generator. The lower end of the retort is heated by means of a compressed-air burner supplied with hot producer gas, and is maintained at a high temperature, *e.g.*, about 1300°, with a fuel which is converted into coke at 700°, in order that coke should be produced as rapidly as it is used in the gas generators below, so that the process is continuous. The distillation gases and the water-gas are withdrawn by suction at the upper end of the retort. The producer gas is similarly withdrawn by suction at the upper end of the producer section. Tar residues obtained from the distillation gas are returned to the retort to be cracked. A. B. MANNING.

Apparatus for fractional distillation of tar, crude oils, petroleum, and other liquids. O. ELSTERMANN and A. BAUMHÖR (B.P. 307,243, 30.4.28).—The liquid is distilled in a column made up of a number of superimposed funnel-shaped units, which are jacketed and heated to successively higher temperatures by suitable heating medium. The distilled products from each unit pass out through a discharge pipe to a condensing system; the remaining liquid passes into the next lower unit. The liquid entering each unit is ejected in a thin layer over the inner wall of the unit by means of a rotary distributor attached to a common central shaft, and moves down the wall in a helical path. A rotating scraper attached to the central shaft in each unit prevents any sediment sticking to the walls.

A. B. MANNING.

Separation of the acid constituents from low-temperature tar or its fractions. C. BUNGE (B.P. 306,738, 27.3.28).—The tar or tar fraction is emulsified with water or distillation liquor and a little soap solution, and the emulsion treated with a solvent for the acid constituents, *e.g.*, an alkali solution, alcohol, etc. The mixture is kept, or the separation of the hydrocarbons and the solution containing the phenols may be effected by centrifuging.

A. B. MANNING.

Apparatus for preliminary separation of crude benzoles. SOC. DES ETABL. BARBET (B.P. 295,591, 13.8.28. Fr., 12.8.27).—Crude benzol, free from water, passes through a heater to a rectifying column from which are obtained three distinct fractions, *viz.*, a fraction containing light impurities, a 90–95% benzol, and a residual toluol fraction. Light products are removed from the cooled benzol and the crude benzol fraction is passed to a second rectifying column, where it is exhausted of its xylol. The xylol is withdrawn leaving a residue of still heavier products, which are passed to a discontinuous vacuum rectifier for the production of a solvent naphtha fraction and a tarry residue from which naphthalene can be crystallised. H. S. GARLICK.

Manufacture of hydrocarbons of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,437, 20.10.27).—Components of mineral oils soluble in liquid sulphur dioxide and liquid at ordinary temperatures are converted into hydrocarbons of low b.p. and rich in aromatic compounds by treatment with anhydrous aluminium chloride and/or aluminium metal at above

100° while passing an inert gas (dry hydrogen chloride etc.) through the reaction mixture, with or without application of pressure. H. S. GARLICK.

Production of light hydrocarbons. J. M. F. D. FLORENTIN, A. J. KLING, and C. MATIGNON (B.P. 279,488, 20.10.27. Fr., 23.10.26. Cf. B., 1927, 548, 836).—Coal tar is treated with hydrogen under high pressure and at 350–480° in the presence of a dehydrating catalyst, *e.g.*, thoria, and a "substitutive" catalyst, *e.g.*, a metal halide, to hydrogenate the phenols and polynuclear compounds (naphthalene, anthracene).

A. B. MANNING.

Apparatus for distilling oil. H. S. PETTINGILL, JUN., ASSR. to EMPIRE REFINERIES, INC. (U.S.P. 1,698,507, 8.1.29. Appl., 14.12.22).—Means for scraping the still are claimed.

Refining petroleum oils. SHARPLES SPECIALTY CO., ASSEES. OF L. D. JONES (B.P. 280,559, 9.11.27. U.S., 9.11.26).—Petroleum oils containing crystalline wax may be centrifugally dewaxed if, before chilling, sufficient oil-free petrolatum or oil-containing amorphous wax is added to produce a plastic precipitate of sufficient density that the wax is separated from the oil on centrifuging. Substances other than hard asphalt, normally removed by extensive acid or clay treatment, may be retained in or added to the oil; by adding the amorphous wax and chilling they are concentrated in the precipitate.

H. S. GARLICK.

Treatment of hydrocarbon oil. C. ARNOLD. From IMPERIAL OIL, LTD. (B.P. 305,108, 30.8.27).—Hydrocarbon vapours are led into the lower part of a rectification tower and passed upwards through a contact zone, to which is fed a descending stream of liquid oil carrying in suspension a finely-divided solid absorbent, *e.g.*, clay, with or without the addition of a reagent for removing sulphur, with which the vapours are brought into intimate contact. The temperature is regulated so that part of the desired product comes into contact with the clay temporarily as liquid, thereafter passing off as vapour to suitable condensing apparatus. The bottom of the tower is connected to a settling tank in which condensate is separated from the clay for return to the distillation zone.

H. S. GARLICK.

Treatment of mineral oils by heat. W. J. PERELIS (B.P. 277,983, 22.9.27. U.S., 23.9.26).—In the type of pressure-cracking processes under flow in which, after the heating period, an extended cracking treatment is given in heat-insulated tanks or drums, carbon formation and coke deposition are avoided by agitation either mechanically or by means of a tubular cooling apparatus through which the material is passed under conditions of turbulent flow before entering the tank; in either case the temperature of the material is kept within the cracking range. The agitation may be effected during the heating period by heating the material under low pressure whereby the lighter constituents vaporise, thus increasing the volume to be moved and causing turbulent flow, thereafter increasing the pressure during the cracking reaction.

H. S. GARLICK.

Cracking of petroleum products. A. SACHANEN and M. TILITSCHYEYEV (B.P. 307,105, 2.12.27).—Oil, preheated by the heavy residue from the expansion

chamber, is forced under high pressure at sufficient velocity to prevent carbon deposition through two sets of tubes connected in series and disposed in the same chamber maintained at the required high temperature by the admission of furnace gases. The first set of tubes (the heating tubes) are of considerably smaller diameter than the second set (the reaction tubes). The resulting products are led into a coil within and at the bottom of the rectifying column, and thence through a reducing valve into an expansion chamber maintained at a pressure not substantially exceeding atmospheric, in which carbon is deposited. The vapours are led into a rectifying column, where they are cooled by contact with coils carrying untreated oil, the condensate passing through a gas separator into a container. Residues from the bottom of the expansion chamber and condensate from the bottom of the rectifying column are returned to the pipe circuit leading to the heating and reaction tubes.

H. S. GARLICK.

Distilling or cracking oils and like carbonaceous liquids. A. L. RISPLER (B.P. 305,263, 3.11.27).—Oil is passed through a heated region of a furnace-fired rotary still in which vaporisation of all but the heavier fractions takes place. The residual carbon and asphaltic matter falls upon crushing members and iron balls which roll loosely within the still, and are sufficiently hot to decompose the asphaltic matter; the resulting carbon is removed by means of a steam blast.

H. S. GARLICK.

Motor fuel. E. C. HENNEN, ASSR. to U.S. NITO CO., INC. (U.S.P. 1,682,561, 28.8.28. Appl., 7.3.28).—High-heat-test lubricating oil, castor oil, and a benzene solution of *p*-chloronitrobenzene are added to gasoline etc.

R. BRIGHTMAN.

Preventing knocking of motor fuel. H. D. ELKINGTON. From GEBR. AVENARIUS (B.P. 277,326, 6.9.27).—Knocking is prevented by the addition of 0.3–0.5% of one or more esters of unsaturated fatty acids of high mol. wt., *e.g.*, an oleic ester, fatty acid glyceryl ester, or vegetable oil, such as olive oil. The anti-detonant may be treated before use with oxygen or ozone.

H. S. GARLICK.

Emulsions from partially oxidised petroleum wax, and their manufacture. ALOX CHEM. CORP. (B.P. 287,514, 10.10.27. U.S., 23.3.27).—Normally solid petroleum wax is treated at 160° under pressure with oxygen in the presence of a suitable catalyst, until the acidic contents amount to 30% by wt. The resulting waxes are partially or completely neutralised with alkali either directly or after washing, pressure filtration, or other treatment to remove highly acidic substances, and emulsified with water. The resulting emulsion is suitable for use in textile dressings or for coating paper.

H. S. GARLICK.

Production of products from brown coal analogous to montan wax. J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 307,111, 3.12.27).—Brown coal is chlorinated and extracted with a solvent containing aromatic hydrocarbons. After evaporation of the solvent the product is melted to expel residual solvent and hydrochloric acid, and may, if desired, be further purified by redissolving.

A. B. MANNING.

Extraction of montan wax. A. RIEBECK'SCHE MONTANWERKE A.-G. (B.P. 288,197, 12.3.28. Ger., 4.4.27).—Brown coal is extracted under a small excess pressure, not exceeding 2 atm., produced by correspondingly raising the temperature of the solvent used. The usual apparatus for the extraction at atmospheric pressure is used, modified only by the substitution of a pressure separating vessel for the Florentine flask and the addition of a pressure regulator. A. B. MANNING.

Plant for washing coal and other minerals. A. FRANCE (U.S.P. 1,704,185, 5.3.29. Appl., 7.11.27. Belg., 24.2.27).—See B.P. 285,864; B., 1928, 737.

Apparatus for carbonising coal. R. L. RODGERS, ASSR. to CHARCOLITE CORP. (U.S.P. 1,704,103, 5.3.29. Appl., 28.9.22).—See B.P. 256,038; B., 1926, 812.

Apparatus for distillation or heat treatment of carbonaceous or other materials. Apparatus for producing coke and gas from solid carbonaceous material. Gas and coke plant apparatus. F. D. MARSHALL (U.S.P. 1,704,092—4, 5.3.29. Appl., [A—C] 10.4.26. U.K., [A, B] 5.6.25. Renewed [B] 10.12.28).—See B.P. 261,919, 261,927, and 261,975; B., 1927, 244.

Regenerative heating apparatus [for coke ovens]. J. VAN ACKEREN (U.S.P. 1,704,229 and 1,704,685—7, 5.3.29. Appl., [A] 23.2.23, [B] 6.1.22, [C] 22.4.22, [D] 24.7.22).—See B.P. 211,828, 191,393, 210,085, and 201,130; B., 1925, 90; 1924, 587, 662, 702.

Rotary [gas] scrubber and washer. T. J. NODEN (U.S.P. 1,704,663, 5.3.29. Appl., 30.6.24. U.K., 16.7.23).—See B.P. 211,409; B., 1924, 326.

Petroleum refining process and reagent for removal of sulphur. AXTELL RES. LABS. (B.P. 282,738, 9.12.27. U.S., 27.12.26).—See U.S.P. 1,645,679; B., 1928, 116.

Quenching coke by means of water. A. H. LYMN, N. J. BOWATER, and CHAMBER OVENS, LTD. (B.P. 307,204, 25.2.28).

Pusher rams for coke ovens. KOPPERS CO., ASSEES. OF C. B. COLLINS and J. A. B. LOVETT (B.P. 284,311, 16.12.27. U.S., 29.1.27).

Charging hoppers for gas producers and other furnaces. J. SAUVAGEOT (B.P. 289,458, 29.3.28. Fr., 29.4.27).

Cleansing of waste lubricating and other oils [by treatment with water]. C. E. FOX (B.P. 306,771, 1.6.28).

Lead alkyl compounds (U.S.P. 1,694,268).—See VII.

III.—ORGANIC INTERMEDIATES.

Detection of maltol and salicylic acid. MERL and BEITTER.—See XIX.

PATENTS.

Manufacture of synthetic organic compounds [from carbon oxides]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,294, 7.7.27).—Oxides of carbon are reduced to ethyl alcohol, higher alcohols, hydrocarbons, and acids at high temperatures and

under normal or high pressures in presence of a catalyst containing cadmium or thallium or their compounds, in addition to considerable amounts of iron, nickel, or cobalt or their compounds. The catalysts are free from metallic halides and the treatment is carried out without an addition of alcohols, esters, or aldehydes. The iron, nickel, or cobalt may be replaced or used in conjunction with one or more elements of the 8th periodic group, and copper, gold, silver, cerium, zirconium, aluminium, vanadium, uranium, chromium, molybdenum, tungsten, manganese, alkali metals, or alkaline-earth metals, or their compounds may be present provided that less than 5% of the catalyst is constituted of difficultly reducible metallic oxides. R. BRIGHTMAN.

Chlorination of hydrocarbons. INTERNAT. FIRE-PROOF PRODUCTS CORP., ASSEES. OF F. S. VIVAS (B.P. 286,726, 8.3.28. U.S., 11.3.27).—Heavy oils are removed from commercial petrol by saponification with alkali and the hydrocarbon oil is chlorinated in presence of a catalyst (aluminium chloride or ferric chloride), carbon tetrachloride, and about 1% of aqueous ammonia. The product, principally heptachlorohexane, is a cleaning solvent. C. HOLLINS.

Manufacture of esters. IMPERIAL CHEM. INDUSTRIES, LTD., W. GIBSON, and J. B. PAYMAN (B.P. 307,137, 21.12.27).—An aliphatic acid amide is heated with an alcohol, with or without a diluent, in presence of a metallic salt (zinc chloride, mercuric chloride). In the case of formic esters having lower b.p. than the alcohol used the ester is with advantage distilled off as formed. The preparation of *n*-butyl formate, *iso*amyl acetate, glycol monoacetate, and cyclohexyl formate (the latter under reduced pressure) is described. C. HOLLINS.

Continuous manufacture of anhydrous acetic acid from its aqueous solutions. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 296,974, 13.1.28. Fr., 10.9.27, and Addn. B.P. 301,415, 3.2.28. Fr., 29.11.27).—Acetic acid is extracted from its solutions in (A) liquid or (B) vapour phase by means of a solvent of b.p. between 120° and 150° (e.g., amyl acetate). The extract is freed from water by means of an entraining liquid (e.g., ethyl acetate), and can then readily be fractionated for the recovery of anhydrous acetic acid. Continuous processes are described. C. HOLLINS.

Manufacture of *o*-nitro- and *o*-amino-diaryl ethers. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 307,130, 15.12.27).—An *o*-dinitro-compound of the benzene series is heated with an alkali phenoxide at 100° for about an hour; one nitro-group is replaced by an aryloxy-group. The nitrodiaryl ether may then be reduced, e.g., with iron and acetic acid. Examples are: *o*-nitro- and *o*-amino-diphenyl ethers; 5-chloro-2-nitrodiphenyl ether, m.p. 85°, and the amine, m.p. 40–41°; 2:5:5'-trichloro-2'-nitrodiphenyl ether, m.p. 97–98°, and the amine, m.p. 74–75°; 5-chloro-2-nitrophenyl β -naphthyl ether, m.p. 109–110°, and the amine, m.p. 108–109°; 5':4'-dichloro-2-nitrodiphenyl ether, m.p. 80–81°, and 5:5'-dichloro-2-aminodiphenyl ether, m.p. 76–77°. C. HOLLINS.

Production of aromatic carboxylic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,223,

21.3.28).—Aromatic hydrocarbons etc. are carboxylated by the action of carbon dioxide at 80–150°/50–60 atm. pressure in presence of aluminium chloride; considerable amounts of diaryl ketones are obtained as by-products. Examples are: benzoic acid from benzene, *p*-chlorobenzoic acid from chlorobenzene, *p*-toluic acid from toluene, 2:4-dimethylbenzoic acid, m.p. 126°, from *m*-xylene. C. HOLLINS.

Manufacture of hydrogenated aromatic amino-compounds. K. and K. S. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,285, 4.5. and 22.11.27).—Secondary and tertiary arylamines of the benzene series and acylated naphthylamines or their nuclear derivatives are hydrogenated in the liquid phase in presence of nickel, cobalt, or copper catalysts. The preparation of the following is described: cyclohexyl-ethylamine and -dimethylamine, and tetrahydro-derivatives of ethyl 2-acetamido-3-naphthoate (hydrolysis gives ar-2-amino-tetrahydro-3-naphthoic acid, m.p. 180–182°), of 1-acetamido-2-naphthyl ethyl ether (hydrolysis gives the amine, m.p. 54–55°, b.p. 176–178°/12 mm.), and of 1-acetamido-7-naphthyl methyl ether, m.p. 161° (hydrolysis gives ar-1-aminotetrahydro-7-naphthyl methyl ether, m.p. 72–73°, b.p. 182–183°/12 mm.). Azo dyes from the amines are described. C. HOLLINS.

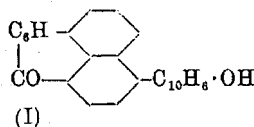
Manufacture of condensation products of dimethylolurea or dimethylolthiourea. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,875, 27.8.27).—The primary condensation products from carbamide or thiocarbamide and formaldehyde are further condensed in an aqueous medium at 100°, a buffer salt being added to produce a *p*_H of 4 to 7, preferably 6.3. A continuous process is described. (Cf. B.P. 258,289; B., 1928, 376.) C. HOLLINS.

Preparation of tetrazoles. A. BOEHRINGER (B.P. 280,529, 1.11.27. Ger., 10.11.26).—The esters, e.g., the benzenesulphonates, of oximes (especially of cyclic ketoximes) or their Beckmann transformation products are treated with sodium azide or azoimide in absence of concentrated mineral acid. Thus cyclohexanoneoxime *O*-benzenesulphonate with sodium azide yields 2:3-pentamethylenetetrazole, ϵ -leucine lactam being formed as by-product. C. HOLLINS.

Purification of crude anthracene. A. O. JAEGER, Assr. to SELDEN RES. & ENG. CORP. (U.S.P. 1,693,713, 4.12.28. Appl., 16.1.28).—Furan derivatives, especially methylfurfuraldehyde, readily dissolve carbazole and phenanthrene but very little anthracene in the cold. Two crystallisations from furfuraldehyde or methylfurfuraldehyde at 80° give pure anthracene. Other solvents may be used in combination with these. C. HOLLINS.

Manufacture of condensation products of the benzanthrone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,331, 25.8.27).—Benzantrones having free 4- and 3-positions are heated with aromatic hydroxy-compounds having free *o*- or *p*-positions in presence of alkali hydroxide in quantity only slightly in excess of that required for the formation of the phenoxide. Benzantrones and β -naphthol at 200° give 1-(4-benzantronyl)- β -naphthol (I), bright yellow

turning red at 260°, m.p. above 300°. 6-Bromo-2-naphthol gives a similar product, and the product from



β -naphthol and 6-chlorobenzanthrone is described. Similar products from α -naphthol or 2-chloro- α -naphthol and benzanthrone, from α -naphthol and benzo-

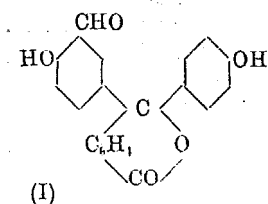
benzanthrone-13-carboxylic acid, and from benzanthrone and *m*-2-xylene (yellow) are described.

R. BRIGHTMAN.

Sulphonic acids (G.P. 451,534).—See XV. **Betaine hydrochloride** (B.P. 304,071).—See XVII.

IV.—DYESTUFFS.

Aldehydophenolphthalein and dyes derived from it. R. N. SEN and K. C. KAR (J. Indian Chem. Soc., 1929, 6, 53–63).—The Reimer-Tiemann reaction with



phenolphthalein affords 20% of aldehydophenolphthalein (I), m.p. 97–99° (phenylhydrazones, m.p. 148°: dioxime, decomp. 235°), which yields a condensation product, phenolphthaleinoin, m.p. 152°, analogous to benzoin. An alkaline solution of I is more

deeply coloured than an alkaline solution of phenolphthalein. Condensation of I with *p*-toluidine (m.p. 140°), β -naphthylamine (m.p. 154°), aminoazobenzene (m.p. 235°), benzidine (m.p. 191°), *o*- and *p*-phenylenediamines (m.p. 218° and 230°), chrysoidine, safranin, and rosaniline gives the corresponding azomethine dyes having the m.p. given in parentheses; each amino-group reacts with 1 mol. of I. A diminution in the intensity of colour of the last three substances is thereby produced. These azomethines dye wool and silk with shades varying from light yellow to bluish-red. Pyronine dyes are obtained by condensing I with resorcinol, β -naphthol, quinol, pyrogallol, and *m*-diethylaminophenol, in presence of warm sulphuric acid. These dyes form red potassium salts, and dye wool and silk with shades varying from light brown to red. Condensation of I with dimethylaniline in presence of hydrochloric acid gives pp'-tetramethyldiaminodiphenylphenolphthaleinylmethane, m.p. 187°, which when oxidised with lead dioxide gives a bluish-green dye (phenolphthalein-green). When I is condensed with *o*-cresotic acid in presence of sulphuric acid and the resulting leuco-compound, m.p. 215°, oxidised with nitrosyl sulphate at 50–60° a red dye is produced. This dye wool with a red shade which deepens to black on after-chroming.

H. BURTON.

Fluorescence of dyes in Wood's light. A. SEYEWETZ and J. BLANC (Compt. rend., 1929, 188, 714–715).—A preliminary classification of the fluorescence of dyes in the light of a mercury-vapour lamp, filtered through a screen of Wood's glass (3650 Å. and a slight band at 3500–3700 Å.), is made. Aqueous or aqueous-alcoholic solutions are used, since examination of the powdered dyes does not afford consistent results. Substances such as halogens, alkali halides, silver nitrate,

etc. are found to destroy the fluorescence of fluorescein. Dyed textiles show generally the same fluorescence as the corresponding solution; undyed textiles exhibit but a slight fluorescence.

G. A. C. GOUGH.

Colouring matters from sucrose. GARINO and TOSONOTTI.—See XVII.

PATENTS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (B.P. [A] 274,128, 11.7.27. Ger., 9.7.26, and [B] Addn. B.P. 275,613, 28.7.27. Ger., 5.8.26).—(A) 6-Halogeno- or 2:6-dihalogeno-*m*-xylydines, obtained by halogenating *as-m*-xylydine in sulphuric acid, are diazotised and coupled in substance or on the fibre with an arylamide of 2:3-hydroxynaphthoic acid, other than the *m*-4-xylylamide or its derivatives. Thus 6-chloro- or 6-bromo-*m*-4-xylydine coupled with the 4-chloro-*o*-aniside, 5-chloro-*o*-toluidide, or the β -naphthylamide give bright yellowish or neutral red shades very fast to kier-boiling, light, and chlorine. 2:6-Dichloro-*m*-xylydine similarly gives red, scarlet, and claret shades, respectively. (B) 2-Halogeno- or 2-halogeno-6-alkyl-*m*-xylydines are coupled with an arylide of 2:3-hydroxynaphthoic acid. Thus 2-chloro-*m*-xylydine (cf. Nölting, Braun, and Thesmar, B., 1901, 797) and 3-chloro- ψ -cumidine, m.p. 66–67° (obtained by chlorinating ψ -cumidine sulphate), with the 5-chloro-*o*-toluidide give bluish-red dyeings of good fastness to kier-boiling and chlorine.

R. BRIGHTMAN.

Production of [acid] azo dyes. K. HEUSNER and M. SIMON, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,783, 6.11.28. Appl., 7.6.27. Ger., 6.4.25).—Diao compounds are coupled with β -naphthylsulphamic acids; e.g., sulphanilic acid \rightarrow β -naphthylsulphamic acid (orange), metanilic acid \rightarrow 6-sulpho- β -naphthylsulphamic acid (yellowish-orange).

R. BRIGHTMAN.

Manufacture of [acid azo] dyes. SOC. CHEM. IND. IN BASLE (B.P. 281,713, 3.12.27. Switz., 4.12.26).—Dyes obtained by coupling a pyrazolone with diazotised *m*-aminoacetanilide-4-sulphonic acid, 4-aminoaceto-*o*-toluidide-5-sulphonic acid, or 5-chloro-*m*-aminoacetanilide-4-sulphonic acid, give level yellow shades, fast to light, on wool (cf. B.P. 9968 of 1910).

C. HOLLINS.

Manufacture of azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 307,104, 2.12.27).—*p*-Aminoacetophenone, *p*-aminobenzophenone, or a *m*-nitro-derivative of these is diazotised and coupled with acetoacetic arylamides to give green-yellow dyes for oil pigments and size colours. Examples are: *p*-aminoacetophenone \rightarrow acetoacetanilide; 3-nitro-4-aminoacetophenone \rightarrow acetoacetic *o*-chloroanilide (product, m.p. 269–270°); *p*-aminobenzophenone \rightarrow acetoacetic *o*-chloroanilide (product, m.p. 159–160°).

C. HOLLINS.

Manufacture of insoluble [azo] dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 307,150, 31.12.27).—Safranisoie (3:7-diamino-8-methoxy-5-*o*-anisylphenazonium chloride) is tetrazotised and coupled in substance or on the fibre with a 2:3-hydroxynaphthoic arylamide. The *p*-phenetide, β -naphthylamide, and *m*-toluidide give deep blacks, the *m*-nitroanilide and *m*-aniside black-violets, and the anilide a black-brown. Special fastness to chlorine is claimed.

C. HOLLINS.

Manufacture of vat [benzanthrone] dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,874, 22.8.27 and 1.2.28).—Dark blue to violet to black vat dyes result from the alkaline fusion of benzanthrone-1-aminoanthraquinones obtained by condensing an amino- or diamino-anthraquinone with mono- or di-chloro-benzantrones (other than those prepared from 1-chloroanthraquinone) containing no substituents or containing in the 3- or 4-position an etherified hydroxyl group or a 4'- or 3'-benzanthranyl group. Examples are the alkaline fusion of condensation products of 1-aminoanthraquinone with 2-, 9-, 10-, or 11-chlorobenzanthrone, 9-chloro- or 9:9'- or 10:10'-dichloro-4:4'-dibenzantranyl, dibrominated 4:4'-dibenzantranyl, 9:9'-, 10:10'-, or 11:11'-dichloro-3:3'-dibenzantranyl, 9-, 10-, or 11-chloro-3-methoxybenzanthrone, brominated 3-methoxybenzanthrone, 9-halogeno-3-phenoxybenzanthrone, 9-chloro-4:3'-dibenzantranyl, or dibrominated 4:3'-dibenzantranyl; of 1-amino-4-methoxyanthraquinone with 9-chlorobenzanthrone, 9:9'-dichloro-3:3'- or -4:4'-dibenzantranyl, 9-chloro-3-methoxybenzanthrone, or brominated 4:3'-dibenzantranyl; of 2-aminoanthraquinone or 1:6- or 1:5-diaminoanthraquinone with 9-chlorobenzanthrone; of 1:6-diaminoanthraquinone with 9:9'-dichloro-3:3'-dibenzantranyl or dibrominated 4:3'-dibenzantranyl; of 1-amino-4-hydroxyanthraquinone with 9:9'-dichloro-3:3'-dibenzantranyl; of 3-aminoalizarin with dibrominated 4:3'-dibenzantranyl; and of 1-chloroanthraquinone-2-carboxylic acid with 9-aminobenzanthrone.

C. HOLLINS.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,432, 1.2.28).—Tetrahalogeno-2:2'-dibenzantranyls are heated with aminoanthraquinones in presence of copper or other metallic compounds and of an acid-fixing agent. *Tetrabromo-2:2'-dibenzantranyl*, obtained, e.g., by brominating 2:2'-dibenzantranyl in chlorosulphonic acid, with 1-aminoanthraquinone in nitrobenzene in presence of sodium carbonate and cupric oxide gives a fast black vat dye; a similar dye is obtained at 320–340° in absence of nitrobenzene; with 1-amino-4-methoxyanthraquinone a blue-violet vat dye is obtained, with 1:6-diaminoanthraquinone, a greyish-black vat dye, with 2-aminoanthraquinone or, e.g., 3-chloro-2-aminoanthraquinone at 300–340°, a greyish-black vat dye, and with 3- or 4-aminoalizarin a blue-black vat dye. 9:9'-Dichlorodibromo-2:2'-dibenzantranyl, obtained by brominating 9:9'-dichlorodibenzantranyl with excess of bromine, with 1-aminoanthraquinone gives a blue-black vat dye.

R. BRIGHTMAN.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,219, 7.7.27).—2-Halogenoanthrones containing a further nucleus attached to the *meso*- and 1-positions by means of a nitrogen atom at least in the 1-position, are heated at 200–250° with a metal, to give bright yellow or orange vat dyes. 3-Halogenopyrazolantrones, e.g., 3-bromopyrazolanthrone (from 2-bromo-1-aminoanthraquinone), are previously acetylated, the acyl group being removed from the condensation product by hydrolysis if desired. *Acetyl-3-bromopyrazolanthrone*, m.p. 209–211°, affords a yellow acetyl condensation product, hydrolysed by

sulphuric acid to a yellow dye. 4-Bromopyrimidone-anthrone gives a *methyl ether*, m.p. 285–290°, converted when heated with copper and naphthalene into a yellow vat dye. Dyes similarly obtained from 4-iodomethylpyrimidanthrone, m.p. 210–215° (from *C*-methyl-2:1-anthraquinoneoxazole and ammonia through 4-amino-methylpyrimidanthrone), 6-iodocoeramidonine, m.p. 203–205°, 6-bromocoeramidonine, m.p. 210°, and from the *methyl ether*, m.p. 255–260°, of 4-bromopyridonanthrone are described. R. BRIGHTMAN.

Manufacture of indigoid vat dyes. I. G. FARBENIND. A.-G. (B.P. 300,114, 8.3.28. Ger., 5.11.27. Addn. to B.P. 282,805, 1928, 849).—In the process of the prior patent isatin α -chlorides are used in place of α -arylamides. C. HOLLINS.

Manufacture of sulphur dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,909, 4.8.27).—Sulphide dyes giving light-fast green shades are obtained by melting with a polysulphide in presence of a copper salt the indophenols obtained by oxidation of *p*-aminophenols with alkyl ethers of *p*-hydroxyaryl-1-naphthylamine-6-, -7-, or -8-sulphonic acids. The dyes from 1-*p*-anisylamino-, 1-*p*-phenetyl-amino-, and 1-*p*-anisylamino-4-*p*-hydroxyphenylamino-naphthalene-8-sulphonic acid are described. The *p*-anisyl-1-naphthylamine-6-, -7- and -8-sulphonic acids and *p*-phenetyl-1-naphthylamine-8-sulphonic acid are obtained by the process of B.P. 7337/1892 (B., 1893, 350). R. BRIGHTMAN.

Finely-divided phenolphthalein and its manufacture. E. B. PUTT (U.S.P. 1,693,666, 4.12.28. Appl., 11.5.26).—Phenolphthalein is precipitated from an alkaline solution containing gum acacia to retard crystallisation. C. HOLLINS.

Azo dyes and their preparation. H. EICHWEDE, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,694,117, 4.12.28. Appl., 21.12.26. Ger., 23.12.25).—See B.P. 263,816; B., 1928, 183.

Anthraquinone vat dye. W. MIEG, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,236, 6.11.28. Appl., 20.1.26. Ger., 21.1.25).—See B.P. 297,133; B., 1928, 849.

Vat dyes of the 2:2'-indole-thionaphthen-indigo series. C. KRAUSS, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,691,660, 13.11.28. Appl., 11.6.27. Ger., 15.6.26).—See B.P. 295,239; B., 1928, 740.

Derivatives of dibenzpyrenequinone and their preparation. G. KRANZLEIN, K. ZAHN, P. OCHWAT, and M. CORELL, ASSTS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,693,447, 27.11.28. Appl., 28.12.26. Ger., 30.11.25).—See B.P. 263,845; B., 1927, 870.

[Manufacture of] vat dye. H. NERESHEIMER, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,703,948, 5.3.29. Appl., 22.12.27. Ger., 23.12.26).—See B.P. 282,629; B., 1928, 517.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Colour reactions of lignin. F. PODBREZNÍK (Bull. Inst. Pin, 1928, 233–236, 245–250; Pulp & Paper Mag., 1929, 27, 229–248).—The colour reactions of lignin with various primary and secondary aromatic

amines, phenols, naphthols, and certain oxidising and reducing agents are reviewed and a new characteristic reaction is described depending on the action of hydrochloric acid and potassium chlorate solution, with which pine sawdust and lignin give, on warming, an orange colour turning yellow on addition of ammonia. From the facts that (a) extracted lignins (Willstätter's lignin, phenol-lignin, alkali-lignin), oxidised lignin, and lignin freed from pentosans give the same colour reactions as lignified tissue, and (b) that the alcoholic and ethereal extracts of sawdust do not give some of the most characteristic colour reactions of lignin, it is concluded that these reactions are due in the main to the lignin complex itself, and not, as previously suggested, to methyl-pentosans or aromatic compounds such as vanillin, coniferyl alcohol, etc., though these last-named, which are probably not present in the original wood, but are formed by depolymerisation or hydrolysis of the lignin during the extraction process, may influence the reactions to a certain extent. D. J. NORMAN.

Alteration of sugar-like substances in sulphite-cellulose cooking. II. E. HÄGGLUND and T. JOHNSON (Svensk Kem. Tidskr., 1929, 41, 55–59; cf. B., 1929, 202).—The sugars (mainly mannose, xylose, and dextrose, with small amounts of galactose and levulose) formed during the manufacture of cellulose are readily oxidised to carboxylic acids by boiling with the calcium sulphite liquor. In an extract prepared by boiling 332 g. of air-dried wood with 1500 c.c. of a sulphite liquor containing 4.6% of sulphurous acid and 1.32% of lime for 20 hrs. at 135° (maximum temperature), the presence of mannonic and xylonic acids has been definitely proved, the former being identified by its brucine compound and phenylhydrazones, the latter by its double salt with cadmium bromide. Tests for gluconic acid, on the other hand, were negative. H. F. HARWOOD.

Detergent action of soap. RHODES and BRAINARD.—See XII. **Treatment of [wool] trade waste.** SNELL. **Treatment of wastes from paper mills.** FALES.—See XXIII.

PATENTS.

Manufacture of colloidal solutions of natural silk. T. MUTO, S. HIDA, and KANEGAFUCHI BOSEKI KABUSHIKI KWAISHA (B.P. 306,699, 18.2.28).—Silk fibre is treated with a solution of a magnesium salt, e.g., nitrate, which is afterwards removed from the colloidal solution by dialysis etc. F. R. ENNOS.

Production of [non-creasing] cellulosic fabric. TOOTAL BROADHURST LEE Co., R. P. FOULDS, and J. T. MARSH (B.P. 304,900, 9.1.28).—The fabric is mercerised with caustic soda, washed, and impregnated, while still in the wet swollen condition, with a synthetic resin or the components thereof. After drying at a low temperature it is heated for a short time, and is finally soaped, washed, and dried. F. R. ENNOS.

Preparation of artificial silk. S. I. VLES and D. VAN DER WANT (B.P. 306,050, 10. and 12.9.27).—A workable artificial silk yarn is made from untwisted bundles of filaments by treating them with substances which are solvents or swelling agents at the working temperature or a few degrees above it, so that different

parts of each bundle are fixed together by adhesion at regular or irregular intervals throughout its length.

F. R. ENNOS.

Production of artificial filaments or threads.

BRIT. CELANESE, LTD., H. DREYFUS, E. KINSELLA, J. BOWER, and W. I. TAYLOR (B.P. 304,674, 24.10.27. Addn. to B.P. 300,998; B., 1929, 203).—Cellulose acetate is spun vertically downwards into heated chambers or cells each of which, containing any desired number of spinning nozzles, is either open at the end remote from the nozzles or is partially closed to ensure even distribution of the air. The chambers are provided with a collector device, so that substantially the whole of the air is drawn through the vicinity of the spinning nozzles.

F. R. ENNOS.

Preparation of artificial textile products [viscose silks] with reduced lustre. G. BONWITT (B.P. 288,222, 7.9.27. Holl., 7.4.27. Addn. to B.P. 285,066; B., 1929, 203).—In applying the process of the prior patent to viscose silks having denier numbers (D) of 6 or less, the chlorobenzene particles must be of diameter not greater than x microns, where $D^2 = \frac{2}{3}(x^2 + x)$.

C. HOLLINS.

Treatment of materials consisting of or comprising cellulose derivatives. [Preservation of lustre of acetate silk.] BRIT. CELANESE, LTD. (B.P. 282,722, 23.11.27. U.S., 30.12.26).—Addition of 0.1–30% of a thiocyanate (ammonium, sodium, potassium, calcium) to the bath prevents or diminishes the delustering of acetate silk by boiling liquids.

C. HOLLINS.

Manufacture of cellulose ethers. O. LEUCHS and E. DÖRR, Assrs. to I. G. FARBERIND. A.-G. (U.S.P. 1,694,127, 4.12.28. Appl., 31.10.27. Ger., 8.11.26).—Alkali-cellulose and ethyl chloride are heated in a closed vessel containing a perforated vessel charged with solid sodium hydroxide; the latter absorbs the water of reaction, dissolves gradually, and falls into the reaction mixture.

C. HOLLINS.

Moth-proofing substance. L. E. JACKSON and H. E. WASSELL, Assrs. to MUNDATECHNICAL PRODUCTS Co. (U.S.P. 1,694,219, 4.12.28. Appl., 18.11.27. Cf. B.P. 263,092; B., 1928, 227).—Quinoidine, the mixture of amorphous alkaloids remaining in the acid extract of cinchona bark after removal of crystalline alkaloids, is used for moth-proofing.

C. HOLLINS.

Purifying dry-cleaning solvent. A. E. HATFIELD, Assr. to ACHILLE SERRE, LTD. (U.S.P. 1,704,604, 5.3.29. Appl., 30.9.26. U.K., 31.12.25).—See B.P. 266,850; B., 1927, 579.

Manufacture of cellulose ethers. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 306,857, 21.10.27).—See U.S.P. 1,694,127, preceding.

Manufacture of compound yarns or threads. O. RASCH (B.P. 306,007, 10.10.27).

Apparatus for drying webs of woven material. RUDOLPH & KÜTNE GES.M.B.H. (B.P. 288,175, 26.3.28. Ger., 2.4.27).

Pumps for viscose or other liquids. I. FRIEDMANN and N. SCHOPP (A. FRIEDMANN and G. NEMETZ (B.P. 307,225, 22.3.28).

Nitrocellulose solutions etc. (B.P. 307,085 and 307,528). **Pigmented products** (B.P. 277,949, 277,989, and 307,516).—See XIII. **Tanning materials** (G.P. 451,913).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications of ozone [for bleaching]. A. VOSMAER (Chem. Weekblad, 1929, 26, 184–186).—An account of the use of ozone for bleaching beeswax, linseed and soya-bean oils, mineral oils, cotton, paper, sugar, and other organic products.

S. I. LEVY.

Bleaching of wood pulp. BAIRD and DOUGHTY.—See V.

PATENTS.

Increasing wetting and penetrating capacity of liquids for treatment of textiles and leather. H. T. BÖHME A.-G. (B.P. 291,070, 20.9.27. Ger., 27.5.27).—Mixtures (or salts) of pyridine bases with alkylated aromatic sulphonic acids are used as wetting-out agents. Either or both ingredients may be hydrogenated.

C. HOLLINS.

Treatment of textiles. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 306,067, 15.11.27).—Materials consisting either wholly of natural silk or of mixtures of natural silk with cellulose acetate are degummed by heating with an alkaline liquor containing alkali hydroxides, borates, silicates, phosphates, phenolates, cresolates, soap, or a sulphonated soap or oil, the p_H value of which is maintained at 10–10.5 throughout the treatment; if the temperature is raised above 80° the cellulose acetate may be wholly or partially delusted.

F. R. ENNOS.

Dyeing of animal fibres. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 306,632, 5.12.27).—Deep black shades on wool are obtained by oxidising on the fibre a sulphonated p -phenylenediamine or NN' -di-(p -aminophenyl)- p -phenylenediamine, in which one or both amino-groups carry a naphthyl residue, or a sulphonated naphthylenediamine in which one or both amino-groups carry a p -aminophenyl residue. Examples are: 4'-amino-2' (or 3')-sulphophenyl- β -naphthylamine; p -aminophenyl-Cleve acid; 1:4-di-(6-sulpho- β -naphthylamino)benzene; 4-amino-4'- α - and - β -naphthylaminodiphenylamine-2-sulphonic acids; 4- β -naphthylamino-4'-hydroxydiphenylamine-2-sulphonic acid; 1:4-di-(p - α - and - β -naphthylamino- o -sulphoanilino)benzenes; 1:5-di-(4'- p -toluidino-3'-sulphoanilino)naphthalene, $C_{10}H_6[NH \cdot C_6H_3(SO_3H) \cdot NH \cdot C_6H_4Me]_2$; 2:6-di-(4'-amino-2'-sulphoanilino)naphthalene.

C. HOLLINS.

Production of fast dyeings and printings. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 306,637, 6.12.27. Addn. to B.P. 306,632, preceding).—Wool, silk, acetate silk, and mixtures of these are dyed in level shades by oxidising on the fibre a salt of a non-sulphonated p -aminoanilino-derivative of naphthalene. Examples are: 2:7-di-(p -aminoanilino)naphthalene (bluish-grey to blue-black); 2- p -aminoanilino-7-naphthol (blue); p -aminophenyl- β -naphthylamine (blue-black); p -aminophenyl- α -naphthylamine (corinth); 2-amino-7- p -aminoanilidonaphthalene (blue to blue-grey).

C. HOLLINS.

Colouring of cellulose derivatives. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 306,981, 28.11.27).—Acetate silk etc. is dyed with dehydrothioluidine either diazotised and coupled in substance or on the fibre with suitable non-sulphonated coupling components (phenol, β -naphthol, 2:3-hydroxynaphthoic acid, dimethylaniline, β -naphthylamines, 2:3-aminonaphthoic acid, 1:2-aminonaphthol, ethyl ether, etc.), or condensed with a chloronitrobenzene, especially 1-chloro-2:4-dinitrobenzene (yellow). C. HOLLINS.

Dyeing of fibres consisting of cellulose mono- or di-acetate. G. B. ELLIS. FROM CHEM. WORKS FORMERLY SANDOZ (B.P. 306,877, 27.10.27).—Cellulose which has been mono- or di-acetylated but retains the original fibrous structure is dyed with the usual acetate silk dyes, excluding basic di- and tri-phenylmethane, azine, thiazine, and oxazine dyes. C. HOLLINS.

Dyeing of vat, sulphur, and mineral khaki colours on fabrics. J. WAKEFIELD (B.P. 307,238, 23.3.28).—The scoured and bleached fabric is batched through water containing a little alkali and hyposulphite (for vat dyes) or sodium sulphide (for sulphide dyes), and dyed in a closed kier, from which air has been displaced by steam, by forcing into the kier a fully reduced vat solution or a sulphide dye bath or a mineral bath. The dye solution is then removed, and the colour is developed with an oxidising solution (in the case of vat dyes) in the kier. Level shades are produced. C. HOLLINS.

Treatment of materials made of or containing cellulose derivatives. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 306,611, 28.11.27 and 26.6.28. Addn. to B.P. 302,775; B., 1929, 169).—Acetate silk etc. is treated with sodium or potassium zincate of such alkalinity as to produce partial hydrolysis; absorption is assisted by addition of swelling agents. The treated material has affinity for basic, acid, mordant, direct, and vat dyes, and is particularly suited for production of discharge effects. The tin-treated material of the prior patent shares the last-mentioned property. C. HOLLINS.

Alkaline treatment [mercerisation] of natural and artificial cellulosic materials. IMPERIAL CHEM. INDUSTRIES, LTD., and E. CHAPMAN (B.P. 307,239, 14.4.28).—As a wetting-out agent for use with mercerising liquors or other strongly alkaline media, a mixture of cresol (or other homologous phenol) with 1—15% of benzyl alcohol, butyl alcohols, amyl alcohols, or the synthetic alcohols, b.p. 120—200°, obtained by hydrogenation of carbon oxides is found effective. C. HOLLINS.

Production of metallised fabrics. O. TREBITSCH (B.P. 285,921, 24.2.28).—Fibrous materials are treated with a metallic salt solution and the metal is precipitated thereon by means of a suitable reducing agent. After dissolving out any compounds not completely reduced by means of a suitable acid solution, the material is subjected to mechanical treatment until the precipitated metal is consolidated to a tenacious coating. F. R. ENNOS.

Production of fast tints on vegetable fibre. F. STRAUB, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P.

1,704,637, 5.3.29. Appl., 23.5.27. Switz., 27.5.26).—See B.P. 271,897; B., 1929, 14.

Washing, bleaching, and like machines. J. L. RUSHTON, J. LEVER, and H. HILL (B.P. 307,113, 7.12.27).

Bleaching and dyeing plants. G. P. GASS, and JACKSON & BRO., LTD. (B.P. 307,632, 16.3.28).

Covers for low-pressure kiers. W. and J. B. SADLER (B.P. 307,621, 2.3.28).

Printing patterns on plush fabrics etc. W. C. FAIRWEATHER. FROM PLÜSCHWEBEREI GREFRATH A.-G. (B.P. 308,123, 14.6.28).

Decorating the surface of textile materials etc. F. A. H. HEYNERT, and BRIT. BEAD PRINTERS, LTD. (B.P. 307,241, 19.4.28).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Kinetics of the contact sulphuric acid process, and modern views on adsorption. M. BODENSTEIN (Z. physikal. Chem., 1929, B, 2, 345—348).—Fink and Bodenstein's interpretation of their experimental data for the rate of combination of sulphur dioxide and oxygen on the surface of platinum (B., 1908, 120) requires modification to make it accord with modern views on adsorption, for it is no longer possible to accept the view that a layer of trioxide many molecules thick is adsorbed on the surface of the catalyst. If, however, there is a unimolecular surface layer of trioxide, and if combination occurs only at certain active points or lines, the measured reaction velocity will still be the rate of diffusion of the reactants to the seat of reaction, but this will be a diffusion which is not perpendicular to the surface, but along it, and is hindered by the two-dimensional motion of the adsorbed trioxide molecules. With this alteration, the explanation previously advanced still holds good. R. CUTHILL.

Action of calcium carbonate and of dolomite on zinc sulphate solutions. L. CAMBI, G. BOZZA, and D. MASPERI (Giorn. Chim. Ind. Appl., 1929, 11, 3—8).—In the electrolytic production of zinc, calcium carbonate is almost universally employed to neutralise the electrolyte, precipitate the iron, and flocculate the silica. Results are given of determinations of the velocity of reaction of calcium carbonate (mostly marble) and of dolomite, ground to various degrees of fineness, on 2*N*-zinc sulphate solution at 40.2° and 100°. The values of the velocity coefficients of the reaction at these two temperatures are 0.000282 and 0.00124 for calcite and 0.0000884 and 0.000636 for dolomite; for calcite the amounts of zinc rendered insoluble at these temperatures are approximately proportional to these numbers, whereas with dolomite the amount is relatively much greater at the higher temperature. The results indicate that the layer of insoluble matter which forms on the calcium or magnesium particles is only slightly permeable to the zinc sulphate solution, and that diffusion virtually ceases when a very thin layer has been deposited. The precipitate formed contains very little calcium or magnesium, and consists mostly of zinc hydroxide, together with small proportions of zinc sulphate and carbonate. T. H. POPE.

Determination of [bismuth in] bismuth hydroxyiodide. G. BÜMMING and K. FERREIN (Arch. Pharm., 1929, 267, 142—143).—In the determination of bismuth, the hydroxyiodide, which may be brown in colour, must be boiled with twenty times its weight of nitric acid until no more iodine vapours are evolved; the mixture is then evaporated and ignited and the residue of Bi_2O_3 weighed. S. COFFEY.

Extraction of iodine from Black Sea algæ. S. KOMARECKYJ (Chem. Listy, 1929, 23, 5—7).—The yield of iodine from algæ is 24—41% less when air is freely admitted during combustion than when the supply is restricted. A mixture of various Black Sea algæ contained 0.0859—0.172% of iodine, or 0.138—0.338% of the ash remaining after combustion. The corresponding figures for *Cystosira* are 0.095 and 0.146% of iodine, and for *Phyllophora* 0.645 and 1.01%.

R. TRUSZKOWSKI.

Absorption of oxygen by dilute alkaline solutions of pyrogallol. T. J. DRAKELEY and H. NICOL (J.S.C.I., 1929, 48, 62—64; cf. B., 1925, 944).—All alkaline solutions of pyrogallol evolve carbon monoxide when used as an absorbent for oxygen of high purity. For constant concentration of alkali the percentage of carbon monoxide increases as the amount of pyrogallol diminishes, but no stoichiometrical relation was found. Agitation of the solution during absorption of the oxygen is an important factor in minimising the evolution of carbon monoxide.

Applications of ozone. VOSMAER.—See VI.

PATENTS.

Preparation of alkyl compounds of lead. C. A. KRAUS and C. C. CALLIS, ASSTS. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,694,268, 4.12.28. Appl., 13.4.23).—A lead-sodium alloy containing 20% Na is treated with ethyl iodide, and water equivalent to the sodium is added at such a rate as to keep the mixture boiling. Lead tetraethyl is then distilled off in steam.

C. HOLLINS.

Production of finely-divided sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,498, 20.2.28. Addn. to B.P. 177,103; B., 1922, 373 A).—A solution of ammonium polysulphide containing a protective colloid, e.g., cellulose pitch, flowing by gravity along the walls of a rotating pipe, is treated with a current of steam flowing in the same direction or in counter-current.

W. G. CAREY.

Preparation of hydrogen. H. BOMKE, ASSEE. of BERGWERKSVERBAND ZUR VERWERTUNG VON SCHUTZRECHTEN DER KOHLENTÉCHNIK G.M.B.H. (B.P. 279,128, 17.10.27. Ger., 16.10.26).—Magnesium oxide in conjunction with substances absorbing carbon dioxide is used as the catalyst for preparing hydrogen from carbon monoxide, or water-gas, and water vapour. Calcined magnesian lime may be employed, or uncalcined magnesian lime, the carbon dioxide being first set free in the process.

W. G. CAREY.

Acetic acid (B.P. 296,974 and 301,415).—See III. **Treatment of pyrites** (B.P. 306,691).—See X. **Electrolytic cell** (G.P. 452,922).—See XI. **Potassium chloride from molasses** (B.P. 304,071).—See XVII.

VIII.—GLASS; CERAMICS.

Physico-chemical phenomena exhibited by minor constituents in glass. W. E. S. TURNER (J.S.C.I., 1929, 48, 65—67 r).—Glass melted at 1400° from mixtures containing moisture, or in tank furnaces the gases of which contain water vapour, retains 0.02—0.08% of water. Arsenious oxide is tenaciously retained in glass melted at 1400°; moreover, except in the presence of large excess of reducing agents, much of it is converted into arsenic oxide, and only at 1500° does this appear slowly to be dissociated into arsenious oxide and oxygen. Ferric oxide is the more stable form in soda-lime-silica glass at 1400—1500°. In solution in glass, melted electrically in a neutral atmosphere, for concentrations between 0.005 and 1.0% Fe_2O_3 , the actual ferric oxide in the glasses was found to be 92—94% of the total iron oxide. Under reducing conditions, with sodium tartrate as reducing agent in the mixture, the maximum proportion of ferrous to total iron oxide obtainable was 89%. Glass containing ferrous oxide has a greater transmission in the ultra-violet than when the iron is present as ferric; for a concentration of 0.1% of iron oxide, the difference in the transmission between the ferrous and ferric oxide glasses is about 100 Å. Irradiation by sunlight or electric arc converts the ferrous into ferric oxide. When the glass so irradiated is heated at 600—700° the effect is reversed. (Cf. B., 1929, 245.)

Determination of the source and means of prevention of stones in glass. H. INSLEY (J. Amer. Ceram. Soc., 1929, 12, 143—152).—Distinction is made according to their source between devitrification, batch, and refractory "stones." The former may consist of tridymite, wollastonite, diopside, jeffersonite, and $\text{Na}_2\text{O} \cdot 3\text{CaO} \cdot 6\text{SiO}_2$. Photomicrographs show the crystalline forms characteristic of each type. "Cordy" devitrification is due to imperfect mixing of the batch or entrainment of the scum. Random distribution of devitrification stones is avoided either by alteration of the cooling schedule or of batch composition. Batch stones are caused by the incomplete dissolution of one of the batch constituents (usually sand or clay) and can be avoided by (1) finer grinding of batch, (2) more intimate mixing of batch, (3) higher melting temperature, and (4) longer melting time. Microscopical examination distinguishes two types of refractory stones: (a) those formed by the action of alkaline vapours on the refractory above the glass level and usually composed of large corundum crystals, and (b) those formed below the level of the glass and composed of smaller corundum crystals associated with nephelite and mullite. Some stones show only partial attack by the glass (quartz grains remaining), and are probably from the cooler parts of the system. Corundum stones dissolve very slowly and are therefore most objectionable.

J. A. SUGDEN.

Physical structure of refractory materials. T. S. CURTIS (J. Amer. Ceram. Soc., 1928, 11, 904—916).—The relationship of the grog to the matrix of aluminosilicate refractories is investigated, using photomicrographs in natural colour (cf. B., 1928, 693). The products under consideration are divided into three categories, viz., (1) refractory bodies with large developments of mullite, such as firebricks for boiler settings

and glass tank blocks for the slag line; (2) refractory clay wares, in which mullite is relatively unimportant, such as saggars; (3) vitrified products, such as stoneware, sanitary ware, and whiteware, in which mullite is an incidental inclusion in fused felspathic material. In making products of a high refractoriness-under-load value, grog containing 52% Al_2O_3 with less than 5% of basic oxides, and completely inverted to mullite and glass, is bonded with clay of a similar composition and fired to cone 17. For the best results the grog should be fired to cone 30–32, the conditions being alternately reducing and strongly oxidising. The addition of 10–20% of an inert mineral fibre improves the rigidity, strength, and toughness of a low-fire refractory and a vitreous body. Careful control of the grain size and the amount of non-plastic additions to vitreous bodies is essential.

A. T. GREEN.

Effect of heat treatment on diatomaceous earth.

H. M. KRANER (J. Amer. Ceram. Soc., 1928, 11, 875–883).—Samples of various deposits of diatomaceous earth were fired to temperatures of 1100°, 1260°, and 1300°, and held at these temperatures for 2 hrs. It is shown that the diatom structure may be destroyed without any indication of the deformation of the material. Further, there is a direct relationship between the maintenance of the diatom structure and the purity of a diatomaceous earth. Thus the silica content of such an earth gives an indication of its resistance to the influences of high temperatures. A pure sample shows little deterioration at 1300°.

A. T. GREEN.

Effect of thermal shock on the transverse strength of fireclay brick. C. W. PARMELEE and A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1928, 11, 884–895).—A description of an apparatus designed to give data concerning the effect of repeated thermal shock on the transverse strength of fireclay brick is given. After heating the fireclay brick to a uniform temperature of 1100°, its central portion was cooled by a blast of air at room temperature, the remaining surface being protected by insulating material. The results indicated that stiff-mud bricks were more affected than dry-pressed bricks. Plastic clay products showed deterioration. Brands having a high transverse strength suffered a comparatively large decrease in strength, whilst porous bricks were less affected.

A. T. GREEN.

Correction of an extreme case of cracking in the drying of brick.

H. FRÉCHETTE and J. G. PHILLIPS (J. Amer. Ceram. Soc., 1929, 12, 153–161).—Attempts to manufacture stiff-mud brick from a very plastic, low-permeability clay resulted in high drying breakage even when drying was very slow. Laboratory tests showed that this could be completely overcome by preheating the clay to 400–500° (heating above 500° makes the clay too short to work) or by treating the clay with aluminium, ferric, or sodium chloride together with the addition of grog. The temperature range of the preheating treatment is too narrow for rotary kiln operation. Plant-scale tests led to the adoption of chemical treatment of the clay using 1% of ferric chloride, 0.5% of sodium chloride, and 10–15% of grog. The colour of the brick was also improved and a slight tendency to scumming was overcome.

J. A. SUGDEN.

Physical tests for vitreous enamels. C. J. KINZIE (J. Amer. Ceram. Soc., 1929, 12, 188–192).—Methods of test are described for resistance to thermal shock, impact, and abrasion. In the first of these water is allowed to drip on to the surface, which is heated to 360°; the resulting cracks are shown up by rubbing with lamp-black. In the impact test a steel ball is allowed to fall repeatedly on the surface from a fixed height; the extent of injury is judged by the loss in weight or by visual examination. It is noted that an enamel with a high resistance to impact usually has a high resistance to cross-bending. The abrasion test consists in coating the inside of a metal cup with the enamel to be tested, partially filling with the abrasive charge, and rotating in a suitable clamp on the end of a horizontal shaft at 63 r.p.m. for 41 hrs. The abrasive charge is made up of water, cleansing and washing powders, and $\frac{1}{4}$ -in. porcelain balls, and it half fills the cup. The extent of abrasion is measured by the loss in weight sustained by the cup. The results show that there is little or no connexion between resistance to abrasion and other properties of enamels.

J. A. SUGDEN.

Vitreous enamel and its defects. A. MALINOVSKY (J. Amer. Ceram. Soc., 1929, 12, 180–187).—Literature dealing with the defects in enamelled cast-iron ware is reviewed. The characteristics of ground coat, enamel, and metal surface are discussed, and the various defects due to the metal surface are illustrated by photographs.

J. A. SUGDEN.

PATENTS.

Manufacture of glass. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 307,563, 23.12.27).—Batch materials, particularly such as will give a glass of low thermal expansion, preferably water-free, are finely ground and mixed, then pressed into small rods, and sintered together at or above 900°. The rods are melted and worked by holding directly in the flame without the use of a furnace. Alternatively, sintering may be dispensed with by adding a suitable bonding agent before pressing.

A. COUSEN.

Metal-glass union. MANHATTAN ELECTRICAL SUPPLY Co., Assees. of W. F. HENDRY (B.P. 293,292, 9.12.27. U.S., 2.7.27).—To obtain a seal between glass and metal, the glass contains as an ingredient either the same metal or one of its salts, such glass being used alone or joined to a body of ordinary glass.

A. COUSEN.

Production of enamel. F. C. R. MARKS. From LEICHTMETALL-VERWERTUNGS-GES.M.B.H. (B.P. 307,259, 15.6.28).—The major portion of an enamel for iron articles consists of lepidolite, or chemically similar material, natural or synthetic, which is ground to extreme fineness and floated simply with water upon the objects. A ground-coat enamel is dispensed with. Substances such as silica, aluminite, lime, or magnesia may be added to vary the coefficient of expansion, the m.p., or the viscosity. The enamel is fired on at 1000°.

F. SALT.

Production of a coloured coating similar to ceramic enamels. USINES DE LA BASSE-MEUSE, Soc. ANON., and O. RUPPEL (B.P. 306,615, 26.11.27).—A cement enamel suitable for application in the cold

state to cement etc. consists of 1—3 pts. of Portland cement mixed with 1 pt. of slag cement, together with 10—15% of colouring matter and 0.1—0.5% of an organic acid such as valeric acid. Articles coated with the enamel are treated in drying rooms or stoves with moist air containing up to 5% CO₂. The setting period can be shortened by treating the articles, after they have been in the stoves for 2 days, in baths containing 100 pts. of water, 8—10 pts. of a carbonate soluble in water, and 1—2 pts. of a soluble silicate. F. SALT.

Opacifying substance for adding to enamelling compositions. I. KREIDL (B.P. 283,130, 17.11.27. Austr., 4.1.27).—A powerful opacifying agent for enamels consists of a synthetically prepared alkali or alkaline-earth silico-aluminate, in which the alkali or alkaline-earth oxide is present in the proportion of 1 mol. to 2 mols. of silica. In an example, kaolin is introduced into a boiling solution of caustic soda and the product is dried. F. SALT.

Production of non-splinterable or safety glass. J. E. THORNTON (B.P. 308,117 and 308,119, 25.5.28 and 29.5.28).

Detergents etc. (B.P. 307,141).—See XII.

IX.—BUILDING MATERIALS.

Fire-resistance of hollow load-bearing wall tile. S. H. INGBERG and H. D. FOSTER (Bur. Stand. J. Res., 1929, 2, 1—334).

Fusibility of mixtures of ferric oxide and lime. JACQUÉ.—See X.

PATENTS.

Method and apparatus for burning lime and natural cement. E. C. R. MARKS. From LOUISVILLE CEMENT Co. (B.P. 306,856, 21.10.27).—Raw material for calcination is moved by suitable means (gravity etc.) through a preheating zone, in which it is exposed to hot gases the temperature of which is increasing instead of decreasing, thence through a combustion zone, in which the dried and preheated material is rapidly raised to calcination temperature, and finally through a roasting zone. In the combustion and roasting zones the products of combustion and the material move in the same direction; in the preheating zone the reverse is the case. The hot gases from the exit end of the roasting zone are passed to the exit end of the preheater. Apparatus is described, consisting of a rotary kiln and a preheating chamber. F. SALT.

Preventing evaporation from concrete during curing. H. P. HAYDEN, Assr. to BARBER ASPHALT Co. (U.S.P. 1,684,671, 18.9.28. Appl., 12.10.25).—An adherent film, impervious to water, is applied to the concrete before curing. F. G. CLARKE.

Treatment of artificial stone, cement, etc. W. MAGUIRE (B.P. 307,132, 16.12.27).—A weathered or aged appearance is imparted to artificial stone, cement, etc. by applying a layer of soluble material to the surface during the moulding process. The soluble substance, e.g., magnesium sulphate, is subsequently dissolved out, leaving a pitted surface on the stone. The pitted surface may be coloured and then scraped or brushed to produce special effects. F. SALT.

Impregnation of wood with substances toxic to animal and bacterial life and fungus growths. C. B. LIPMAN and A. GORDON, Assrs. to UNIV. OF CALIFORNIA (U.S.P. 1,693,930, 4.12.28. Appl., 9.9.26).—Copper arsenate or other metal salt solution is injected (from a reservoir with a head of about 20 ft.) into a hole bored transversely three quarters through the trunk of the living tree. This is followed, preferably, by an injection of glucose solution to ensure reduction. Two to three days after the injection the tree is felled, and the wood is found to be uniformly impregnated. C. HOLLINS.

Handling, mixing, and pouring concrete. II. P. PARIS (B.P. 308,006, 9.1.28).

Concrete and like mixers. J. F. ROBB (B.P. 308,007, 9.1.28).

[Making pressure-tight joints for] drying, impregnating, and similar treatment of timber. C. GOODALL (B.P. 308,102, 24.4.28).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Eutectic cast iron. A. MITINSKY (Rev. Mét., 1929, 26, 84—87).—The properties of cast iron depend on the relative proportions and distribution of metal and graphite. The mechanical resistance to external stresses is a maximum when the graphite is distributed regularly in a non-laminated form. Cast iron with excellent mechanical properties can be obtained (1) by diminishing the total carbon content to less than 3%, the desired amount of graphitisation being produced by hammering or by raising the silicon content; (2) by decreasing the silicon content, graphitisation being obtained by pouring into a pre-heated mould. When the graphite is uniformly distributed and is non-laminated, the presence of ferrite increases the ductility, shock-resistance, and machinability. Solidification of the eutectic cast iron gives the most uniform distribution of all the elements and also leads to the formation of the best graphite, and, as its m.p. is a minimum, it is more easily heat-treated than are other cast irons. The pure eutectic iron-carbon contains 4.3% C, but this is decreased by the presence of silicon and phosphorus. It is possible to calculate approximately the carbon content of the eutectic of a commercial cast iron of known composition. M. E. NOTTAGE.

Heat-treatment and volume changes of grey cast iron between 15° and 600°. J. W. DONALDSON (Rev. Mét., 1929, 26, 78—84).—Four series of cast irons with varying content of one of the elements, carbon, manganese, phosphorus, or silicon, were used. The heat treatments were carried out at 450° and at 550° for periods of 5 days of 8 hrs. each, the specimens being allowed to cool overnight. As a consequence of the decomposition of the combined carbon, the tensile strength and the hardness were affected to an extent depending on the temperature, duration of heating, and composition of the cast iron, equilibrium being reached after 200 hrs. The stability of the combined carbon, and consequently the resistance to heating, diminished with increase in carbon content from 2.71 to 3.51%; increase of manganese content from 0.52 to 2.43%.

and of phosphorus from 0.20 to 1.06% increased the resistance. The cast iron was stable when less than 1.10% Si was present, the stability increasing as the silicon content decreased to 0.65%; the critical content of silicon for the decomposition of combined carbon lies between 1.10 and 1.17%. In the heat treatments at 550° expansion during the first heating is due to the decomposition of the combined carbon; the penetration of air along the laminae and consequent oxidation in their neighbourhood produces a secondary expansion. When the total carbon content is low the expansion is small; increase in the manganese content is accompanied by an increase in the expansion, whilst increase in phosphorus decreases the expansion; 1.1% of silicon and less produces small expansion, which becomes more marked and is accompanied by oxidation of the iron when it increases from 1.2 to 1.5% and becomes very pronounced in cast irons containing 1.5–2.2%.

M. E. NOTTAGE.

Influence of heat treatment below the A1 point on the properties of technical iron. W. KÖSTER (Arch. Eisenhüttenw., 1928–9, 2, 503–522; Stahl u. Eisen, 1929, 49, 357–358).—The solid solubility of cementite in α -iron increases with rising temperature between 600° and the A1 point, so that steels with less than 0.9% C age-harden after quenching from above 600°, the maximum effect being obtained with 0.09–0.1% C aged at room temperature. In this case the yield point increases by 60%, the tensile strength by 55%, and the hardness by 65%, whereas the elongation decreases by 50% and the reduction in area by 10%. The decomposition of the supersaturated solid solution begins to be appreciable in 1 hr. at 50°, and is completed at 200–250°; the precipitated carbide particles first separate as black points throughout the grains, but with rising temperature they migrate to the grain boundaries. The coercivity increases slightly with increase of quenching temperature below A1, but the remanence undergoes a marked decrease; on ageing or annealing the quenched steel the values for these properties return more or less rapidly to the normal. A. R. POWELL.

Useful and deleterious action of gases in steel. F. RAPATZ (Z. Metallk., 1929, 21, 89–92).—The brittleness produced in a steel by hardening at too high a temperature is due to the presence of oxygen; a thoroughly deoxidised steel shows no signs of brittleness even after quenching from a temperature well above the normal. Oxygen causes also the formation of white spots in the fracture of hardened steel. Nitrogen in small quantities has little effect on the properties of steel, but in conjunction with sufficient carbon it tends to increase the hardness of surfaces. The effect of gas inclusions on the crystal structure is briefly discussed and previous work on the subject reviewed.

A. R. POWELL.

Determination of gases in metals, particularly of oxygen in steel. W. HESSENERBRUCH (Rev. Mét., 1929, 26, 93–114).—The influence of elements other than iron on the results obtained is reviewed. Metallic elements such as manganese and aluminium, the oxides of which have less tendency to dissociate than ferrous oxide, will reduce it and so retard the reduction by

carbon. At 1200° the formation of water vapour has not been proved; sulphides of carbon and hydrogen are formed in small quantities, but influence the result only when the sulphur content is greater than 0.05%. The formation of sulphur dioxide has not been detected, whilst ammonia, methane, and phosphine cannot exist at high temperatures and low pressures. By adding fluxes which lower the m.p. of iron, the extraction of the dissolved gases is rendered complete, but not the reduction of oxides at 1200°; hence an apparatus which can be used at a higher temperature is necessary. A high-frequency furnace suitable for the purpose is described. Owing to surface oxidation, the sample should be in the form of a single lump. The analysis can be made in 20–40 min., the total content of oxygen, hydrogen, and nitrogen being determined in one operation.

M. E. NOTTAGE.

Recovery of austenitic steels. A. MICHEL and P. BENAZET (Compt. rend., 1929, 188, 912–915).—Special steels which may be rendered austenitic by tempering at high temperatures fall into two classes, according to their behaviour on recovery:—(1) There is a sudden dilatation accompanied by the liberation of heat at a temperature which depends on the composition of the steel and the rate of heating and corresponds with the $\gamma \rightarrow \alpha$ transformation. (2) Above a certain temperature there is a gradual negative anomaly in the dilatation, which is more accentuated when the rate of heating is rapid. It is shown that the two types of recovery are analogous, and that the difference is due only to the different rates of reaction, the passive resistance being too feeble in the first type to inhibit the $\gamma \rightarrow \alpha$ transformation during the heating process.

J. GRANT.

Microscopic composition and structure of basic slag after different heat treatments and their relation to the citric acid solubility. H. SCHNEIDER-HÖHN (Stahl u. Eisen, 1929, 49, 345–352).—Microscopical examination of the structure of basic slag with and without addition of silica after various annealing and quenching treatments has shown that these do not affect the nature of the different constituents or their relative proportions; hence it appears that the constituents of basic slag are in equilibrium with one another at all temperatures. The following constituents have been identified: silicocarnotite ($5\text{CaO} \cdot \text{P}_2\text{O}_5 \cdot \text{SiO}_2$), crystallised lime, magnesia, ferrous oxide, ferric oxide, and alumina, and compounds and solid solutions of these oxides. In slag to which no silica has been added hilgenstockite ($4\text{CaO} \cdot \text{P}_2\text{O}_5$) is also found. The lower citric acid solubility of the phosphoric acid in the last-named type of slag is attributed to the depressing effect of soluble lime salts on the solubility of silicocarnotite. The rapid disintegration of quenched basic slags without added silica is attributed to hydration and carbonation of the lime crystals. The usual methods of determining free lime in basic slags are shown to be untrustworthy.

A. R. POWELL.

Fusibility of mixtures of ferric oxide and lime. L. JACQUÉ (Compt. rend., 1929, 188, 917–919).—Mixtures of lime (5–60%) and ferric oxide are readily fused by rapid heating below 1500°, the greatest fusi-

bility being at 1220° for 20–30% of lime. The ferric oxide is partly decomposed, particularly in a non-oxidising atmosphere, with the formation of magnetite.

J. GRANT.

[Determination of copper.] H. F. BRADLEY (Chemist-Analyst, 1928, 17, No. 4, 14).—Low's short iodide method is modified. The ore (0.5 g.) is moistened with water, a little potassium chlorate being added if sulphides are present, and decomposed with 5 c.c. of concentrated nitric acid and a few drops of hydrochloric acid. Digestion is continued for 6 min.; if brown fumes appear, a few drops of a mixture of hydrochloric acid and hydrogen peroxide are added. Then concentrated sulphuric acid (7 c.c.) is added, and the mixture boiled until it fumes freely. After cooling, 3 c.c. of water followed (cautiously) by 133 c.c. of concentrated ammonia-solution are added. To this slightly acid solution a concentrated solution of ammonium acetate is added in excess (5 c.c.) of the amount necessary to produce a red colour. After cooling, 0.5 g. of sodium fluoride is added, and the liquid stirred until the red colour disappears; 3 g. of potassium iodide are added, and the titration is performed in the usual way.

CHEMICAL ABSTRACTS.

Brass for screw-cutting. P. DEJEAN (Rev. Mét., 1929, 26, 45–67).—This alloy is hot-rolled, annealed, and cold-drawn, with or without intermediate annealings. The method of manufacture fixes its composition within narrow limits. Hot-rolling of a brass containing more than 61.5–62% Cu is difficult, whilst cold-drawing of one containing less than 57.5–58% Cu may produce cracks either during the process or long afterwards; hence the copper content should be between 58.5 and 61%. The narrowness of the permissible zone of composition is due to the fact that the α -constituent is malleable when cold but cracks when hot, whilst the β -constituent is plastic when hot but only slightly malleable when cold. The plasticity decreases with increase in copper content for temperatures above 500°, and this effect becomes more marked the higher is the temperature. All brasses undergo a transformation at about 475°; this change starts at a little above 300° and is probably connected with the β -constituent, which increases the plasticity above 480° and decreases it below this temperature. The increase in strength and elastic limit and the decrease in elongation with cold-hardening are the more rapid the greater is the copper content. The strength and the elastic limit decrease, whilst the elongation increases, with higher temperatures, the rate of decrease reaching a maximum between 300° and 400° and becoming very slight between 550° and 800°. The orientation due to rolling and drawing has only commenced to disappear at 600° for brass containing 61.4% Cu, whilst it has completely disappeared with 56.8% Cu; in the former case grain growth has not commenced at 700°, whilst at 850° it is very noticeable; in the latter case extensive grain growth takes place at 800°, and this produces a sudden decrease in the elongation when the annealing temperature is above 700°. The best annealing temperature is 500°, and it should not be raised above 700°. The addition of 1–2% Pb is necessary to prevent the alloy from machining badly.

M. E. NOTTAGE.

Nickel-chromium [plating] process. W. PFANHAUSER (Chem.-Ztg., 1929, 53, 207–208).—A chromium plating which will entirely protect iron, steel, or brass from corrosion can be obtained only by 4–6 hrs.' treatment in the bath. As this is quite uneconomical for technical work, the article is usually plated with nickel first, but, to obtain adherent coatings with no tendency to peeling, it is essential that the nickel layer be thick enough and relatively free from hydrogen, and that the surface to be plated be completely free from grease and oxide. With a nickel coating 0.02–0.025 mm. in thickness a sufficiently protective chromium coating is obtained by electrolysis for 10–15 min. in the usual plating solution.

A. R. POWELL.

Age-hardening of standard silver. M. HAAS and D. UNO (Z. Metallk., 1929, 21, 94–96).—Dilatometric and micrographic examination of standard silver after quenching from 720–800° and ageing above 200° has confirmed Norbury's work (B., 1928, 267). Decomposition of the supersaturated solid solution becomes visible in the microstructure at 180°, and at 360° agglomeration of the precipitated particles is rapid. The maximum hardness value is obtained when the grain boundaries are surrounded by numerous precipitated particles of copper; this first occurs at about 290°.

A. R. POWELL.

Recovery of platinum from dunite rock at Onverwacht, Transvaal. T. K. PRENTICE and R. MURDOCH (J. Chem., Met., Min. Soc. S. Afr., 1929, 29, 157–166).—The dunite ore of the Onverwacht mine contains platinum in association with chromite, hornblende, diopside, and hortonolite. By stage-crushing and concentration on Wilfley, curvilinear, and corduroy tables an extraction of 83% of the platinum metals in a concentrate weighing 1–2% of the ore and assaying about 30 oz./ton is readily obtained. Of this recovery about 65% is in the form of a product which may be dressed by hand-panning and acid treatment to obtain a concentrate assaying about 82% of platinum metals. The remainder is in a concentrate which is rich in chromite and metallic iron from the machinery; this product is agitated in barrels with zinc amalgam and an acid solution of copper sulphate whereby the platinum mineral is taken up by the amalgam leaving a residue of chromite etc. The discharge from the barrels passes over bateas, amalgamated plates, and curvilinear tables and the recovered amalgam is cleaned, pressed, and retorted. The residue in the retorts is crushed, cleaned with acid, panned to remove slimes, and cleaned from iron by magnetic separation. All the by-products from the cleaning and the concentrates from the curvilinear tables are returned to the barrel to be re-treated, the amalgam being worked up as before. More than 97% of the platinum in the original concentrate is recovered by this amalgamation process.

A. R. POWELL.

Corrosion. XIV. Protection against corrosion of iron in steam boilers. A. THIEL and H. LUCKMANN (Korrosion u. Metallschutz, 1928, 4, 169–177; Chem. Zentr., 1928, ii, 1610).—Experiments on the production of protective coatings of ferrous oxide with (a) water and dilute sodium hydroxide solution at 100° in nitrogen or

air, (b) water at 100° and air, (c) sodium chloride or sulphate solutions at 100° in absence of air, (d) sodium hydroxide and various salts at 200° are recorded.

A. A. ELDRIDGE.

Method of testing opacity of protective [metal] coatings against corrosion. J. Cournot (Rev. Mét., 1929, 26, 76—77).—Filter paper impregnated with ferricyanide and an alkali chloride is soaked in water and placed on the cleaned surface. The pores are indicated by transparent spots which are blue in colour where the base metal is a ferrous alloy and brown where it consists of copper or its alloys. The paper is then washed with water to remove excess of ferricyanide. In the case of ferrous alloys which have been plated with chromium or nickel over copper, if the holes pass through both layers blue spots are formed, the alkali ferrocyanide masking the coloration due to the copper, whilst if they pass only through the copper, brown spots are formed. The method is not so good for deposits of zinc and cadmium, as the ferrocyanides of these metals mask to some extent the blue or brown colorations. The method can also be used for testing the opacity of thin sheets of tin or aluminium used for wrapping food products, the sheet being placed on a polished steel plate and the ferricyanide paper on top; the formation of blue spots indicates the presence of holes.

M. E. NOTTAGE.

Electric annealing furnaces for metals. M. TAMA (Z. Metallk., 1929, 21, 77—86).—An illustrated description of various types of electric muffles for annealing metal rods, tubes, and bands.

A. R. POWELL.

Penetration of hydrogen into metal cathodes and its effect on the tensile properties of metals and their resistance to repeated stresses. Effect of non-electrolytic baths and nickel plating on these properties. F. C. LEA (Proc. Roy. Soc., 1929, A 123, 171—185).—Static tensile tests, single-blow impact tests, and repeated-stress experiments in torsion and under direct stress were carried out on various metal cathodes in acid and alkaline baths, both when current was and was not flowing. The results indicate that nascent hydrogen liberated at a cathode penetrates the crystal boundaries of mild steel (0.14% C), nickel, and a rustless steel containing about 15% Cr and 10% Ni, but the penetration does not influence the tensile strength or the resistance to repeated stress. It does, however, diminish the elongation of specimens of mild steel very considerably, although it does not lower the resistance of mild steel specimens to the Izod impact test. The effect of polishing mild steel is to prevent, or modify the action of, hydrogen penetration between the crystals. Acid corrosion reduces the resistance to torsional stress, but a caustic soda solution, with or without the presence of hydrogen, protects the specimen and the fatigue range is raised. It is considered that the presence of hydrogen at the crystal boundaries affects the continuity of slip and causes a crack to occur as in quenched and tempered materials, and that in all such materials the failure is at the boundaries, whilst the failure of plastic materials occurs by cracking within the crystal. Some tests were made on nickel-plated specimens, and the results are compared with those for mild steel and drawn nickel bar. Under direct repeated

stress the effect of the deposited surface is to lower the fatigue range very considerably and to make the results erratic. A tool mark, having a depth equal to the thickness of the nickel deposit and sharp at the bottom, lowers the fatigue range in bending by about the same amount as the nickel plating, and it is concluded that the nickel is deposited under stress or acts as a discontinuity like the tool mark, or else that a crack is first produced in the deposited nickel and causes the lowering of the repeated-stress range.

L. L. BIRCUMSHAW.

Action of calcium carbonate and dolomite on zinc sulphate solutions. CAMBI and others.—See VII.

PATENTS.

Induction [melting] furnace. E. F. RUSS (B.P. 305,388, 2.3.28).—In an induction furnace for melting copper, bronze, etc., the melting channel, opening at different heights into the melting chamber, ascends throughout its course and surrounds the upper half of the primary coil.

J. S. G. THOMAS.

Reduction of iron ores or oxides. J. W. HORNSEY (B.P. 306,561, 18.8.27).—The ore is fed into a rotary preheating cylindrical furnace in which it is heated by combustion of the gases from the reducing cylinder, then into the reducing cylinder where it is mixed with a solid fuel and reduced to iron sponge. Fuel and air are introduced into the discharge end of the reducing cylinder by means of a water-jacketed conveyor in such a way that the air does not come into contact with the material being reduced.

A. R. POWELL.

Roasting and reduction of metallic [iron] ores. F. L. DUFFIELD (B.P. 281,338, 28.11.27. U.S., 29.11.26).—The roasting and reduction of iron ores is carried out simultaneously in two rotating furnaces each of which is alternately used as the roasting and as the reducing furnace. Reduction is effected in one furnace by means of a current of reducing gas, and the waste gases from this furnace are burnt with air in the roasting furnace to supply the necessary heat.

A. R. POWELL.

Hardening iron and steel articles. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 291,423, 18.1.27. Ger., 4.6.27).—Iron or steel articles are carburised in molten cyanide mixtures without addition of finely-divided carbonaceous material. The cyanide mixture is heated to 920° or higher, avoiding frothing, and the surface of the melt may be protected by a layer of graphite.

C. A. KING.

Production of castings of cast iron mixed with steel. J. ROBINSON (B.P. 296,080, 16.7.28. India, 25.8.27).—A molten mixture of cast iron and steel is mixed without loss of heat by immersing an aluminothermic mixture in the metal contained in a ladle or other container.

C. A. KING.

Manufacture of steel. H. J. VAN ROYEN (B.P. 285,814, 16.2.28. Ger., 21.2.27).—Steel is rendered non-susceptible to cold deformation and ageing by previously deforming it as extensively as possible as soon as the metal has cooled below the Ar₁ point, after which it is normalised.

C. A. KING.

Continuous [steel] annealing and cleaning process. H. M. NAUGLE and A. J. TOWNSEND, Assrs. to

COLUMBIA STEEL Co. (U.S.P. 1,704,015, 5.3.29. Appl., 1.7.25).—Sheet steel is heated quickly and uniformly to annealing temperature, which is maintained only to obtain correct microstructure, at which point the steel is cooled rapidly. C. A. KING.

Reduction process and apparatus. T. F. BAILY (U.S.P. 1,704,029, 5.3.29. Appl., 6.3.26).—The charge is fed to a shaft furnace by means of an aerating pump, and gases from the reaction chamber are conveyed to a combustion chamber surrounding the shaft. C. A. KING.

Treatment of pyrites. S. I. LEVY and G. W. GRAY (B.P. 306,691, 10.2.28).—Pyrites or pyrrhotite is heated in chlorine so as to volatilise sulphur and form ferrous chloride and chlorides of any non-ferrous metals present. The ferrous chloride formed is then heated in pure dry air whereby it is decomposed into ferric oxide and ferric chloride; the latter volatilises and is passed over further quantities of pyrites to effect chlorination as before. Both processes may be carried out in the same furnace, partial chlorination being effected by means of chlorine at 400—600° followed by oxidation with dry air at 600—1100°. The low-temperature process results in the retention of zinc, copper, and other chlorides by the ferric oxide residue, whereas in the high-temperature operation these chlorides are volatilised and may be separated from the sulphur by fractional condensation. A. R. POWELL.

Roasting of zinc sulphide ores. S. ROBSON (B.P. 306,569, 24.10.27).—Zinc sulphide ore or concentrate is mixed with 20—25% of its weight of crushed sinter from a previous process and the mixture is roasted in an apparatus so designed that a current of air is drawn through the heated mass in such a way that only sufficient air is supplied to effect the roasting and a porous sintered mass of oxide is produced together with a gas having a high content of sulphur dioxide. A. R. POWELL.

Reduction of ores, oxides, etc. [of volatile metals, e.g., zinc]. H. E. COLEY (B.P. 306,425, 14.9.27).—In the process in which volatile metals are reduced from their ores by spraying liquid hydrocarbons on to the heated mass, the vapours issuing from the furnace are passed upwards through a water-jacketed tower down which a spray of water from a series of fine jets is allowed to fall. The sludge from the tower is filter-pressed and re-distilled with carbonaceous material to recover the metal. A. R. POWELL.

Treatment of ores etc. containing platinum. S. C. SMITH (B.P. 306,566, 21.10.27).—Ores containing platinum metals in association with copper, nickel, and iron sulphides are smelted to obtain a matte which is ground finely and roasted, with or without previous bessemerisation. The roasted product is leached with sulphuric acid to remove the greater part of the copper and the residue is washed, dried, and reduced to metal by heating it with carbonaceous material or in a stream of reducing gas. The metal sponge so obtained is digested with sulphuric acid, which dissolves nickel and iron. The residue from this treatment may be roasted again and leached to remove the remaining copper and the reduction process repeated. The final product con-

tains all the platinum metals in a form ready for refining by the ordinary methods. A. R. POWELL.

Metallic [nickel-tungsten] alloy. H. L. COLES and J. G. DONALDSON, ASSRS. to GUARDIAN METALS Co. (U.S.P. 1,702,765, 19.2.29. Appl., 13.6.22).—The alloy contains 14% Ni, 2—5% C, and not more than 86% W. A. R. POWELL.

Composite metal plate. H. L. COLES and J. G. DONALDSON, ASSRS. to GUARDIAN METALS Co. (U.S.P. 1,702,766, 19.2.29. Appl., 4.2.26).—The plate comprises a core of an alloy containing 10% Ni, 14% Cu, and more than 70% W inside a casing of a metal having a high heat conductivity, the whole being heat-treated to effect superficial alloying between the outside of the core and the inside of the casing. A. R. POWELL.

Manufacture of steel products. R. A. HADFIELD (U.S.P. 1,704,385, 5.3.29. Appl., 7.7.26. U.K., 29.4.26).—See B.P. 273,855; B., 1927, 658.

Sintering zinc ores. H. J. STEHLI (B.P. 307,595, 7.2.28).—See U.S.P. 1,661,813; B., 1928, 337.

Conveyer furnaces [for heat-treating metals etc.]. BRAYSHAW FURNACES & TOOLS, LTD., and S. N. BRAYSHAW (B.P. 307,963, 15.12.27).

Furnaces [for heating tubes etc.]. "ILVA" ALTI FORNI E ACCIAIERIE, and A. AURELI (B.P. 307,853, 12.12.27).

Electrolytically depositing metal on tubes. J. STONE & Co., C. J. LYTH, and F. J. PIKE (B.P. 307,726, 12.12.27).

Metal-glass union (B.P. 293,292).—See VIII. **Attaching rubber etc. to metal** (B.P. 307,180 and 307,628).—See XIV.

XI.—ELECTROTECHNICS.

Electrical conductivity of thin oil films. I. General nature of the phenomenon. H. E. WATSON and A. S. MENON (Proc. Roy. Soc., 1929, A, 123, 185—202).—Static friction measurements have been made by means of an apparatus somewhat similar to that used by Hardy (cf. *ibid.*, 1925, A, 108, 1). With "wiped films" the result obtained by Hardy, that the coefficient of friction μ decreases considerably with increase of pressure, was confirmed. Microscopical examination and measurements of the thickness of these non-conducting films suggested that they consist of a number of small oil globules between two adsorbed films of air or water vapour on the metal surfaces. There is no relation between the value of μ and the thickness of the film. Air films have a high insulation resistance, and when they break down the resistance usually becomes very low. "Flooded films" of paraffin oil will withstand a high potential gradient (over 100,000 volts/cm.) as long as their thickness is greater than about 15 μ . Thinner films (below 11 μ) break down frequently on application of 2—40 volts and become partially conducting. The breakdown thickness does not vary much with the voltage. The film becomes conducting in two stages: in the first the resistance is of the order of 1 megohm, in the second it is very low. With both films the *E.M.F.* increases with the current for small values of the latter, but Ohm's law is not obeyed; with

larger currents the *E.M.F.* becomes nearly constant, and on still further increase it decreases.

L. L. BIRCUMSHAW.

Electric annealing furnaces. TAMA. Nickel-chromium plating. PFANHAUSER.—See X.

PATENTS.

[Terminals for] electric heating devices. BRIT. THOMSON-HOUSTON CO., LTD., H. W. H. WARREN, and R. G. GREEN (B.P. 305,160, 11.9.28).—A paste of finely-powdered graphite and, if desired, carborundum is pressed round the ends of the heating unit and then treated so as to harden it; *e.g.*, the paste made up with a bonding agent such as acacia gum is pressed round the unit and heated at 2000° while embedded in coke and sand.

J. S. G. THOMAS.

Electrolysis in pressure-resisting vessels. W. VOGT (G.P. 453,275, 13.3.25).—Water or other electrolyte is electrolysed under such a pressure that one of the gases produced is denser than the surrounding liquid; *e.g.*, oxygen at 100° and 1100 atm. is denser than the electrolyte. The electrodes employed consist of conical shells having points on the upper surfaces and joined together with vertical sheet-metal strips.

J. S. G. THOMAS.

[Bi-polar] electrolytic cell. R. EDGEWORTH-JOHNSTONE (B.P. 307,093, 4.11.27).—Closely-positioned electrodes in the form of sheets or plates, normally insoluble and upon which metals are electrolytically deposited, are held together in pack formation and provided with perforations for passage of electrolyte and escape of gases.

J. S. G. THOMAS.

Electrolytic cell, especially for electrolysis of alkali chlorides. SIEMENS & HALSKE A.-G. (G.P. 452,922, 22.10.25).—The electrolyte vessel is closed by a glass cover which absorbs chemically active radiation, and current is supplied by conductors passing through the cover and reinforced immediately below the cover.

J. S. G. THOMAS.

Device for controlling electrolytic operations. J. PORZEL (U.S.P. 1,700,178, 29.1.29. Appl., 1.9.23).—The sides and ends of a frame of insulating material extend beyond the faces of the anode or cathode, which is enclosed and supported within the frame.

J. S. G. THOMAS.

Colloidal diaphragms for electrolytic cells. G. F. JAUBERT (B.P. 281,674, 30.11.27. Fr., 1.12.26).—A diaphragm, permeable to electrolyte but impermeable to gaseous products of electrolysis, is prepared by depositing a hydrogel of a colloidal organic or inorganic substance upon a porous diaphragm, *e.g.*, wire gauze. Thus the gauze may be immersed successively in aqueous solutions of magnesium sulphate and sodium silicate. Suitable hydrogels comprise magnesium or iron silicate, kaolins, clays, magnesium or aluminium phosphate, graphite, india-rubber, etc.

J. S. G. THOMAS.

[Electrolyte for Leclanché type] galvanic cell. MANNESMANN LICHT A.-G. (F.P. 626,678, 23.12.26. Ger., 13.4.26.).—Potash end-liquor either alone or mixed with other salt solutions is used, and the efficiency of the electrolyte is increased, if desired, by addition of mercury salts.

J. S. G. THOMAS.

Electrolyte for accumulators. W. M. JOWETT (Austral. P. 1755, 8.5.26).—Magnesium, aluminium, potassium, and ammonium sulphates and glycerin are added to sulphuric acid of *d* 1.27. J. S. G. THOMAS.

Negative electrode for zinc electric accumulators. A. POUCHAIN (B.P. 303,823, 10.5.28. Ger., 10.1.28).—The surface of a support of a metal which is a better conductor than zinc is covered with zinc only where zinc would be deposited during charging of the accumulator, the rest of the surface being covered with insulating material, *e.g.*, india-rubber or cellulose. The electrode is then coated with a solution of gum and sodium silicate.

J. S. G. THOMAS.

Alkaline [electric] accumulator. F. JIRSA and B. KRAUPNER (G.P. 453,129, 18.6.25).—The accumulator comprises an iron electrode and an anode composed of the higher oxides of silver, produced by immersing a silver electrode in an alkaline electrolyte containing telluric hydroxide or its derivatives.

J. S. G. THOMAS.

Manufacture of photo-electric cells. GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 306,996, 1.12.27).—A photo-electric cell having a greatly increased sensitivity to red light comprises a cathode composed of a surface, *e.g.*, a silvered portion of the wall of the bulb, covered with a very thin invisible layer of photo-sensitive material, *e.g.*, potassium, which has been sensitised after its formation, by an electric discharge in hydrogen. Preferably the thin layer is formed by heating a thick visible layer.

J. S. G. THOMAS.

Electron emitter. E. E. SCHUMACHER, ASSR. to WESTERN ELECTRIC CO. (U.S.P. 1,700,454, 29.1.29. Appl., 8.7.24).—A metallic sleeve fitted to a core of refractory metal is coated with an alkaline-earth oxide, the metal of the sleeve being such that its oxide, when heated in air, reacts with the alkaline-earth oxide to form compounds which, when heated *in vacuo*, decompose to form alkaline-earth oxide.

J. S. G. THOMAS.

Production of moulded articles suitable as electric insulating material. SCHIEFERWERKE AUSDauer A.-G. (B.P. 286,732, 9.3.28. Ger., 10.3.27).—Filling materials are cold-pressed at very high pressures with about one sixth of their weight of a viscous artificial resin obtained as described in B.P. 286,731 (B., 1929, 294). Subsequent hardening is effected by heating at about 80° between porous stone slabs.

S. S. WOOLF.

[Impregnating paper] insulating material for electrical condensers. J. E. G. LAHOUSSE (F.P. 629,408, 29.4.26).—Paper is impregnated with the isomerisation products of frankincense or colophony, *e.g.*, abietic acid or pimaric acid. The m.p. is reduced by using a mixture of abietic acid with its sodium salt, wax, or paraffin.

J. S. G. THOMAS.

Galvanic cell. A. SCHMID (B.P. 281,698, 1.12.27. Ger., 2.12.26).

Lead electrode for electric accumulators. A. POUCHAIN (B.P. 303,824, 10.5.28. Ger., 10.1.28).

Pasting battery plates. F. B. DEHN. From VESTA BATTERY CORP. (B.P. 307,618, 27.2.28).

Filaments for electric incandescence lamps.

FALK STADELMANN & Co., Assees. of NEUE GLÜHLAMPEN GES.M.B.H. (B.P. 301,014, 24.7.28. Ger., 23.11.27. Addn. to B.P. 297,052).

X-Ray tubes [with metallic envelope]. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 307,377, 5.12.27).

Induction furnace (B.P. 305,388).—See X. **Deposits of organic substances for dispersion (B.P. 307,585).**—See XIV.

XII.—FATS; OILS; WAXES.

Extraction of orujo [olive press-residue] with carbon disulphide. J. BOHLE (Chem.-Ztg., 1929, 53, 165—167, 187—189).—The preparation of orujo before extraction is described. After grinding and sifting (10 mm. mesh) the orujo, which contains on the average 10—12% of oil and 25% of moisture, should be dried to a water content not exceeding 16%; careful operation with rotating drums heated by direct fire is satisfactory, but investigation is suggested of methods of drying by exhaust steam under vacuum; drying by hot air involves serious mechanical difficulties, and steam-drying (without vacuum) is too expensive. The employment of trichloroethylene as solvent is discussed; the oil obtained is not of such good quality as sulphur olive oil, being poor in colour and not amenable to bleaching. Such oil cannot be used for the production of white soap, contains a greater proportion of non-oleaginous material (colouring matter, waxes, etc.), and solidifies in conditions under which sulphur oil remains liquid. Attention is drawn to the considerable fire risks attending the use of carbon disulphide in the large extractors (capacity 17,000—18,000 litres) in current use, especially when discharging spent pulp: the risk of explosion is increased when very wet orujo is used, as balling-up may occur, causing the solvent to be retained in pockets after the steaming-off. The usual type of plant consists of extractors coupled in pairs; a description is given of an improved lay-out of a battery of extractors designed to give a maximum throughput with minimum risk of explosion. E. LEWKOWITSCH.

Detection of coconut and palm-kernel oils by testing for lauric acid. J. GROSSFELD and A. MIERMEISTER (Z. Unters. Lebensm., 1928, 56, 423—437).—The method of identifying lauric acid in alcoholic liquors (B., 1929, 145) has been applied to the examination of fats for coconut and palm-kernel oils as follows:—(A) 1—100 mg. of the fat are saponified with 2.5 c.c. of 0.5*N*-alcoholic potash solution and the alcohol is removed by evaporation. The residue is dissolved in 2 c.c. of water mixed with 2 c.c. of glycerin solution (300 g. per litre), heated for 5 min. on the boiling water-bath, 2.0 c.c. of magnesium sulphate solution (150 g. of the crystallised salt per litre) are added, and the hot solution is filtered clear. A cloudiness immediately on cooling or a white flocculent precipitate after some time indicates the presence of 10% or more of lauric acid (20% of coconut oil) in the fat. (B) If test A is negative, 1.0 g. of fat is saponified by heating with 0.4 c.c. of 50% aqueous potash solution and 2 c.c. of glycerin; 200 c.c. of water and 50 c.c. of glycerin solution (300 g. per litre) are added to the soap solution, which is then

heated to boiling and 10 c.c. of magnesium sulphate solution as above are added. It is heated for 10 sec. more, filtered clear, and cooled. A flocculent precipitate indicates 2.5% or more of lauric acid. (C) If test B is negative the filtrate is acidified with 5 c.c. of dilute hydrochloric acid and shaken with 60 c.c. of ether. The ether extract is evaporated to dryness, the residue dissolved in 2.5 c.c. of alcoholic 0.5*N*-potash solution and re-tested as in test A. A flocculent precipitate indicates 0.5% or more of lauric acid (1% or more of coconut oil). Myristic acid and higher fatty acids give negative results. Decoic acid gives a negative result when less than 4 mg. are present, and nonoic acid when less than 30 mg. are present. Lauric acid was found in small quantities in butter fat, hardened arachis oil, hardened and unhardened cotton-seed oil. It was not found in cacao butter, soya-bean oil, or hardened whale oil. Of rancid samples two cotton-seed oils and an oleostearin contained no lauric acid, but one of hardened arachis oil and another of hardened cotton-seed oil gave positive tests. A self-extracted arachis oil gave a negative result, but after oxidation of the fatty acids with potassium permanganate a faintly positive test was obtained. A quantitative separation of lauric acid from the higher fatty acids by distillation and precipitation of the latter by magnesium sulphate was not found possible. W. J. BOYD.

Oil of [seeds of] *Pistacia lentiscus*. F. L. VODRET (Annali Chim. Appl., 1929, 19, 76—84).—This optically inactive oil, obtained in 12.4% (in some years 14%) yield from seeds of plants grown in Sardinia, has: d_{20}^{25} 0.9182, sp. viscosity at 22° 10.65, n_D^{22} 1.4671, butyrefractometer index 61.8, solidification point —6°, Tortelli thermo-value 42.0, solubility in alcohol at 95° 5.5:100, deep yellow fluorescence in Wood's light, acid value 28.05, saponif. value 209, acetyl acid value 21.09, acetyl saponif. value 228.6, Reichert-Meissl value 0.96, Hehner value 91.9, iodine value 81.57, absolute iodine value 100.56. The percentage composition is approx.: palmitic acid 24.82, stearic acid 11.63, oleic acid 47.62, linoleic acid 6.25, glycerol 9.53, unsaponifiable substances (phytosterol, resins, etc.) 0.96, linusic and isolinusic acids traces. From the phytosterol an acetate, m.p. 119—121°, was prepared. Tannin is detectable and, under Livache's conditions, 100 pts. of the oil absorb 0.15 pt. of oxygen in 48 hrs. A number of colour reactions of the oil are given, and the changes in characters produced by boiling are described (cf. Sernagiotto and Vita, B., 1915, 1061). T. H. POPE.

Sulphonated oils. I. Preparation of pure sodium ricinoleosulphate and its isolation from commercial sulphonated oils. K. NISHIZAWA and K. WINOKURI (Chem. Umschau, 1929, 36, 79—81).—Ricinoleosulphuric acid was prepared by treating pure ricinoleic acid dissolved in anhydrous ether with chlorosulphonic acid (cf. Grün and Woldenberg, A., 1909, i, 284); the hydrochloric acid and ether may be removed in the vacuum desiccator, but better yields of a purer product were obtained by salting out the aqueous solution of the ester with hydrochloric acid and extracting impurities with ether. The ester was then neutralised with alcoholic caustic soda and the sodium salt obtained

in a state of purity after repeated crystallisation from alcohol. The composition agrees with the formula $\text{SO}_4\text{Na} \cdot \text{C}_{17}\text{H}_{32} \cdot \text{CO}_2\text{Na} \cdot \text{H}_2\text{O}$. It is readily soluble in water, and almost insoluble in organic solvents; it is sparingly soluble in cold 95% alcohol, but the solubility increases rapidly with decreasing concentration or rising temperature. By similar methods ricinoleo-sulphuric acid and its sodium salt were isolated from three commercial sulphonated oils.

E. LEWKOWITSCH.

Determination of organically combined sulphuric acid in sulphonated oils. C. RIESS (Chem. Umschau, 1929, 36, 77—79, and Collegium, 1928, 657—661).—The organic sulphate in a sulphonated oleic acid and in a commercial Turkey-red oil was determined by Herbig's gravimetric method and by the more convenient volumetric method (American official method, except that 5—6 g. of oil were used and refluxed for $\frac{1}{2}$ hr.); the results from both methods agreed within practical limits, whether hydrochloric or sulphuric acid was used for the hydrolysis (volumetric). Traces of sulphate could be found in the oil separated from the Turkey-red oil, after boiling with acid, by extraction with ether and washing with dilute brine; the amount retained was slightly greater when sulphuric acid was employed for the hydrolysis (cf. B., 1928, 646).

E. LEWKOWITSCH.

Determination of iodine values. II. Action of iodine chloride on fatty acids with conjugated double linkings. E. T. GELBER and J. BÖESEKEN (Rec. trav. chim., 1929, 48, 377—385).—The addition of iodine chloride (Wijs' solution) to linoleic acid proceeds in two stages. The first addition is rapid, whereas the second is slow and only tends to go to completion after a long time and with a large excess of iodine chloride (cf. B., 1927, 427). Free iodine is produced at the beginning of the reaction. Ingle's theory that the iodine results from the hydrolysis of the iodochloride is probably incorrect, since free iodine is obtained even when the determination is carried out in water-free media. Under these conditions saturation of both double linkings occurs. Determination of the iodine value of linoleic acid by Marshall's solution (J.S.C.I., 1900, 19, 231) gives correct values. No free mineral acid is produced, and any possible substitution is thus avoided. When elæostearic acid is acted on by Wijs' solution two of the three double linkings are saturated rapidly, the third only after several days, a large excess of iodine chloride being essential. The production of free iodine is explained by assuming that the first stage of the addition of iodine chloride to a conjugated doubly unsaturated higher fatty acid is a 1:4-addition of chlorine. This is a rapid reaction, and it is followed by the saturation of the remaining double linking with iodine chloride. When a carboxyl group is adjacent to a double linking as in sorbic acid, no free iodine is produced. Elæostearic acid combines with four atoms of chlorine to give a tetrachloro-oleic acid (first stage), which is then saturated with iodine chloride.

H. BURTON.

Detergent action of soap. F. H. RHODES and S. W. BRAINARD (Ind. Eng. Chem., 1929, 21, 60—68).—For the determination of the detergent power, a piece of

cotton cloth is soiled by immersion in a mixture of lamp-black, lubricating oil, and tallow in carbon tetrachloride, freed from the excess of liquid, dried at 80° for 1 hr., and allowed to age for 11 hrs. The cloth is then sewn up in the form of a bag which is filled with glass beads, and is placed in a rubber-covered cage inside a glass cylinder which holds the soap solution; after heating the latter to the desired temperature, washing is carried out by rotating the cylinder. The brightness of the soiled cloth is measured before and after washing by means of an integrating photometer whereby indices of detergent power for the various soap solutions are calculated. For short periods of washing (30 min.) the detergent effect of distilled water is about one half that of a 0.25% solution of soap, but on increasing the period to 5 hrs. the cleansing action of the former increases whilst that of the latter diminishes, probably owing to the increased degree of dispersion of the dirt. The detergent effect for each washing reaches a maximum after a certain time, viz., $7\frac{1}{2}$ min. with the conditions obtaining in these tests. Increasing the concentration of soap above 0.25% produces little change in the detergent effect. Although solutions containing 0.05 and 0.1% of soap remove about the same amount of dirt in 5 washings as the more concentrated solutions, the rate of removal in the first few washings is slightly lower; 0.01% soap solutions are distinctly less efficient. Soap solutions 24 hrs. old show about the same ultimate efficiency as those freshly prepared, although less dirt is removed in the early stages. Variations in temperature between 20° and 60° have little effect on the detergent power.

F. R. ENNOS.

Viscosity of soap solutions. B. WALKER (Allgem. Oel- Fett- Ztg., 1928, 25, 448—450; Chem. Zentr., 1928, ii, 1633).—The viscosity of 0.3% (of fatty acid) soap solutions at 20° was 52—56.2 sec. (water at 20°, 50.8 sec.). Generally, potassium soaps gave higher values than sodium soaps, tallow and lard excepted. Values for 30°, 40°, and 50° are recorded.

A. A. ELDRIDGE.

Determination of iodine value [of fats]. J. J. A. WIJS (Z. Unters. Lebensm., 1928, 56, 488—490).—See B., 1929, 179.

Applications of ozone. VOSMAER.—See VI. **Determination of fat in malted milk.** BALLARD.—**Glycerides in butter.** DE'CONNO and SCOPINARO.—See XIX.

PATENTS.

Detergent, cleansing, and polishing compositions. BRITISH DYESTUFFS CORP., LTD., J. BADDILEY, and E. CHAPMAN (B.P. 307,141, 23.12.27).—Pastes or aqueous solutions of complex sulphonic acids or their salts ("wetting-out" agents, e.g., sulphonated mineral oils) with the addition of inorganic acid detergents (e.g., nitre-cake) are claimed.

E. LEWKOWITSCH.

Oil presses. J. JAKOBSEN (B.P. 307,770, 12.12.27).

Manufacture of dry, non-caking, readily-soluble soap in the form of threads. A. WELTER (B.P. 307,549, 14.11.27).

Butter etc. (B.P. 307,167).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Effect of light on paints containing lead. G. ZEIDLER and W. TOELDT (Farben-Ztg., 1929, 34, 1547—1549).—The nature and extent of colour changes occurring when iron plates coated with a range of paints containing lead pigments were suspended in an atmosphere of hydrogen sulphide for 15 min. are detailed. The panels were next exposed to each of three sources of artificial light and the relative bleaching action was observed. The results indicate the unreliability of fading and weathering tests carried out with the mercury-vapour lamp as source of artificial light. Although rapid in action, the latter does not reproduce the effect of the sun's rays, and more significant results are to be obtained with the slower-working "Nitalamp."

S. S. WOOLF.

New raw materials for the [cellulose] lacquer industry. A. NOLL (Farben-Ztg., 1929, 34, 1486—1490, 1549—1552).—The properties and uses of a number of modern solvents, diluents, plasticisers, and synthetic resins are detailed.

S. S. WOOLF.

"Gum-running." E. PYHÄLÄ (Farben-Ztg., 1929, 34, 1552—1553).—A new process for "gum-running" is described wherein hard copal resins are melted in the presence of white oil (liquid paraffin) which is subsequently distilled off and recovered. The gum kettle, which is heated by means of a bath of molten zinc, is closed by a cover fitted with suitable outlet and condensing apparatus etc. The gum swells in the white oil, which functions as a protective agent, taking up and removing from the system oily ingredients liberated from the melting gum. The losses in gum running are reduced and a much lighter product is obtained. It is considered that uniform varnishes can be prepared by mixing, in the cold, solutions at varnish consistency of "tempered" copal, ester gum, and prepared tung and linseed oils, in suitable varnish solvents.

S. S. WOOLF.

Finnish turpentine. VII. isodiprene, a new terpene of the sylvestrene group. O. ASCHAN (Bidr. K. Finlands Natur. Folk, 1926, H. 80, No. 6, 18 pp.).—Of Finnish turpentine, about 60% has b.p. 160—170°. The fraction of b.p. 163—167° contains an unidentified terpene, pinonene. The fraction of b.p. 167—170° contains isodiprene, d_4^{20} 0.8561, n_D^{20} 1.47536, R_{44-84} (monohydrochloride, d_4^{20} 6.9775, n_D^{20} 1.48175, R 50.57). Elimination of hydrogen chloride affords sylvestrene. When distilled with acetic anhydride and concentrated sulphuric acid the terpene gives a characteristic reddish-violet colour. Terpenes of the sylvestrene group are classified as follows:—(1) those which give an intense blue colour with acetic anhydride and sulphuric acid, (2) *m*-menthadienes which give a violet colour, and take up only 1 mol. of hydrochloric acid without difficulty, (3) dicyclic terpenes containing the same double ring as carone. CHEMICAL ABSTRACTS.

Ash content of podophyllum resin. L. D. HAVENHILL (J. Amer. Pharm. Assoc., 1929, 18, 129—130).—Eight commercial samples of the resin gave 0.18—0.89% of ash and 5 others gave 0.65—0.89%; the comparative densities of the samples ranged from

0.6 to 1.8. Experiments with different precipitants indicated that alum water offers no advantage over dilute hydrochloric acid, and that yellow resins are produced by alum water. The author is of the opinion that the maximum ash content should be fixed at 0.5%, and the raising of the ash limit to 1.5% in the U.S.P. VIII is not justifiable.

E. H. SHARPLES.

Disposal of wastes. MOHLMAN and BECK.—See XXIII.

PATENTS.

Printing ink. A. R. TRIST (B.P. 307,535, 9.12.27 and 9.10.28).—A printing ink for use with printing plates having mercurised non-printing areas consists of an intimate mixture of partly polymerised linseed oil containing about 7½% of fatty acids, pigments, and mercury, with or without the addition of an aqueous dispersing agent for the oil, e.g., an aqueous solution containing 2½—5% of borax and 5—10% of shellac.

S. S. WOOLF.

Manufacture of pigments and pigmented products. E. C. DE STUBNER (B.P. 277,949, 30.8.27. U.S., 23.9.26).—A pigment is precipitated in an aqueous medium and the precipitate, after preliminary washing, is dehydrated by washing with a non-aqueous water-miscible liquid compatible with the product to be pigmented, until the water has become replaced by this liquid. For application to lacquers the pigment can be precipitated on a soluble cellulose derivative of fibrous cellular structure, e.g., cellulose nitrate, and 86—95% ethyl alcohol used as dehydrating agent. For application to paints, printers' inks, etc. the precipitated pigment is dehydrated with acetone, pyridine, etc.

S. S. WOOLF.

Manufacture of coloured or pigmented products having a cellulosic base. E. C. DE STUBNER (B.P. 277,989, 22.9.27. U.S., 7.7.27, and 307,516, 30.8.27. Cf. preceding abstract).—Pigmented soluble cellulose derivatives are prepared by precipitating a preformed pigment, e.g., ultramarine blue, zinc sulphide, etc., from a colloidal dispersion on to a soluble cellulose derivative. In a preferred process the pigment is electrically of the same sign as the cellulose derivative to be pigmented, and a substance of opposite sign is first deposited in the latter, thus promoting deposition of the pigment. A pigmented soluble cellulose derivative suitable for use in lacquers, printing inks, etc. is obtained by precipitating a pigment, e.g., lead chromate, or depositing a metal, e.g., copper, silver, antimony, gold, on a cellulose derivative of cellular structure.

S. S. WOOLF.

Lacquers or varnishes. BRIT. THOMSON-HOUSTON CO., LTD., H. W. H. WARREN, and R. NEWBOUND (B.P. 306,914, 31.8.27).—A "glyptal" resin is plasticised by the addition of oleic acid, butyl phthalate, tolyl phosphate, etc. and dissolved in a mixture of solvents with graded b.p., with or without the addition of colouring matter or pigment. After application, the lacquer so obtained is baked for ½ hr. at 100° and subsequently for ¾ hr. at 200°.

S. S. WOOLF.

Nitrocellulose products [lacquers etc.]. W. J. JENKINS, and IMPERIAL CHEMICAL INDUSTRIES, LTD. (B.P.

307,528, 10.11.27).—The use of derivatives of β -hydroxypropionic acid in nitrocellulose lacquers is claimed.

S. S. WOOLF.

Nitrocellulose solutions and coating compositions. W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 307,085, 26.8.27 and 26.6.28).—Monoalkyl (C_1 — C_5) ethers of butylene glycols, especially of isobutylene glycol, are used as solvents for nitrocellulose, particularly for coating lacquers and enamels.

C. HOLLINS.

Manufacture of synthetic resins and of varnishes therefrom. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 306,501, 22.11.27).—Crude solvent naphtha free from bases is treated with ferric chloride in the presence of about 10% of phenolic substances. Iron is removed by dilution with benzene, xylene, etc., and after removal of the precipitate the solution is treated with quicklime and fuller's earth and filtered. On evaporation of the solvent, preferably *in vacuo*, pale resins are obtained which are soluble in drying and stand oils, and hence suitable for use in varnishes.

S. S. WOOLF.

Manufacture of coloured varnishes. SOC. CHEM. IND. IN BASLE (B.P. 285,058, 7.2.28. Switz., 10.2.27).—Nitrocellulose or phenol-aldehyde varnishes are coloured with non-sulphonated monoazo dyes of the type: nitrated or halogenated arylamine \rightarrow arylamine. Examples are: *p*-nitroaniline \rightarrow cresidine (scarlet), aniline (orange), *m*-aminoacetanilide (yellow), diphenylamine; 2:5-dichloroaniline \rightarrow aniline (yellow), *m*-toluidine (yellow); *m*-nitroaniline \rightarrow aniline (yellow); 4-nitro-*o*-anisidine \rightarrow dimethylaniline (yellow). The shades are fast to light.

C. HOLLINS.

Cellulose nitrate solution. M. B. HOPKINS and H. E. BUC, Assrs. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,694,097, 4.12.28. Appl., 3.4.22).—Nitrated non-benzenoid hydrocarbons (petrol, C_6 — C_{12}) are used with alcohols and benzene to dissolve cellulose nitrate.

C. HOLLINS.

Impregnating solution. L. V. REDMAN and H. C. CHEETIAM, Assrs. to BAKELITE CORP. (U.S.P. 1,693,939, 4.12.28. Appl., 24.4.23. Renewed 23.2.28).—Phenol-aldehyde resins for impregnating purposes are dissolved in furfuraldehyde together with hexamethylenetetramine as hardening agent; the ammonia evolved reacts with the furfuraldehyde.

C. HOLLINS.

[Spray]-lacquering process [to form drops]. ELEKTRIZITÄTSWERK LONZA (B.P. 297,711, 12.1.28. Switz., 25.9.27).

Condensation products of dimethylolurea etc. (B.P. 306,875).—See III. Moulded articles (B.P. 286,732).—See XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation phenomena in *Hevea* latex. VII. Phenomena in alkaline latex. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 34; Arch. Rubbercultuur, 1928, 12, 454—471).—The power of coalse to coalesce the floccules in *B*-mixture (B., 1925, 17) is destroyed not only by heat, quinosol, or acidifying to the second stable zone, but also by the

addition of alkali such as ammonia or sodium hydroxide. Acidification shortly after the addition of the alkali restores the coalescing activity only in part. The lower limit of concentration for killing coalse is 0.15*N*-ammonia or 0.015*N*-sodium hydroxide, the p_H approximating to 8 with both. Sodium acetate does not destroy coalescing power as it is unable to attain this degree of alkalinity. On account of this effect of alkali, ammonia-preserved latex has no coalescing power.

D. F. TWISS.

Coagulation phenomena in *Hevea* latex. VIII. Rubber obtained by freezing latex. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 37; Arch. Rubbercultuur, 1928, 12, 675—685).—Coagulation by freezing occurred only at temperatures considerably below 0°, several days being required for complete effect even at -15° . The coagulum was yellower than the normal; the viscosity, hardness, and rate of vulcanisation of the rubber were somewhat greater than the standard, whilst the nitrogen content, 0.31%, although a little below the normal, was considerably higher than for rubber from washed cream obtained by centrifuging. The serum had p_H 6.2 and possessed coalescing activity.

D. F. TWISS.

Preserving [rubber] latex with borax. N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 33; Arch. Rubbercultuur, 1928, 12, 441—453).—For use as an anti-coagulant, 1.2—1.5 g. of borax is needed per litre of latex, whilst as a preservative the minimum proportion is 30 g. per litre. Latex treated with borax has a lower viscosity than ordinary latex. The rubber from borax-treated latex dries more slowly, and is more plastic than rubber from untreated latex; the viscosity of such rubber and the time of vulcanisation are initially lower than for ordinary rubber, but gradually change in the direction of the normal values.

D. F. TWISS.

Determination of the rubber content in latex preserved with sodium phosphate and formalin. W. SPOON and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 36; Arch. Rubbercultuur, 1928, 12, 659—674).—Investigation of the influence of various factors in the coagulation of latex preserved with 0.2% of trisodium phosphate and 0.2% of formalin leads to recommendation of the following method for determination of the rubber content. After thorough shaking of the latex, 200 g. in an aluminium bowl are coagulated by the gradual addition of 30 c.c. of 10% acetic acid or of 5% formic acid with constant stirring. The bowl is then covered and left undisturbed for 2—3 hrs. The coagulum is kneaded and then milled in a standardised manner, the wet crêpe being dried overnight in the air and then for 2 hrs., with occasional exposure of a fresh surface, in a vacuum dryer; it is finally kept in a desiccator over quicklime, and next day weighed to the nearest centigram. Each determination is made in duplicate.

D. F. TWISS.

Cream from [rubber] latex. O. DE VRIES, R. RIEBL, and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 35; Arch. Rubbercultuur, 1928, 12, 559—575).—Concentrated latex cream ("primary cream") obtained by centrifuging latex containing

ammonia was diluted with water, the mixture being again centrifuged to give a "secondary cream," which by the addition of ammoniated water and further centrifuging was made to yield a "tertiary cream." Rubber from the secondary and tertiary creams was remarkably little different in composition (ash, aqueous and acetone extracts, and nitrogen) from that from the primary cream. The tensile strength of the rubber from the creams was low; the rate of vulcanisation was lower than the standard, especially for the rubber from the "washed" creams, which also showed a greater plasticity and a lower viscosity, this feature being particularly marked in the "tertiary" rubber. Rubber from the "primary skim" had high viscosity, normal plasticity, rather rapid vulcanisation, and normal tensile strength but high slope; the rubber from the "secondary skim" was variable in character. Examination of a number of samples of rubber from different "primary" creams revealed rather wide variations in composition and in vulcanisation behaviour, and did not support the view that centrifuging would yield an especially uniform rubber. D. F. TWISS.

Plasticity determinations in crude rubber. VI. **Changes in plasticity on keeping.** O. DE VRIES (Comm. Proefstat. Rubber, No. 32; Arch. Rubbercultuur, 1928, 12, 411—422).—Samples of smoked sheet showed a considerable decrease in plasticity when kept for from one to four years; a sample of pale crêpe showed no such decrease. Samples of crêpe and smoked sheet after twelve years' storage had become somewhat hard and brittle, but tensile strength and slope after vulcanisation indicated no great deterioration, the only marked alteration being the decrease in viscosity. Shipment from Java to America and back had no definite influence on the rubber. The changes in plasticity on storage cause a much greater degree of variability than the combined factors in the preparation of the rubber, and it is possible that modern standardised methods of preparation are tending to a product of more constant characteristics. D. F. TWISS.

Physical influence of selenium-red on accelerated rubber mixtures. R. DITMAR (Chem.-Ztg., 1929, 53, 239).—Selenium-red (12 pts.) in various mixtures containing also rubber (100 pts.), sulphur (3 pts.), zinc oxide (10 pts.), whiting (40 pts.), and organic accelerator (2 pts.) had no physical influence on some, but on others it had a hardening effect dependent on the accelerator present. The vulcanised products showed satisfactory resistance to deterioration by ageing. D. F. TWISS.

PATENTS.

Production of creams and pastes from aqueous dispersions of rubber or rubber-like materials. DUNLOP RUBBER CO., LTD., and R. C. DAVIES (B.P. 306,994, 1.12.27).—Creams or paste-like mixtures are obtained from aqueous dispersions such as of rubber by adding, under agitation, a concentrated sodium cellulose xanthate gel containing, *e.g.*, 25—30% of cellulose. The dispersions may have been compounded previously; compounding ingredients also may be incorporated in, or during the addition of, the cellulose xanthate or in the resulting creams or pastes. The final mixtures

may be used for such purposes as moulding, spreading, etc., or may be coagulated. D. F. TWISS.

Production of coherent deposits of organic substances from aqueous dispersions thereof by electrodeposition. ANODE RUBBER CO., LTD. From S. E. SHEPPARD and C. L. BEAL (B.P. 307,585, 21.1.28).—In the electrophoretic deposition of organic substances such as rubber from aqueous dispersions, the temperature of the bath and particularly of the deposition surface is kept low by cooling. D. F. TWISS.

Manufacture of ingredients for the compounding of rubber. IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, W. J. S. NAUNTON, and A. SHEPHERDSON (B.P. 307,155, 11.1.28).—Rubber-compounding ingredients such as fillers, reinforcing agents, colours, and accelerators are coated with a thin film of suitable material, *e.g.*, oils, higher fatty acids, or paraffin wax. These may be applied to the compounding ingredients in the form of solutions or dispersions in media in which the latter are substantially insoluble. Compounding ingredients so treated can be mixed into rubber with exceptional ease. D. F. TWISS.

Production of rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,375, 5.12.27 and 19.6.28. Addn. to B.P. 300,719; B., 1929, 66).—Emulsions of synthetic rubber, with or without the presence of natural latex, and after removal of part of the stabilising agent, are coagulated by proteolytic enzymes such as papain, pancreas extract, or banana juice. The results may be modified by the addition of substances, *e.g.*, hydrocyanic acid or hydrogen sulphide, which assist the enzymes; the properties of the product can also be improved by imparting such a p_H that coagulation may be retarded and by introducing saline buffering substances. D. F. TWISS.

Manufacture of artificial rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,808, 12.9.27).—In the production of rubber-like polymerides of butadiene, this hydrocarbon is produced by way of α -butylene glycol, aldol, acetaldehyde, and acetylene, the last-named being formed from methane, *e.g.*, by passage through an electric arc so as to ensure the absence of substances harmful to the subsequent processes. D. F. TWISS.

Making rubber conversion products. A. E. WHITE. From B. F. GOODRICH CO. (B.P. 307,134, 20.12.27. Cf. B.P. 249,172; B., 1926, 453).—Rubber is converted into isomeric products by heating a solution containing also phenol with a strong non-oxidising mineral acid or a sulphonic acid or sulphonyl chloride. The resulting solutions may be applied as adhesive paints or the acid-freed products may be employed for the production of articles by moulding. D. F. TWISS.

Preparation of halide additive products, polymerides, and oxides of rubber. GOODYEAR TIRE & RUBBER CO., ASSEES. of H. A. BRUSON (B.P. 285,071, 16.12.27. U.S. 11.2.27).—If rubber, in solution or in the solid state, is treated with metallic salts, especially halides, *e.g.*, stannic chloride, antimony pentachloride, titanium tetrachloride, or ferric chloride, additive compounds are obtained; the metallic chloride can then be

removed, *e.g.*, by treatment with alcohol, acetone, or water. The remaining polymeric hydrocarbon is obtained in the form of white powder or flakes. If air or oxygen is admitted during the reaction, powdery insoluble oxides or polymerides of rubber are produced.

D. F. TWISS.

Rubber-like substances. E. W. HULTMAN, Assr. to F. P. DUNCLEE, J. MONTELEONE, and W. R. SIMONS (U.S.P. 1,704,194, 5.3.29. Appl., 29.10.27).—Mineral oil substantially free from constituents boiling below 205° is treated in the liquid state below 205° with a polymerising agent, *e.g.*, cerium oxide or tin oxide; it is then cooled, *e.g.*, to 17°, and treated with an activated reducing gas (produced for example by subjecting carbon monoxide and/or methane to contact with platinum or nickel at about 205°). It is then subjected to a halogenating agent, such as chlorine, bromine, or boron trifluoride, when a soft rubbery and vulcanisable product is obtained and can be separated from the bulk of the oily material.

D. F. TWISS.

Attachment of india-rubber or the like to metal or other surfaces. DUNLOP RUBBER CO., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 307,180, 8.2.28).—Coverings of gauze or mesh work are fixed to the surfaces; the facing material is then applied in the form of solution or aqueous dispersion, *e.g.*, by spreading, spraying, or electrodeposition. The dispersion of rubber or similar materials may be in a concentrated and/or compounded condition and vulcanisation may be effected concurrently with drying.

D. F. TWISS.

Vulcanisation of rubber or the like coverings of metal rolls. C. MACINTOSH & CO., LTD., and H. W. WOLTON (B.P. 307,628, 10.3.28).—In the vulcanisation of rubber coverings on metal rolls an electric current at low potential but high amperage is passed through the central spindle or an alternating current is passed through a wire coil introduced into the interior of the hollow metal roll. Evenness of heating and of vulcanisation are thus obtained.

D. F. TWISS.

XV.—LEATHER; GLUE.

Chemistry of the liming process [for hides]. A. STEIGMANN (Collegium, 1928, 653—657).—Cystine was treated with milk of lime, alone and with 8% solution of sodium hydroxide, ammonia, ammonium chloride, and calcium chloride, respectively. After 8 days sodium hydroxide had liberated sulphur from the cystine, whereas no free sulphide was present in the cystine treated with calcium hydroxide alone. This is attributed to the formation of a calcium hydroxide-cystine complex. The hydrolysis of cystine was accelerated by sodium hydroxide and ammonia, and hindered by the addition of calcium chloride to the milk of lime. Solutions of cystine, tyrosine, and albumin when treated with sodium sulphide solution yielded additive products.

D. WOODROFFE.

Periodic and continuous tanning processes. B. SCHWARZBERG (Collegium, 1928, 661—667).—In most tanning processes the liquors are strengthened periodically, and the rate of tannage is greatest immediately after each strengthening, and then gradually

diminishes. A continuous process of strengthening to maintain a constant rate of tannage could be arranged by providing for a slow continuous flow of tan liquor through each pit, paddle, or drum. The liquor in the last-named is allowed to run off at the same rate as the strengthening liquor is run into it.

D. WOODROFFE.

Determination of the volatile acids in tannin baths. A. PONTE (Boll. Uff. Staz. Sperim. Ind. Pelli, 1929, 7, 57—63).—This method, based on that applied by Rota to wines, consists in diluting a certain volume of the bath with 10 c.c. of 96% alcohol and water to a total volume of 200 c.c., and distilling 100 c.c. of the liquid until exactly 72 c.c. of distillate are collected. This quantity of distillate, which contains one half of the total volatile acid, is titrated and the acid found multiplied by two. If a sulphited bath is tested in this way the distillate will contain sulphur dioxide. In this case the titrated solution is acidified with sulphuric acid and titrated with 0.02*N*-iodine solution, and a correction applied to the result of the acidimetric titration.

T. H. POPE.

State of combination of acid sulphate in chrome [tanned] leather. H. B. MERRILL and J. G. NIEDERCORN (Ind. Eng. Chem., 1929, 21, 252—253).—Portions of well-washed chromed hide powder were each shaken for 2 hrs. with different amounts of *N*-sodium hydroxide or *N*-sulphuric acid, then washed for 1 hr., and dried. The leather as tanned contained a chromium complex of 32.3% acidity. Treating the leather with acid did not much increase the acidity of this complex. Partial neutralisation of the leather with alkali, however, decreased the total acid sulphate in direct proportion to the amount of alkali added until the acidity of the chromium complex was one half of its original value. The chromium-bound and protein-bound acid contents both decreased with increasing neutralisation of the leather. When the chromium-bound acid had been reduced to 17.4% acidity, the leather no longer contained protein-bound acid. Further neutralisation reduced the acidity of the chromium complex.

D. WOODROFFE.

Consistency of animal glue. D. BROUSE (Ind. Eng. Chem., 1929, 21, 242—247).—The viscosity of solutions of a standard, a higher-grade, and a lower-grade animal glue, respectively, was determined at different temperatures and shown to be a hyperbolic function of the temperature. The better the grade of glue or the greater the concentration the greater is the viscosity. The viscosity-temperature curve was altered with respect to the axes by changes in concentration or by using a different grade of glue, but its shape remained the same. The temperature of gelation increased as the grade of glue improved, but the different grades showed the same sharp change from viscosity to plasticity, only at different temperatures. The usual methods for expressing consistency or viscosity in the form of an equation do not apply, and the Herschel expression is favoured. There is no difference in the fundamental properties underlying glueing technique. If the gelation temperature must be reached quickly, a low concentration of a high-grade glue or a more concentrated solution of a lower-grade glue can be used. For long

assembly periods, a low-grade glue or a dilute solution of a high-grade glue may be used. D. WOODROFFE.

Preparation of standard gelatin. J. M. HUDSON and S. E. SHEPPARD (Ind. Eng. Chem., 1929, 21, 263—264).—The following specifications are suggested. The gelatin should be prepared from well-limed calfskin and only the first extract taken at about 54°; this should be dried at a concentration > 5%, de-ashed to an ash content < 0.05%, and should have a constant iso-electric point p_H 4.7—4.9 and a viscosity of about 4 centipoises at 5% concentration, or 6 centipoises at 7% concentration and 40°. The jelly strength should be about 300 g. (Bloom) for a 7% solution chilled by melting ice for 16—24 hrs. The gelatin should be nearly colourless, free from grease, fats, or heat-coagulable protein, and have a minimum absorption of blue light at a definite thickness of a 5% solution. D. WOODROFFE.

Food gelatin values relative to concentrations. M. BRIEFER and J. H. COHEN (Ind. Eng. Chem., 1929, 21, 264—265).—The jelly strengths of different solutions (1—10% concentration) of various grades of gelatin were found to vary directly as the concentration. These determinations are only of value commercially at about 3% concentration, at which the jelly strength-concentration curve is not linear. Gelatin should be graded on a 3% concentration basis instead of on a 6.67% value as at present. D. WOODROFFE.

Treatment of tanning wastes. FALES.—See XXIII.

PATENTS.

Method of depilating hides. J. PEINY (F.P. 621,577, 16.9.26).—The flesh side of the hides is painted with a solution, at 35°, of sodium chloride, sulphur compounds, *e.g.*, alkali or arsenic sulphides, and sodium hydroxide or carbonate, to which may be added calcium carbonate. The hair is loose in 0.5—3 hrs.

D. WOODROFFE.

Process for tanning hides and skins. J. HELL (G.P. 451,988, 13.4.24).—Hides or skins are treated for about 20 hrs. with pastes of suitable alkalinity, obtained by mixing insoluble prepared or natural carbonates, *e.g.*, chalk, limestone, dolomite, magnesite, precipitated chalk, with solutions of suitable salts or mixtures of salts, *e.g.*, calcium or magnesium chloride or magnesium sulphate, to which may be added formaldehyde, quinone, or other known tannins. The product is dark coloured, but very soft and tough. D. WOODROFFE.

Manufacture of sulphonc acids with tanning properties. M. MELAMID (G.P. 451,534, 26.3.22).—Anthracene oil or soft pitch is strongly oxidised, the product extracted with mineral oil, the solvent distilled off, and the residue sulphonated and condensed or esterified with either formaldehyde or acetylene respectively and an aromatic sulphochloride.

D. WOODROFFE.

Manufacture of tanning materials from sulphite-cellulose liquors. C. and C. P. HÜTTENES (G.P. 451,913, 12.4.25).—Sulphite-cellulose liquors, from which the sugars may have been removed by fermentation, are treated with an alkali or alkaline-earth hydroxide and/or

sulphide to remove heavy metals, heated, concentrated to d 1.26, treated with alum and sulphuric acid, or with sodium, ammonium, or chromium sulphate to precipitate the lime, and finally concentrated to form a pulverulent mass when cold. D. WOODROFFE.

Dressing of leather, skins, etc. HANSEATISCHE MÜHLENWERKE A.-G., and B. REWALD (B.P. 306,672, 23.1.28).—A lecithin or phosphatide other than egg yolk is mixed with an oil, which may be sulphonated, emulsified with water, which may contain a small quantity of alkali, ammonia, or soap, and the emulsion employed in fat-liquoring of leather. The oil may be replaced by an aqueous swelling or a solution of lecithin, to which has been added an aliphatic or aromatic sulphonc acid and/or albumin etc. D. WOODROFFE.

Impregnated material [leather]. L. F. WHITNEY and W. E. WHITNEY, Assrs. to RAJET Co. (U.S.P. 1,682,652, 28.8.28. Appl., 15.1.24).—Chrome-tanned leather is impregnated at 55—60° with a solution of cellulose nitrate or acetate in a low-boiling solvent, such as tetrachloroethane, acetone, or methyl alcohol, containing a filler, *e.g.*, paraffin wax, carnauba wax, an anti-shrinking agent, such as glycerin or ethylcarbamide, and a high-boiling solvent or gelatinising agent, *e.g.*, ethyl phthalate, phenyl phosphate. R. BRIGHTMAN.

Manufacture of glue, gelatin, etc. in the form of globules or pellets. BRIT. GLUES & CHEMICALS, LTD., and R. B. DREW (B.P. 306,622, 1.12.27).—A concentrated solution of the material is delivered in the form of drops on a moving surface upon which a film of ice has been frozen or ice shavings or snow has been delivered. D. WOODROFFE.

Treatment of gelatin. J. T. DIXON (B.P. 306,439, 18.11.27).—An alkaline solution of gelatin or glue is mixed with 30—50% of a solution of a saponified fatty compound, *e.g.*, castor or linseed oil, previously sulphonated if desired, with or without the addition of a tanning or hardening agent (a chromate, formaldehyde, or hexamethylenetetramine). The product may be subsequently treated in a hardening bath. Finally it is mixed with 1—2% of phenol or sodium or potassium iodide, spread, rolled or moulded, and dried.

D. WOODROFFE.

Increasing wetting capacity of liquids (B.P. 291,070).—See VI.

XVI.—AGRICULTURE.

Routine mechanical analysis of soils. P. KÖTTGEN and H. HEUSER (Z. Pflanz. Düng., 1929, 13A, 137—159).—The relative merits of sedimentation and pipette methods and the use of sieves for large particles are discussed. In pipette methods the streaming motion of the suspension caused by the withdrawal of the sample affects the downward fall of the particles. This effect is slight with large particles, but marked with the finer ones. The size and shape of the jet of the pipette and the period occupied in sampling therefore influence the composition of the sample. A wide horizontal jet minimises the disturbance of the motion of the smaller particles. The use of glycerin and glycerin-water suspensions, by decreasing the rate of sedimentation,

allows accurate values to be obtained for larger particles by the pipette method. Details of apparatus and laboratory technique for pipette sampling, filtration of suspension, and sieve-washing are described.

A. G. POLLARD.

Method of water control for sand cultures.

O. V. S. HEATH (Ann. Bot., 1929, 43, 71—79).—An apparatus for measuring the moisture contents of sand cultures is described. It consists of a porous porcelain candle full of water, buried in the sand, and with a mercury manometer attached. By calibrating the manometer in terms of sand moisture content, measurements of the latter may be made to 1·2% of the weight of sand.

E. A. LUNT.

Phosphate and lime contents of Oldenburg soils.

S. GERICKE (Z. Pflanz. Düng., 1929, 8B, 1—15).—The soils examined are largely acidic and respond to liming in pot experiments. The marsh and sandy soils are characterised by an apparent sufficiency of total phosphate, of which, however, only a relatively small proportion is root-soluble.

A. G. POLLARD.

[Soil] reaction experiments with mustard and oats in connexion with nutrient-requirement trials. L. HELLER (Z. Pflanz. Düng., 1929, 8B, 37—40).—The importance of determinations of the effect of soil reaction, lime content, and lime requirement on the growth of individual crops as a necessary complement to fertiliser-requirement experiments is emphasised.

A. G. POLLARD.

Determination of small quantities of nitrates in soils and plants. J. BLOM and C. TRESCHOW (Z. Pflanz. Düng., 1929, 13A, 159—190).—The method is based on the formation of 5-nitro-*m*-4-xylanol, its subsequent distillation in steam, and colorimetric determination after rendering alkaline with sodium hydroxide. Organic matter in the sample is removed by heating with sulphuric acid and permanganate. No loss of nitrate occurs during this process, and there is no appreciable oxidation of ammonia or of amino-acid to nitrate. Excess of permanganate may be removed by means of oxalic acid before addition of the nitroxylanol. With 0·05 mg. of nitrate ion an accuracy of $\pm 2\%$ is attainable.

A. G. POLLARD.

Cold- and hot-fermented manure. GERLACH and SEIDELL (Z. Pflanz. Düng., 1929, 8B, 15—37).—Published literature is critically reviewed. Hot-fermented manure has rather less dry weight and total nitrogen, but more water-soluble nitrogen than cold-fermented manure. The work of Krantz and of Löhnis and Ruschmann on the fermentation process was not confirmed. The decomposition in soil of hot-fermented manure was slightly slower than with the cold-fermented product, and crop yields were rather lower.

A. G. POLLARD.

Determination of the nitrogen fixation by legumes by means of the nitrogen-base ratio. A. STORCK and A. RIPPEL (Z. Pflanz. Düng., 1929, 13A, 158—159).—Comparison is made of the nitrogen-base ratio (see Rippel and Ludwig, A., 1926, 439) of legumes at two successive stages of growth, e.g., before and after flowering. Relative differences in the nitrogen equi-

valent in the two cases are indicative of the extent of nitrogen fixation.

A. G. POLLARD.

Chemical effect of gypsum, sulphur, iron sulphate, and alum on alkali soil. W. P. KELLEY and A. ARANY (Hilgardia, 1928, 3, 393—420).—Gypsum precipitated the soluble carbonate as calcium carbonate, whilst the other materials either decomposed the carbonate or converted it into hydrogen carbonate. The exchange complex was also affected. Acid formed by the oxidation of sulphur or hydrolysis of ferrous sulphate or alum causes calcium to pass into solution, whence the sodium content of the exchange complex is decreased.

CHEMICAL ABSTRACTS.

Fertiliser experiments in the Vladimir district.

L. L. BALASHIEV (Trans. Sci. Inst. Fert., Moscow, 1926, No. 37, 5—60).—Loam soils respond best to phosphorus, and light soils to nitrogen and potassium; manganese oxides were not advantageous. Potassium fertilisers lower the starch content of potatoes.

CHEMICAL ABSTRACTS.

Fertiliser experiments in the Tver district.

A. V. KAZAKOV and S. L. SHAPIRO (Trans. Sci. Inst. Fert., Moscow, 1926, No. 37, 61—91).—The sandy and loam soils respond primarily to phosphorus.

CHEMICAL ABSTRACTS.

Field experiments with fertilisers in the Tula district. A. V. KAZAKOV (Trans. Sci. Inst. Fert., Moscow, 1926, No. 35, 9—42, 43—83).—Experiments on podzolised loams, sandy loam, grey forest soils, dark forest soils, degraded and leached chernozem during the period 1903 to 1925 are summarised. Fertilisation experiments with phosphate are recorded.

CHEMICAL ABSTRACTS.

Fertiliser experiments in the Moscow district.

L. L. BALASHEV (Trans. Sci. Inst. Fert., 1926, No. 35, 83—153).—Grain crops respond to fertilisers in the order: nitrogen > phosphorus > potassium; potatoes, flax, and clover respond in the order: potassium > phosphorus > nitrogen. Meadow hay responds primarily to potassium.

CHEMICAL ABSTRACTS.

Phosphate requirement of barley at different periods of growth. W. E. BRENCHLEY (Ann. Bot., 1929, 43, 89—110).—Experiments have been carried out in water cultures to investigate the effect of the withdrawal of the phosphorus supply on the barley plant at varying stages during its growth. The minimum period from germination during which phosphates must be supplied in order to obtain normal growth is six weeks; during this period phosphate absorption is sufficient to enable the plant to make its maximum dry weight.

E. A. LUNT.

Plant nutrition. I. Effect of manurial deficiency on the respiration and assimilation rate in barley. F. G. GREGORY and F. J. RICHARDS (Ann. Bot., 1929, 43, 119—161).—The effect of nitrogen, phosphorus, and potassium deficiency on the respiration and assimilation rates of the barley plant has been investigated. The following results have been obtained: nitrogen-deficient plants exhibit a subnormal respiration and a slightly subnormal assimilation rate; phosphate-deficient plants a normal respiration and a

slightly supernormal assimilation rate; potassium-deficient plants a supernormal respiration and a subnormal assimilation rate.

E. A. LUNT.

Influence of nutrients on the value of bast-fibre plants (flax and nettle). I. H. FABIAN (*Faserforsch.*, 1928, 7, 1—56; *Chem. Zentr.*, 1928, ii, 1605).—Lack or excess of potassium, nitrogen, or phosphorus is unfavourable for the production of fibre. Diminution of fibre yield occasioned by nitrogen can be to a certain extent compensated by means of potassium. The most favourable fertilisation for the production of fibre is that which best stimulates growth, but does not afford the maximum production of stem.

A. A. ELDRIDGE.

Specifications for petroleum oils to be used on plants. E. R. DE ONG (*J. Econ. Entomol.*, 1928, 21, 697—702).—The presence of 0.05% of sulphur may be injurious; oil emulsion containing free sulphur is not recommended. Specifications are given of oils for various purposes.

CHEMICAL ABSTRACTS.

PATENTS.

Fertilisers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,230, 30.3. and 31.8.28).—Siliceous material, e.g., river sand, is heated above 250° with a quantity of phosphoric acid corresponding to 1.5—8 times the weight of silica present. The product may be treated with ammonia or mixed with other fertilisers.

L. A. COLES.

Preparation of fertilisers. F. ROTHE and H. BRENEK, Assrs. to RHENANIA VER. CHEM. FABR. A.-G. (U.S.P. 1,704,218, 5.3.29. Appl., 11.6.25. Ger., 23.6.24).—See B.P., 235,860; B., 1925, 731.

XVII.—SUGARS; STARCHES; GUMS.

Drying of sugar beet and extracting the sugar from it in a diffusion battery. N. L. KARAVAIEV and A. P. PALKIN (*Bull. Sakharotrest*, 1928, No. 5, 40—44, and *Acta Univ. Asia Med.*, 1928, [vi], 3—12).—Sliced sugar beet sun-dried to 8—10% H₂O can be stored indefinitely without increase of invert sugar. Extraction of the dried beet slices requires 50% longer time than with raw beets, but the diffusion juice is twice as concentrated.

CHEMICAL ABSTRACTS.

Treatment of beet juices with sodium carbonate, and the juice alkalinity. V. STANEK (*Z. Zuckerind. Czecho-Slov.*, 1929, 53, 173—177).—Sodium carbonate added for the reduction of the calcium content of juices during their clarification should be introduced before the first carbonatation, e.g., to the juice in the measuring tanks. If triple carbonatation is applied, the alkali should be mixed with the second carbonatation juice before turning on the gas, the colloidal calcium carbonate formed being thus entrained by the carbonatation scums. In practice, it is found that the quantity of sodium carbonate added should be somewhat more than the equivalent of the calcium present, otherwise the desired final alkalinity will not be realised.

J. P. OGILVIE.

Solubility of carbon dioxide in sugar factory juices, and its formation through some amino-acids. V. MAJER (*Z. Zuckerind. Czecho-Slov.*, 1929, 53,

213—229).—Diffusion, first carbonatation, second carbonatation, and boiled clarified juices were compared in respect of carbon dioxide absorption. It was found that carbon dioxide is present in such juices in three forms: (a) in solution, (b) combined with alkalis, and (c) combined with aspartic and glutamic acids as carbamates. When boiled in aqueous solution, these last-named compounds slowly decompose, which fact probably explains the gradual separation of calcium carbonate observed during the boiling-up of second carbonatation juices in the factory.

J. P. OGILVIE.

Pressure evaporation [in the beet factory]. K. VESELY (*Z. Zuckerind. Czecho-Slov.*, 1919, 53, 204—206).—In pressure evaporation in white sugar manufacture, colour formation is due to defects in the design of the apparatus, whilst other factors are incorrect clarification and filtration, and the absence of sulphites. There is no question as to the suitability of such apparatus under proper operating conditions.

J. P. OGILVIE.

Rapid filtration of syrup through sand. G. J. T. HEYNING (*Archief Suikerind. Nederl.-Indië*, 1928, 36, 1179—1182).—The filter consisted of a cylindrical tank filled with sand (having grains about 2 mm. diam.) to a depth of about 1 metre, the syrup being allowed to percolate downwards. Before commencing the filtration, air was expelled from the medium by passing juice upwards through it, in which manner also later cleaning was accomplished. The insoluble matter in the syrup before and after this sand filtration was found to be 0.028 and 0.005%, using a laboratory centrifugal for the determination.

J. P. OGILVIE.

Alkaline defecation-carbonatation process for clarifying cane juices. K. DOUWESDEKKER and P. C. NIKOLA (*Archief Suikerind. Nederl.-Indië*, 1928, Med. 13, 721—749).—Sufficient lime is added to the cold juice to impart a slightly alkaline reaction, after which carbon dioxide is pumped in until an acidity corresponding to about 1000 mg. CaO per litre is reached, or about p_H 6.7. The main quantity of lime is then added until the alkalinity is between 700 and 800 mg. After being allowed to settle, the clear juice is treated with carbon dioxide, filtered, and sent to the evaporators, whilst the mud is heated, settled, and filtered, the clear liquids being added to the acid juice before the addition of the main quantity of lime.

J. P. OGILVIE.

Modification of the sulphitation process of clarification of cane juices. H. H. E. SUSSBACH (*Archief Suikerind. Nederl.-Indië*, 1928, 36, 1187—1189).—The raw juice is neutralised by the addition of a suitable quantity of milk of lime (d 1.1), and allowed to remain at 55° for about 5 min., which conditions are stated to be suitable for colloid separation. A further addition of milk of lime is then made, so as to complete the usual amount, following which the juice is sulphited in the usual manner. A better clarification and less glucose decomposition than ordinarily are claimed.

J. P. OGILVIE.

Acidity determination in sulphitation, using the p_H value. M. DEN HARTOGH (*Archief Suikerind. Nederl.-Indië*, 1928, 36, 1175—1179).—In the juice sulphiting operation, treatment with the gas is carried

on till the is p_H 7.1—7.2, using the colorimetric method of determining hydrogen-ion concentration, and bromothymol-blue as the indicator. For syrup sulphitation control is effected by means of chlorophenol-red, so as to obtain an indication of p_H 5.3—5.5. A comparator in connexion with a rotating disc containing the standard buffer solutions recommended for such determinations is described.

J. P. OGILVIE.

Refining qualities of Philippine raw sugars. P. SENGSON (Sugar News, 1928, 9, 807—811).—It is advised to maintain the polarisation between 96.5 and 97.0 in the production of raws for the American refiners. The water content of such sugars has now been reduced to an average of 0.785%, whilst the ash (direct incineration) is 0.246%. Improvements have been effected in the clarity of the re-dissolved sugars, and also in the filtration rate. It has further been possible to reduce the percentage of "total smalls" (grains having diameter from 0.15 to 0.60 mm.), so that this figure, which in 1924—5 was 54%, is now about 25%, the limit acceptable to the refineries.

J. P. OGILVIE.

Comparative examination of "Norit" and "Carboraffin" [in sugar refining]. K. REICH and G. VAVRINECK (Z. Zuckerind. Czecho-Slov., 1929, 53, 285—289).—Analytical figures observed for "Norit Supra 3X" and "Carboraffin" were, respectively, loss of weight at 150°, 12.73, 11.10; ash, 6.50, 2.98; water-soluble ash, 2.27, 1.57%; reaction, alkaline, neutral; Fe_2O_3 , 0.68, 0.34%. Inverting effect was observed by heating 2 g. of each carbon with 100 c.c. of a 50% solution of sugar for $\frac{1}{2}$ hr. at 90°, and determining the copper reduced from 50 c.c. of Fehling's solution, the figures found being 30.9 and 58.6 mg. (blank 26.0 mg.). To charge a Danek filter of 13 sq.m. surface, the weights of the two carbons to give the same thickness of layer were 30 kg. and 20 kg. After a factory run lasting 49 hrs. using such filters, the colour removed by the two media was, respectively, 29.9 and 39.4%. Boiling the used carbon for 1 hr. with water showed only a slight revivification with the "Norit," but restored the "Carboraffin" to 50% of its original decolorising power.

J. P. OGILVIE.

Instrument for determining the specific gravity of refined sugar. V. YANOVSKI (Bull. Sakharotrest, 1928, No. 1, 53—60).

CHEMICAL ABSTRACTS.

Colouring matters formed by the caramelisation of sucrose and by the action of lime on invert sugar. M. GARINO and A. TOSONOTTI (Giorn. Chim. Ind. Appl., 1929, 11, 8—13).—The constituents of caramel prepared by heating sucrose and termed caramelan, caramelen, and caramelin by Gélis (Ann. Chim. Phys., 1858, [iii], 52, 352) are not individual compounds but mixtures. Caramelan may be freed from caramelen by repeated treatment with 84% alcohol, followed by evaporation of the solution obtained to dryness. This procedure gives a caramelan completely soluble in 84% alcohol and of constant colouring power if entirely dissolved, but when only a small quantity of the alcohol is used the substance dissolved has far less colouring power (about 260 Stammer units instead of 800). Caramelan tenaciously retains alcohol, which is

removed only by repeated boiling with water or by vacuum evaporation almost to dryness. Caramelen does not dissolve any large amount of caramelin, and may be freed from this by means of cold water, in which caramelin is insoluble; the colouring power of caramelen is about 18,000 Stammer units. Caramelin is probably not produced during the manufacture of sugar. The products obtained by caramelisation of dextrose or invert sugar resemble those formed from sucrose chemically, but differ in other ways, the colouring power, for instance, being enormously lower. Glucic and apoglucic acids, formed when invert sugar is heated in presence of an alkali, are described. Certain physical and chemical properties of all the above products have been investigated, the most noteworthy results being that solutions of both caramelan and the calcium salt of glucic acid exhibit marked surface activity, and that the intensity of colour of a caramelan solution is greatly increased on addition of alkali.

T. H. PORE.

Alteration of sugar-like substances in sulphite-cellulose cooking. HÄGGLUND and JOHNSON.—See V. Furfuraldehyde derivatives. YAMADA.—See XVIII. Fluorescence of honey. ORBÁN and STITZ. Hydroxymethylfurfuraldehyde in honey. FIEHE and KORDATZKI.—See XIX.

PATENTS.

Saturation of sugar juice. A. G. VERHEUL (Dutch P. 16,981, 12.12.23).—The production of carbon dioxide by fermentation of molasses and the saturation of the raw juice are so adjusted as to balance one another.

W. J. BOYD.

Decolorisation of sugar juice by means of hyposulphites. SOC. IND. DES DÉRIVÉS DU SOUFRE (F.P. 628,128, 26.1.27).—A solid hyposulphite is added at intervals during concentration, a total of 15—20 g. $Na_2S_2O_4$ per ton of sliced beets being used. The evaporated liquor retains its pale colour and can be very highly concentrated owing to the reduction in viscosity due to the salts.

W. J. BOYD.

Purification of molasses [for manufacture of spirit and yeast]. A./S. DANSK GAERINGS IND. (B.P. 283,970, 20.1.28. Austr., 21.1.27).—The diluted molasses is partly or entirely freed from betaine and related products by the action of betaine-decomposing bacteria. This process may be combined with, or may follow, the usual production of lactic acid by bacteria. After decantation or filtration a further purification may be effected by precipitating the alkaloids with tannin, removing the excess of the latter by means of lime, precipitating the colouring matters by producing a precipitate of aluminium hydroxide, and removing the sulphuric and carbonic acids by adding a barium compound, e.g., barium aluminate. Excess of barium and aluminium ions may then be removed by addition of water-glass or phosphoric acid. The liquid is finally pressed through a filter.

W. J. BOYD.

Separation of betaine hydrochloride and potassium chloride from waste molasses. Y. TAKAYAMA (B.P. 304,071, 13.3.28).—Crude potassium chloride is crystallised from concentrated waste liquor of beet molasses by addition of hydrochloric acid (as gas or

solution) and removed by filtration, and betaine hydrochloride containing potassium chloride is crystallised by concentration of the filtrate. This is dissolved in water, and the solution neutralised with lime, filtered, and concentrated to crystallise the potassium chloride, the free betaine remaining in solution. The potassium chloride crystals are removed, hydrochloric acid is added, and after further concentration crystals of betaine hydrochloride are obtained containing little ash. Instead of adding hydrochloric acid after removal of the potassium chloride, dilute sulphuric acid equivalent to the calcium chloride present may be added, the calcium sulphate filtered off, and the betaine hydrochloride obtained on concentration of the filtrate. W. J. BOYD.

XVIII.—FERMENTATION INDUSTRIES.

Strengthening of yeast by fermentation under pressure. SCHUSTER (Woch. Brau., 1929, 46, 99).—Small- and large-scale fermentations confirmed the observation of Windisch (B., 1928, 685; 1929, 69) that fermentation under pressure leads to increase of size and improvement in appearance of the yeast. The yeast, however, did not settle so quickly or firmly as in the control fermentations. When subsequently used under ordinary conditions it gave good vigorous fermentation and returned to its original appearance. The strengthening effect is only of secondary importance to the possibilities of the recovery and utilisation of carbon dioxide. F. E. DAY.

Furfuraldehyde derivatives in fermentation products. M. YAMADA (Bull. Chem. Soc. Japan, 1929, 4, 31–35).—Hydroxymethylfurfuraldehyde-*p*-nitrophenylhydrazone was prepared from the distillate (160–180°) from 50% aqueous dextrose solution. The colour reactions with aniline + acetic and barbituric acids and the phloroglucinol test showed that the distillation of l  vulose solutions also yields small quantities of hydroxymethylfurfuraldehyde. Arabinose solutions yield furfuraldehyde, whilst sak  , sh  yu (soya sauce), and sh  chu (Japanese whiskey) yield both in varying proportions, and fusel oil mainly furfuraldehyde. The proportion of furfuraldehyde is increased by repeated distillation, owing to decomposition of the hydroxymethyl derivative. The substances in the distillates from fermented liquids which give a red coloration with the aniline-acetic acid reagent are mixtures of furfuraldehyde with its hydroxymethyl and methyl derivatives, derived from the partial decomposition of pentoses, hexoses, and methylpentoses respectively (cf. Hastie and Dick, B., 1928, 797). F. E. DAY.

Apparatus for determining water content of barley and malt. F. WINDISCH (Woch. Brau., 1929, 46, 96–97).—The apparatus depends on the much greater dielectric capacity of water as compared with other grain constituents, and consists of a condenser which can be filled with a definite quantity of material, together with suitable means for determining the change in the capacity of the condenser. Figures are quoted comparing the results of seven methods of determining the water content of two varieties of wheat. Though all methods are only of relative value, and so far the proposed method yields consistent results, besides

having the advantage that it may be carried out in one minute, it cannot be accepted for barley and malt analysis without further investigation. F. E. DAY.

Titration [of worts and beers] by stages. P. KOLBACH (Woch. Brau., 1929, 46, 91–96).—A critical consideration of the principles involved in titration by stages. The results of the method may be of value simply as a measure of the buffering over a given range of p_H or of the amount of a buffering substance or substances if these are almost exclusively responsible for the buffering over a range of p_H . By the titration of worts by stages, before and after treatment with baryta, it is shown that the titration value between p_H 5.67 and 7.07 is a measure of the amount of phosphates, whilst titration above p_H 7.07 gives a measure of the protein degradation products, though this is more accurately determined by a formol titration. On the other hand, below p_H 5.67 the cause of buffering is not important, but the degree of buffering is so, since it affects the fall of p_H during fermentation. It is suggested that determinations of p_H , titration values with alkali to p_H 7.07 and with acid to p_H 4.27, and a formol titration will give more information regarding a wort than the usual titration by stages, with little more trouble. The initial values can be corrected for the small variation of the observed p_H from 5.67. Since beers of low p_H and less strongly buffered are more stable than those having the converse properties, determinations of p_H , titrations to p_H 5.67 and 7.07, and formol titration should give as much information as can at present be obtained by such methods. F. E. DAY.

Ropiness in wine. G. MEZZADROLI and E. VARETON (Giorn. Chim. Ind. Appl., 1929, 11, 14–17).—When treated with norit, ropy wine loses its ropiness, but this returns after a few days. Treatment of the wine with sulphur dioxide prevents the appearance of further turbidity, but does not diminish the ropiness. When these two reagents are used together the ropiness disappears completely and permanently; vigorous agitation of the wine, a low temperature, and protracted action of the reagents assist the action. T. II. POPE.

Evaluation of wine distillates and wine brandies. G. B  TTNER and A. MIERNEISTER (Z. Unters. Lebensm., 1928, 56, 492–498).—Results of the examination of a number of still wines, wine distillates, and brandies by the method of Graff (B., 1927, 953) are given. It is concluded that the method is useful, especially for the detection of grosser adulterations. W. J. BOYD.

Manufacture, composition, and evaluation of Samos wine. A. KICKTON and P. BERG (Z. Unters. Lebensm., 1928, 56, 397–422).—Samos wine is usually made by addition of spirit to the must at an early stage in the fermentation, but sometimes concentrated must or must from dried grapes is used. Its characteristics are low total acidity (0.28–0.42 g. per 100 c.c.), glycerin, and sugar-free extract (1.7–2.5 g. per 100 c.c.), and approximately equal contents of l  vulose and dextrose. Total extract is usually 21–24 g. per 100 c.c. Of late years the alcohol content has risen from 11–12.5 g. to 14–15 g. per 100 c.c. in consequence of new regulations regarding the composition of wine imported into Germany. W. J. BOYD.

Butyric acid fermentation. A. HEIDUSCHKA and C. REYMAN (Pharm. Zentr., 1929, 70, 87—92).—The production of butyric acid, under various conditions of temperature and sugar concentration, by a typical butyric acid organism belonging to the *Amylobacter* group has been studied. The medium employed was ordinary nutrient broth to which was added 2—10% of dextrose, and chalk sufficient to neutralise 20% above the theoretical maximum yield of acid. Portions each of 2 litres were employed, and fermentation was allowed to proceed till evolution of carbon dioxide ceased. The following figures show the maximum and minimum production of each acid, calculated as percentage of the theoretical yield of butyric acid, together with the corresponding conditions of temperature and dextrose concentration. Total acid, 121.4% at 37°, 2% ; 22.4% at 47°, 5%. Lactic acid, 73.5% at 27°, 2% ; 2.0% at 47°, 5%. Acetic acid, 59.4% at 37°, 2% ; 11.0% at 42°, 10%. Butyric acid, 56.3% at 37°, 5% ; 8.2% at 47°, 5%. Under the conditions for maximum production of butyric acid, 37° and 5% of dextrose, the butyric acid constitutes 60.5% of the acid produced, with 11.4% of lactic and 28.1% of acetic acids. The trace of alcohol produced has no practical significance.

F. E. DAY.

PATENTS.

Apparatus for continuous manufacture of absolute alcohol. E. RICARD, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,704,213, 5.3.29. Appl., 23.1.24. Belg., 16.4.23).—See B.P. 214,581 ; B., 1925, 185.

Molasses (B.P. 283,970).—See XVII.

XIX.—FOODS.

Determination of the degree of heating of milk. P. WEINSTEIN (Z. Unters. Lebensm., 1928, 56, 457—467).—The effect of time and temperature of heating of milk on the oxidase (Rothenfusser), aldehyde-reductase (Scharfing), catalase, and amylase reactions and on the creaming and albumin-sediment tests has been studied in order to utilise these as indicators of the previous heat treatment. Milk pasteurised at 85° for 1 min. gives a negative, milk heated at 70° for 30 min. a positive Rothenfusser test. The latter gives a negative Scharfing reaction. Regulation-sterilised milk gives a positive Rothenfusser reaction, decolorises Scharfing's reagent within 10—11 min., shows a low catalase value not exceeding 8—10 c.c. of oxygen for 100 c.c. of milk, and separates cream on keeping. Insufficiently sterilised milk shows a higher catalase value, and may show a positive amylase reaction. Milk heated at not above 55° has a strong amylase reaction and a catalase value equal to that of normal milk. These tests are also applicable as indicators of the efficiency of sterilisation by the Tödt method of flash-heating. W. J. BOYD.

Chlorine index of goat's milk. Systems of rapid analysis. J. M. CLAVERA and D. GUEVARA POZO (Anal. Fis. Quím. [Tecn.], 1929, 2, 14—22).—The determination of the chlorine index (g. Cl/litre) of goat's milk by a rapid method (Weiss, Lebensm. u. Hyg., 1922, 12, 133 ; Sirot and Joret, B., 1928, 391) is recommended for control of its purity. Four quickly determined values,

which, in the case of goat's milk in Granada and Andalusia, are normally as follows : d_{20}^{25} 1.029—1.032, fat 32—55 g./litre, ash 6.5—8.5 g./litre, chlorine 0.9—1.25 g./litre, then suffice for the estimation of its quality, and the type of adulteration practised, e.g., simple addition of water, or addition of water, sucrose, and salt, may be readily diagnosed. R. K. CALLOW.

Chemistry of sour milk. L. L. VAN SLYKE (N.Y. Agric. Exp. Sta. Tech. Bull., 1928, No. 140, 1—14).—A review. CHEMICAL ABSTRACTS.

Determination of fat in malted milk powders. C. W. BALLARD (J. Amer. Pharm. Assoc., 1929, 18, 122—123).—A modification of the Röse-Gottlieb method recommended in "Methods of analysis," Association of Official Agricultural Chemists, pp. 262 and 275, is described in which the Röhrig tube is made with a detachable portion which facilitates the manipulation of the sample. E. H. SHARPLES.

Presence of mixed glycerides in butter from cows' milk. E. DE'CONNO and E. SCOPINARO (Annali Chim. Appl., 1929, 19, 55—65).—When butter fat, purified by fusion and filtration, is dissolved in hot acetone containing one third of its volume of chloroform, the solution deposits when cooled to the ordinary temperature (about 13°) about 10% (on the weight of dissolved fat) of a white crystalline solid, which after six crystallisations has a constant m.p. (51°) and the composition of a myristodipalmitin. When cooled to —15° the filtrate yields a myristodistearin, m.p. 57.5°, to the extent of about 4% of the weight of the original fat. The residual stable, oily liquid yielded no further fractions, but showed saponification, iodine, and volatile acid values higher than those of the original fat, and thus contained all the glycerides of saturated acids of low mol. wt. (volatile) and of unsaturated liquid acids, mainly oleic. Hence the existence of the liquid glyceride, butyropalmito-olein, recorded by Bell ("The Chemistry of Foods," II, 44) and by Blyth and Robertson (Proc. Chem. Soc., 1889, 5), is not confirmed. T. H. POPE.

Cotrone cheese. E. DE'CONNO and M. FRATTURA (Annali Chim. Appl., 1929, 19, 65—75).—Analysis of this Calabrian cheese gave the following mean percentage composition : water 27.816, acidity as lactic acid 1.308, fat 22.886, fatty acids 4.185, nitrogenous substances ($N \times 6.25$) 32.142, ash 8.193, sodium chloride 5.341. The fat had acid value 25.747 and saponif. value 237.55. The nitrogen exists in the following forms : soluble 2.361, insoluble 2.783, protein 1.382, ammoniacal 0.128, non-protein 0.989, amino- 0.850. The coefficients of maturation, solubilisation, and decomposition are, respectively, 0.458, 0.268, and 0.190. These results differ from the data given by König for this product. T. H. POPE.

Calcium of cheese. K. BLUNT and E. SUMNER (J. Home Econ., 1928, 20, 587—590).—Average values are : Swiss cheese 1.05, Cheddar cheese 0.71, cottage cheese 0.077% Ca. CHEMICAL ABSTRACTS.

Commercial lecithins and lecithin preparations. J. SONOL (Rev. fac. quim. farm. Univ. La Plata, 1928, 4, No. 2, 3—40).—For the preparation of lecithin, brain or fresh egg-yolk is dried with acetone and extracted

with absolute alcohol at 50°, the alcohol being renewed three times every 12–24 hrs. After 12 hrs. at 0° the alcoholic filtrates are filtered and distilled in a vacuum. Before removal of the alcohol is complete the upper (yellowish) layer is separated from the lower (reddish) layer, freed from alcohol, dissolved in a small amount of ether, and precipitated with acetone; the procedure is repeated three times, and the lecithin freed from acetone in a vacuum. Analytical data for commercial lecithins and lecithin preparations are recorded.

CHEMICAL ABSTRACTS.

Rôle of carbohydrates and proteins in staling of bread. L. KARÁCSONYI (Z. Unters. Lebensm., 1928, 56, 479–484).—In staling of bread the carbohydrate material soluble in water but insoluble in alcohol decreases, probably owing to alteration of the starch gel. The amount of protein water-soluble material remains unchanged.

W. J. BOYD.

Determination of hydroxymethylfurfuraldehyde in honey and artificial honey. J. FIEHE and W. KORDATZKI (Z. Unters. Lebensm., 1928, 56, 490–492; cf. Troje, B., 1925, 1004; Fiehe, B., 1929, 146).—A solution of 100 g. of the honey in water is treated with zinc acetate and potassium ferrocyanide and filtered. The filtrate is continuously extracted with ether thrice renewed for 12 hrs. and the ether extract is then mixed with an equal volume of light petroleum and some anhydrous sodium sulphate, left for 24 hrs. with occasional shaking, and then filtered. The filtrate is evaporated at a moderate temperature, the residue extracted in 20 c.c. of water, and the solution filtered. 5 c.c. of the solution were titrated with alkaline iodine solution by Troje's method (*loc. cit.*) and 5 c.c. were precipitated with phloroglucinol. The rest of the solution was titrated with alkaline copper solution by Lenk's method (Z. angew. Chem., 1917, 30, 49). It was found that the iodometric method cannot be used for the determination of hydroxymethylfurfuraldehyde in honey, but that the other methods are suitable for this purpose.

W. J. BOYD.

Fluorescence of honey in ultra-violet light. G. ORBÁN and J. STITZ (Z. Unters. Lebensm., 1928, 56, 467–471).—Numerous samples of honey examined all showed luminescence in ultra-violet light depending on the absorption in the ultra-violet region, on the colour of the honey, and on the thickness of the layer of honey. Honey heated to 100° has weaker luminescence while hot, but recovers its original intensity on cooling to 30°. On evaporating some of the water from the honey the increase in luminescence is greater than can be explained by increase in concentration, and is a function of the viscosity. Slightly caramelised honey luminesces more strongly in thin layers, but more faintly in a test-tube than normal honey. Strongly caramelised honey luminesces very faintly in thin layers and becomes brown in colour. It is difficult to distinguish natural from artificial honey by means of its luminescence, but it is at once evident whether a particular kind of honey has been adulterated.

W. J. BOYD.

Detection of maltol and of salicylic acid in presence of maltol. T. MERL and H. BEITTER

(Z. Unters. Lebensm., 1928, 56, 472–474).—Powdered malt coffee (20 g.) is boiled for a short time with 50 c.c. of chloroform and 3 g. of blood charcoal and filtered after cooling. 10 c.c. of the filtrate are shaken with 1–2 c.c. of water and, after separation of the water, filtered into a centrifuge glass. 1 c.c. of a freshly diluted ferric chloride solution (2 drops of a 10% solution in 20 c.c. of water) is added, the mixture emulsified by shaking, and the liquids are separated by centrifuging. The presence of maltol is shown by the characteristic violet coloration of the aqueous layer. Another 10 c.c. portion of the filtrate is tested for salicylic acid if necessary, as follows: After evaporation of the chloroform 5 c.c. of phosphoric acid (*d* 1.7) and 15 c.c. of water are added to the residue and 15 c.c. of the liquid distilled off. The distillate is evaporated to dryness on the water-bath with a few drops of 5% barium hydroxide solution, cooled, and treated with 10 drops of glacial acetic acid and 3 drops of Mandelin reagent (a 5% solution of ammonium vanadate in 95% sulphuric acid). 0.025 mg. of salicylic acid can be detected by the indigo-blue coloration formed.

W. J. BOYD.

Flesh proteins of various animals. K. BECK and E. CASPER (Z. Unters. Lebensm., 1928, 56, 437–457).—Determinations of the nitrogen distribution in various proteins have been carried out by the Van Slyke method. These include edible gelatin (gold leaf), "glutose" obtained from edible gelatin and from Liebig's meat extract by Striegel's method (Chem.-Ztg., 1917, 41, 313), and the coagulated muscle-protein of the ox, calf, pig, sheep, horse, goose, and cod. The extracts of these muscle tissues were examined for content of albumose, glucose, total nitrogen, amino-acid nitrogen, creatinine, ash, and phosphoric acid. The glucose from Liebig's meat extract was found to be similar to that of gelatin. The extract from cod flesh was shown to differ from those from the other varieties of flesh in that higher proportions of albumose and glucose were present.

W. J. BOYD.

Changes in composition during ripening and storage of melons. J. T. ROSA (Hilgardia, 1928, 3, 421–443).—In the late stages of development and the ripening process the total and soluble solids and sugar, and the sp. gr. of the juice increase. Reducing sugars (lævulose and dextrose in approximately equal proportions) decrease; sucrose increases proportionately more rapidly. The total amount of pectic substances is unchanged, but the protopectin decreases rapidly. Fruits picked when immature show on storage an eventual small decrease in sugars. The total amount of pectic substances decreases slightly, and protopectin is changed to pectin. Unripe fruits, when exposed to the action of ethylene, show no increase in sugar content, but an acceleration in the conversion of reducing sugars into sucrose. The action of the ethylene is attributed to an activation of enzymic reactions.

CHEMICAL ABSTRACTS.

Available carbohydrate content of some fruits and vegetables. M. BELL, M. L. LONG, and E. HILL (J. Metabol. Res., 1925–1926, 7–8, 195–197).—Values for fresh and canned products are recorded.

CHEMICAL ABSTRACTS.

Content of alcohol-soluble material in cinnamon. J. PRESCHER (Z. Unters. Lebensm., 1928, 56, 474—478).—Data are given for the alcohol extract, ash and sand contents of various samples of cinnamon. The alcohol extract of Ceylon cinnamon varied from 13.40 to 9.60%, that of cassia cinnamon from 12.63 to 6.55%.

W. J. BOYD.

Food gelatin. BRIEFER and COHEN.—See XV. **Determination of water content of barley.** WINDISCH.—See XVIII. **Disposal of wastes.** MOHLMAN and BECK.—See XXIII.

PATENTS.

Food product and method of making same. S. GELFAND (U.S.P. 1,697,312, 1.1.29. Appl., 19.12.27).—A cultured milk product is emulsified into an emulsion containing oil and yolk, and sufficient of a solution of an edible organic acid is added to stabilise the product and prevent further activity of micro-organisms.

W. J. BOYD.

Treatment of butter, margarine, and similar oil-in-water or water-in-oil emulsions. J. E. NYROP (B.P. 307,167, 26.1.28. Addn. to B.P. 297,256; B., 1928, 825).—Homogenised emulsions of fatty matter containing a relatively small amount of protective substances (e.g., casein) when spray-dried at low temperatures in the absence of oxygen yield a pasty product, which when kneaded with water will absorb it and reproduce a material of butter-like structure and consistency.

E. LEWKOWITSCH.

Treatment of the yolk of eggs. H. BELHOMMET (B.P. 304,902, 10.1.28).—About 4% of sugar or other suitable substance soluble in water is added to the fresh liquid yolk, from which 50% of its weight of water is then evaporated. The dried yolk can be preserved almost indefinitely and reconstituted in the liquid condition by replacing the water. [Stat. ref.] W. J. BOYD.

Preservation of flour, grain, grain products, etc. R. A. LEGENDRE (U.S.P. 1,702,735, 19.2.29. Appl., 22.3.28. Fr., 2.4.27).—The moisture in the product is maintained at about double the normal and its alkalinity is kept within the range pH 7—10.

A. R. POWELL.

Treatment of fruit to prevent decay. W. R. BARGER, Assr. to U.S.A. (U.S.P. 1,704,072—3 and 1,704,456, 5.3.29. Appl., 14.11.27).—Citrus or other fruit is treated with a solution of cadmium sulphate and/or chloride.

Preservation of edible products. A. G. M. STAB-BACK, and STABAVITE SYND., LTD. (B.P. 304,253, 18.7.27 and 13.12.27. Cf. B.P. 212,979; B., 1924, 489).—The products are covered with a thin layer of gum such as gum tragacanth to which glycerin has been added with or without a preservative, e.g., oil of cloves, an alkali nitrate, or a bisulphite. Sulphuric acid or sodium bicarbonate may be added to the gum solution.

W. J. BOYD.

[Apparatus for] pasteurising or sterilising milk or other liquids. N. J. NIELSEN (B.P. 307,854, 12.12.27).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Volumetric determination of santonin in pharmaceutical preparations. A. LANGER (Apoth.-Ztg., 1928, 43, 815; Chem. Zentr., 1928, ii, 1597).—Benzene is preferred to chloroform for extraction after preliminary treatment with light petroleum saturated with santonin. A weighed aliquot portion of the filtrate is evaporated; the residue is dried at 80°, weighed, treated with a solution of santonin in light petroleum, dissolved in warm benzene and 93% alcohol, heated under reflux with 0.05*N*-potassium hydroxide, and titrated back with hydrochloric acid.

A. A. ELDRIDGE.

Colloidal nature of iron scale salts. D. LOESER (J. Amer. Pharm. Assoc., 1929, 18, 124—128).—Experiments on the injection of non-colloidal, partly colloidal, and completely colloidal ferric salts into white rats show that colloidal iron is much less toxic than non-colloidal iron, the toxicity decreasing as the proportion of the salt in the colloidal state increases. Dialysis experiments with a series of iron scale salts indicated that these salts are partly or wholly colloidal in nature, the amount present in the colloidal state depending on the nature and mode of preparation of the salt.

E. H. SHARPLES.

Determination of ascaridole in chenopodium oils. E. KNAFFL-LENZ and A. HOFMANN (Arch. Pharm., 1929, 267, 117—128).—Biological tests on worms, fish, or white mice are unreliable on account of the antagonistic action of other constituents of the oil. The sp. gr. and refractive index may serve as an index of the ascaridole content of oils of similar origin, but this fails when applied to oils of different origins. The titanous reduction method described by Paget (B., 1926, 462) gives reliable results when an arbitrary factor is introduced, the latter depending on the conditions of the experiment and particularly on the amount of hydrochloric acid used. Ascaridole cannot be determined iodometrically. The intensity of the brown coloration produced by the action of hydrochloric acid on a 1% alcoholic solution of the oil is proportional to the ascaridole content, and this forms the best method of procedure, the oil under examination being compared with a standard ascaridole solution. The colour reaction obtained by heating with phenolphthalein (Langer, A., 1921, i, 259) gives satisfactory results with oils containing above 60% of ascaridole, but fails with oils of poor quality; the same remarks apply also to similar reactions with dimethylaminobenzaldehyde or diphenylamine.

S. COFFEY.

Bamba oil and the terpenes and higher-boiling fractions of cajuput oil. D. B. SPOELSTRA (Rec. trav. Chim., 1929, 48, 372—376).—A specimen of bamba oil, d_{4}^{20} 0.9326, n_D^{20} 1.4816, $\alpha_D +33.6^\circ$, saponification value 1.8, ester value 3.0, was found to contain *d*- α -pinene (47%), *d*-limonene and dipentene (5%), *d*- α -terpineol (15%), dillapiol (24%), and a small amount of a solid, m.p. 113—118°. *l*- α -Pinene, *l*-limonene, and dipentene were isolated from the lower-boiling fraction of cajuput oil. The higher-boiling fractions of the original oil gave, after hydrolysis with 10% alcoholic potassium hydroxide solution, *i*- α -terpineol, a sesqui-

terpene fraction, b.p. 131—138°/22 mm., $d_4^{17.5}$ 0.9054, $n_D^{20.7}$ 1.5023, $\alpha_D +4.3^\circ$, consisting of a mixture of mono- and di-cyclic hydrocarbons which when dehydrogenated with sulphur affords azulene and cadalene. A sesquiterpene alcohol fraction, b.p. 160—165°/20 mm., $d_4^{15.0}$ 0.9758, $n_D^{17.5}$ 1.5086, was also obtained. This has the composition $C_{15}H_{26}O$, but when it is dehydrated by heating with formic acid a mixture of mono- and di-cyclic hydrocarbons, $C_{15}H_{24}$, b.p. 134—139°/16 mm., d_4^{15} 0.9186, n_D^{20} 1.5126, results. Dehydrogenation of this mixture with sulphur yields cadalene. H. BURTON.

Finnish turpentine. ASCHAN.—See XIII.

PATENTS.

Manufacture of derivatives of hydroxy-compounds containing mercury in the nucleus. A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 307,532, 8.12.27).—Phenols are mercurated in presence of thiocyanates, ferrocyanides, or ferricyanides, or mercurated phenols are treated with these salts. The products, $OH \cdot Ar \cdot Hg \cdot X$, in which $X = SCN$ or $\frac{1}{2}$ or $\frac{1}{3}Fe(CN)_6$, are bactericides and fungicides.

C. HOLLINS.

Manufacture of pharmaceutical products [salts of heterocyclic bases]. I. G. FARBEIND. A.-G. (B.P. 298,240, 5.10.28. Ger., 6.10.27).—The salts of heterocyclic bases with carboxylic acids derived from hydroxylated diaryl ketones are almost tasteless, non-irritant, and sufficiently soluble in 30% alcohol. Examples are 3-phenyldihydroquinazoline 2-p-hydroxybenzoylbenzoate, m.p. 187—190° (+EtOH), o-2':4'-dihydroxybenzoylbenzoate, m.p. 119°, and o-4'-hydroxy-naphthoylbenzoate, m.p. 120°.

C. HOLLINS.

Manufacture of benziminazolonearsinic acids. I. G. FARBEIND. A.-G. (B.P. 281,703, 2.12.27. Addn. to B.P. 256,243; B., 1927, 670).—The process of the prior patent is extended to the preparation of arsinic acids by the diazo reaction from 5(6)-aminobenziminazolones carrying an alkyl, alkylenyl, or aralkyl group in position 1. 1-Methylbenziminazolone-5-arsinic acid, the 1-ethyl compound, and the intermediate 2:4-dinitroethylaniline, m.p. 119°, 4-nitro-2-aminoethylaniline, m.p. 138—140°, and 5-nitro-1-ethylbenziminazolone, are described.

C. HOLLINS.

Preparation of a therapeutic product. C. JAEGER (B.P. 290,195, 18.4.28. Switz., 9.5.27).—A therapeutic product having properties similar to those of cod-liver oil and exceptionally rich in vitamins is prepared by exposing dried banana meal in thin layers to ultraviolet light.

E. LEWKOWITSCH.

Manufacture of *Raphanus* preparations for medical use. CHEM. FABR. VORM. SANDOZ (B.P. 292,984, 27.6.28. Ger., 28.6.27. Addn. to B.P. 235,883; B., 1927, 268).—The comminuted roots of *R. sativus* (radish) are treated with adsorptive charcoal and dried as described in the prior patent.

L. A. COLES.

Manufacture of the hormone of the sexual organs. SCHERING-KAHLBAUM A.-G. (B.P. 285,402, 6.1.28. Ger., 15.2.27. Addn. to B.P. 265,166; B., 1928, 348).—Material extracted from fresh sexual organs

as described in the prior patent is suspended in aqueous alkaline solution and extracted with ether; the ether residue is then extracted with cold acetone or with methyl alcohol, and the residual lipoids are hydrolysed after removal of the solvent.

L. A. COLES.

Optic nerve and retina extract. E. H. STUART, Assr. to E. LILLY & Co. (U.S.P. 1,700,691, 29.1.29. Appl., 8.3.27).—The optic nerves and/or retinas of animals are extracted with cold and then repeatedly with hot alcohol, the extracts mixed, and the alcohol is evaporated. The residue may be dissolved in water.

B. FULLMAN.

Manufacture of alkamine esters of [N-substituted] o-aminobenzoic acids. O. EISLEB, Assr. to WINTHROP CHEM. Co. (U.S.P. 1,704,660, 5.3.29. Appl., 11.10.26. Ger., 28.10.25).—See B.P. 260,605; B., 1928, 107.

Manufacture of *i*-menthol. K. SCHÖLLKOPF, Assr. to RHEINISCHE KAMPFER-FABR. G.M.B.H. (U.S.P. 1,704,630, 5.3.29. Appl., 26.6.26. Ger., 23.11.21).—See B.P. 189,450; B., 1923, 743 A.

Production of 1-propenyl-3-ethoxy-4-hydroxybenzene [alkyl ethers of protocatechuic aldehyde]. F. BOEDECKER (U.S.P. 1,704,494, 5.3.29. Appl., 19.11.27. Ger., 14.2.27).—See B.P. 284,199; B., 1928, 547.

Process for arsenating organic compounds. E. SCHELLER, Assr. to DEUTS. GOLD- & SILBER-SCHNEIDANSTALT VORM. ROESSLER (U.S.P. 1,704,106, 5.3.29. Appl., 8.11.26. Ger., 15.11.24).—See B.P. 261,026; B., 1928, 140.

Moth-proofing substance (U.S.P. 1,694,219).—See V.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitivity. I. Effect of heat on sensitivity curve of photographic plates. II. Sensitivity of photographic plates at various temperatures. O. MASAKI (Mem. Coll. Sci. Kyōtō, 1929, A, 12, 1—12, 13—22. Cf. A., 1924, ii, 807; Kellner, B., 1926, 387).—I. The effect of a preliminary heating for 1 hr. at 80° on the sensitivity of photographic plates to various wave-lengths has been investigated. With panchromatic and other slow plates, the sensitivity, as measured by the inertia, is increased for all wave-lengths, the effect near a maximum point on the sensitivity-wave-length curve being greater on the side corresponding with longer wave-lengths, i.e., the maximum point is shifted towards the red. The sensitivity of fast plates shows the same shift, but is usually diminished by heating. With all plates and for all wave-lengths the contrast is increased by heating, and for panchromatic and orthochromatic plates the increase is greatest for long wave-lengths. Plates which have been heated gradually regain their normal sensitivity on keeping at the ordinary temperature. Drying the plate has very little effect on its sensitivity.

II. The relation between the temperature of preliminary heating, t , and the inertia and contrast, γ , for a given plate and a given wave-length has also been

investigated. The inertia varies most between about 30° and 70° , whilst γ and t are connected by the empirical expression $\gamma = e^{at^2 + k}$, where a and k are constants depending on the wave-length of the light to which the plate is exposed, on the time of development, and on the properties of the emulsion. If D_m is the maximum density at 0° , E the length of exposure, and D the density of the developed plate, it is further found empirically that $D = D_m e^{aE^2} \{1 - e^{-(bt^2 + c)E}\}$, where b and c are constants of the same nature as a , b being negative for some rapid plates and positive for other plates. This formula is valid from 10° to 80° . R. CUTHILL.

Sensitisation of photographic emulsions by colloidal materials. B. H. CARROLL and D. HUBBARD (Bur. Stand. J. Res., 1928, 1, 565—588).—Photographic sensitivity is greatly increased by the presence of ultra-microscopic particles on the silver halide grains of an emulsion, and various theoretical explanations are discussed. Emulsions, after ripening and washing, were treated with gelatin-protected sols of silver iodide, silver sulphide, metallic silver, and metallic gold with the object of introducing sensitivity nuclei. Gelatin emulsions with excess of silver salts may be developed normally with certain precautions. The grains carry a positive charge, and are particularly adapted to sensitisation by the negatively charged metallic colloids. All the colloids retarded after-ripening to such an extent that the practical effect was desensitisation. Colloidal silver iodide produced a marked increase in contrast with chemical development, and an increase in speed with physical development. These effects are attributed to increased absorption of the developer. Colloidal gold and silver increased the speed of emulsions with excess of silver. The results are explained in terms of orientation of the photolysis at the sensitivity nuclei formed by the colloidal particle.

C. J. SMITHELLS.

PATENTS.

Photographic film. H. BRADSHAW, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,703,470, 26.2.29. Appl., 4.3.27).—The rear side of the cellulose film base is coated with a layer of a cellulose carbamate.

J. W. GLASSETT.

Photographic etching and light-sensitive resist therefor. A. B. DAVIS, Assr. to KEYSTONE WATCH CASE Co. (U.S.P. 1,703,512—3, 26.2.29. Appl., [A] 30.10.26, [B] 15.11.28).—The resist contains (A) a protein (e.g., glue) and (B) an amino-compound, together with an aliphatic monohydroxy-aldehyde, e.g., aldol, and preferably dichromate.

F. G. CLARKE.

Colour photography and cinematography. L. DUFAY (B.P. 307,437, 7.9.27).—A negative taken through a green, violet, and orange network screen is printed on to a sensitised support associated with a screen coloured yellow, blue, and red, the printing taking place through both screens without necessity for the registration of the patterns. The transmission properties of the coloured elements of the positive are so arranged that each element will transmit the light from two of the elements of the green, violet, and orange screen, but will stop the light from the third, namely, that approximating to its complementary colour.

J. W. GLASSETT.

Reproduction of original cinematograph colour record positive films having a support goffered in lenticular elements. Soc. FRANÇ. DE CINÉMATOGRAPHIE ET DE PHOTOGRAPHIE FILMS EN COULEURS KELLER-DORIAN (B.P. 285,035, 8.2.26. Fr., 9.2.27).—The contrast of the original positive film is lowered by converting the developed silver either into silver chloride, bromide, or iodide by means of a suitable halogenising bath, or into a blue-toned image by means of a mordant dyeing process. In this manner it is possible to counter-balance the increase in contrast which normally takes place during the reproduction of such films when using undiffused light.

J. W. GLASSETT.

Reversal process for developing photographic silver sensitised films. I. G. FARBENIND. A.-G. (B.P. 287,542, 16.1.28. Ger., 25.3.27).—The exposed film is given a short development to ensure that ample silver halide remains for the formation of a strong positive, is then reversed in the usual manner by bleaching and re-development, and finally the image is brought to normal density by treatment with a uniformly acting reducing solution. The following solutions, which are recommended for the reducing process, may also be used for bleaching the negative silver. (A) Iodine 1 g., potassium iodide 2 g., water 200 c.c.; (B) thiocarbamide 4 g., water 100 c.c.; 8 pts. of A are used to 100 of B.

J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Chemical and ballistic stabilities of BAm and BD powders. I. ANON. II—IV. DESMAROUX. V. MARQUEYROL (Mém. Poudres, 1928, 23, 79—112, 113—115, 116—123, 124—127, 128—146).—I. The relative advantages of amyl alcohol and diphenylamine as stabilisers for B powder were investigated on three lots of BM powder (French naval nitrocellulose powder) that had been made from the same nitrocotton. One lot (A) was stabilised with 8% of amyl alcohol and two lots (B and C) with 2% of diphenylamine. All these were steeped in cold water for 24 hrs., whilst B had also four steepings of 8 hrs. each at 80° , and C four of 24 hrs. each at 50° . The powders were then placed in cartridge cylinders and subjected to hot dry storage at 110° and hot moist storage at 60° and 75° . Samples were withdrawn at intervals and tested for colour, flexibility, transparency, loss of weight, gas formation, residual velocity, and vivacity. After 175 and 185 days' treatment ballistic tests were carried out on these powders in the 16 cm. gun M.93/96, and the results compared with those obtained from the unheated powder. Samples B and C showed a slight increase in velocity, pressure, and regularity, which was more marked in C than in B, while A had lost about 10% of its velocity and 20% of its pressure. In these tests the weights taken of the treated and untreated powders were equal, but if the results from the treated powders are calculated to an equal weight of the powders before treatment, A, B, and C would all show a slight decrease in ballistic properties.

II—IV. Samples of gas withdrawn from the cylinders after 85 days' heating at 75° were analysed without giving any positive evidence that nitric oxide had

been formed. When samples of A and B were heated in flasks, 0.7% NO was found in the gas from A and 3% from B.

V. Samples of A, B, and C were heated in wide-mouthed bottles, both open and corked, at 75°, both in dry and moist atmospheres. The evolved gases showed that diphenylamine and nitrosodiphenylamine are the best stabilisers at present available for powders with low content of solvent. In thick powders the superiority of diphenylamine over amyl alcohol is not so marked. This fact may be attributed to the action of the solvent.

S. BINNING.

Transformation products formed from centralite during storage of SD powder. H. LÉCORCHÉ and P. L. JOVINET (Mém. Poudres, 1928, 23, 147—154).—The products either not volatile in steam or rendered non-volatile during dissolution of the powder were examined. The only example of the former class present was a mononitro-derivative of centralite (diphenyldiethylcarbamide), whilst of the second class *p*-nitrophenylethyl-nitrosoamine was detected. The acids formed during the decomposition of the powders are neutralised by bases resulting from the hydrolysis of centralite, which might be effective in nitroglycerin powders, where even feebly basic stabilisers are excluded, if it were not for the fact that apparently it volatilises along with the nitroglycerin.

S. BINNING.

Thermodynamic treatment of explosive reactions. II. A. SCHMIDT (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 90—93).—Considerations previously advanced (B., 1929, 303) are now extended to explosives the oxygen content of which is sufficient for complete gasification, but not for complete combustion to carbon dioxide and water. Nitrocellulose is taken as an example of this class, and it is shown that the composition of its explosion gases is determined, for all practical purposes, by the water-gas reaction, and is independent of the charge-density. The same holds true for the heat of explosion and the explosion temperature. The heat of explosion as calculated by the thermodynamic method is slightly lower than that obtained by calorimetry in an explosion bomb.

S. BINNING.

Vapour pressure of nitroglycerin and nitroglycol. P. NAOUM and K. F. MEYER (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 88—90).—Determinations by a static method gave at 20° vapour pressures of 0.009 and 0.30 mm. Hg for nitroglycerin and nitroglycol respectively. By an aspiration method similar to that used by Marshall and Peace (cf. J.S.C.I., 1916, 35, 298) the vapour pressures at 20° and 35° were 0.011 and 0.036 for nitroglycerin and 0.035 and 0.115 for nitroglycol. The lower values for nitroglycerin obtained by Marshall and Peace are attributed to excessively rapid aspiration. The difference in volatility between nitroglycol and nitroglycerin is greater than would be expected from their relative vapour pressures.

S. BINNING.

Ignition of fire damp by explosives. A. SEGAY (Compt. rend., 1929, 188, 867—868).—The addition of about 25% of salt to amatol lowers the temperature and luminosity of the flame produced on explosion, but since the amount of heat liberated is unaffected it is

assumed that the duration of the flame is increased. The presence in the flame of gases produced as a result of complete detonation (carbon dioxide, nitrogen, water vapour) also inhibits inflammation of fire damp.

J. GRANT.

PATENT.

Manufacture of [book] matches. S. and H. RAKOWITZKY (GEBR. S. & H. RAKOWITZKY) (B.P. 288,620, 8.12.27. Austr., 15.4.27).

XXIII.—SANITATION; WATER PURIFICATION.

Sludge digestion and p_H control. H. W. CLARK and G. O. ADAMS (Ind. Eng. Chem., 1929, 21, 258—260).

—Laboratory-scale experiments on the digestion of Lawrence sludge showed that the addition of sodium nitrate in amounts equivalent to 2.5 pts. of nitrogen per 100,000 reduced the time required to obtain complete digestion. The volume of gas obtained per gram was also reduced while the proportion of nitrogen therein was above normal owing to reduction of the nitrate. An increase in the hardness of sewage and sludge was also found to reduce the digestion period. Gas production reached a maximum when small amounts of acetic acid and 1% of a strong sulphite-paper waste liquor were added, these being the only experiments in which more gas was obtained than from the control. Addition of calcium carbonate had very little effect on gas production, whilst the addition of 1% of strong wool-scouring liquor definitely retarded it. Other sludges were found to differ very considerably with regard to the digestion process, chemical composition being an important factor. In some cases addition of calcium carbonate was necessary before fermentation would proceed at all rapidly, whilst in others it was unnecessary. Apparently some sludges have more available alkalinity than others, though there is always a tendency to accumulate calcium carbonate as digestion proceeds owing to the gasification of the organic matter. Generally speaking, calcium carbonate is unsuitable as an agent for p_H control owing to its slight solubility being insufficient to offset the acids produced by the fermentation process. The p_H value of digesting sludge may be rapidly raised by removing carbon dioxide by aeration.

C. JEPSON.

Effect of pressure on sludge digestion. M. C. WHIPPLE, G. M. FAIR, and L. KLEIN (Ind. Eng. Chem., 1929, 21, 254—256).—A 2:1 mixture of fresh sewage solids and Imhoff tank sludge incubated at 20° under pressures corresponding to 0.6, 1.0, and 1.8 atm. showed very little difference in the time required to obtain complete digestion, though the rate of fermentation in the early stages showed a slight advantage for diminished pressure. The rate of methane production at increased pressure was reduced at first, possibly due to the presence of entrained gases, but there would not appear to be any evidence that deep tanks and the consequent increased pressures would have disadvantages outweighing their advantages with regard to economy of space etc.

C. JEPSON.

Complete sludge-washing plant in a single unit. A. ANABLE (Ind. Eng. Chem., 1929, 21, 223—226).—When economy of floor space is the governing factor, the

standard five-thickener counter-current decantation plant with a washing efficiency of 99% may be replaced by a single multiple-storey tank with a washing efficiency of 97.7%. This unit consists of four superimposed trays, each section being fitted with a Dorr thickener and connected to the one immediately below by a central, sealed opening which permits downward passage of sludge, but prevents the mixing of the wash-waters in the various compartments. The clean wash-water is introduced into the bottom chamber and the sludge to be washed into the top one; the washed sludge is discharged at the bottom and the strong liquor flows over a peripheral sill at the top.

C. JEPSON.

Disposal of industrial wastes. F. W. MOHLMAN and A. J. BECK (Ind. Eng. Chem., 1929, 21, 205—210).—The wastes from a Chicago maize products factory had a biochemical oxygen demand equivalent to the normal requirements of a population of 350,000. By removal of suspended solids the demand was reduced to a population equivalent of 250,000. The chief waste responsible for this demand was water from the steeping of grain prior to grinding, and as a loss equivalent to 2% of the ground maize was incurred it was considered economical to recover. The steep water was re-circulated until it contained 7% of dissolved solids and was then further concentrated in vacuum pans to a syrupy liquid containing 45%, in which form it could be worked up with other products as a stock food. The condensate from the vacuum pans had a population equivalent of 70,000, but as 80% of the oxygen-consuming substances are contained in the first 15% of distillate, it is hoped shortly to reduce the demand by 50% or more. In another instance the wastes from an oil and paint works seriously interfered with the operation of the Calumet sewage disposal plant, the average daily flow of 1.25 million gallons being approximately 3% of the normal sewage flow. The chief source of trouble was an intermittent discharge of sulphuric acid (5%), but others included copper and arsenic from the manufacture of Paris-green and sodium sulphite from the manufacture of β -naphthol. It has been decided to reduce the effect of the acid by installing a balancing tank and to spread the discharge evenly over the 24 hours, to remove the copper, which has a deleterious effect on nitrification, by means of iron filings with recovery of copper, and to keep out the sulphite liquor and use it as a neutralising agent, discharging the sulphur dioxide through a high stack. It is hoped to reduce the volume to be dealt with to 60,000 gals. per day.

C. JEPSON.

Treatment of industrial wastes from paper mills, [wool scouring,] and tannery on Neponset River. A. L. FALES (Ind. Eng. Chem., 1929, 21, 216—221).—Two paper-making plants and a wool-scouring and tannery plant discharge their waste waters into the river Neponset. In winter and spring the effluents are discharged after partial purification, but in summer and autumn full treatment is insufficient to keep the river free from putrefaction, and sodium nitrate solution is added when required to prevent this condition. The full treatment at the paper-making plants consists of chemical precipitation followed by filtration, whilst at

the other plant the wool-scouring liquor is cracked with acid to separate the grease, and the aqueous layer resulting therefrom after mixing with the tannery waste is coagulated with aluminium sulphate and dealt with on sand filters.

C. JEPSON.

Chemical treatment of [wool] trade waste. F. D. SNELL (Ind. Eng. Chem., 1929, 21, 210—213).—Experimental work indicates that, after removal of grit by settlement for an hour, coagulation by means of aluminium sulphate is a more effective method of dealing with wool-scouring waste than the more common one of cracking with sulphuric acid. The effluents thus obtained may be discharged direct to the stream without serious nuisance, and the fat recovered from the sludge by acidification of the cake obtained from either a plate-and-frame press or a vacuum filter. The aluminium thus redissolved may be re-used as a coagulant after neutralisation. The minimum amount of coagulant required to give a satisfactory effluent is 80 lb. per 1000 gals., but anything up to twice this quantity may be required to get a good press-cake for the recovery of fat which may be expected to have a value equivalent to the cost of removal.

C. JEPSON.

Chemical and biological correlations in a polluted stream. W. RUDOLFS (Ind. Eng. Chem., 1929, 21, 256—258).—A direct relation was found in the Raritan river between the biochemical oxygen demand, the numbers of bacteria and plankton, and the ammoniacal nitrogen content, each increasing in rough proportion until tidal water was reached, when all decreased. The rate of oxygen depletion was greater in summer than in winter, though during the year under review no absolute depletion was recorded. The rate of flow during the year was 40% in excess of the average for the preceding five years, and oxygen saturation depends very largely on the incidence of rainfall.

C. JEPSON.

Sterilisation of water by chlorine. F. DIENERT and P. ETRILLARD (Compt. rend., 1929, 188, 826—829).—In a review of recent experiments no evidence is found for the theory that the bactericidal action of chlorine is due to the emission of "abiotic" rays when that element combines with organic matter present in the water. In one case chlorine was added to river water sterilised by means of a Chamberland filter; when sufficient hyposulphite was added to destroy free chlorine the water had no harmful effect on *B. coli*.

G. A. C. GOUGH.

Bacteriological investigation into the state of pollution of the Clyde at Port Glasgow, Greenock, and Gourock. D. ELLIS (J. Roy. Tech. Coll., Glasgow, 1929, 2, 129—142).

PATENT.

Removing oil from sewage or sludge. K. IMHOFF (U.S.P. 1,703,041, 19.2.29. Appl., 18.6.27. Ger., 2.12.26).—The sewage or sludge is passed through a tank fitted at the bottom with means for supplying compressed air. The floating oil is collected in lateral channels separated from the main tank by floating beams which have overflow apertures communicating with the sewage level.

C. JEPSON.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 24, 1929.

I.—GENERAL; PLANT; MACHINERY.

Heat-conduction problems. E. GRIFFITHS (Proc. Physical Soc., 1929, 41, 151—179).—For the purpose of thermal conductivity measurements, materials may be divided into four classes: (1) those of low thermal conductivity, such as cold-storage insulators; (2) those in the form of thin sheets, and those employed in building construction; (3) refractories and materials employed in furnace construction; (4) pure metals and alloys. In testing materials of low thermal conductivity (baked slab cork can be obtained with $K = 0.00007$ c.g.s. unit) attention has to be given to heat leakage from corners and edges if the hot-plate method is used. The simplest procedure is to eliminate the effect by the use of a guard-plate. Substances in the form of thin discs can be tested by the divided-bar method, the correction for the thermal resistivity of the two mercury films being obtained by independent experiments using a thin disc of iron instead of the specimen. For the study of refractories a furnace is made up of "silit" rod heaters which can raise one face of the slab under test to any temperature up to 1000° . On the top of the slab is a water-flow calorimeter fitted with a guard-ring. For the study of metals and alloys the guard-tube method is recommended. The space between the guard-tube and specimen is packed with a powder of low thermal conductivity. In two groups of alloys, aluminium alloys and bronzes, the values of the Lorentz coefficient approximate to the values for the pure metals which form the principal constituents of the alloys. W. E. DOWNEY.

Pyrometer for measuring temperatures by means of a colour charge. G. NAESER (Stahl u. Eisen, 1929, 49, 464—466).—The pyrometer consists of two superimposed wedges filled with gelatin coloured red and green respectively, and fixed in a frame the edges of which are graduated in degrees. Along these edges slides an eyepiece through which the light from an object is observed after passing through the wedges. The eyepiece is moved along the scale until a neutral tint, neither red nor green, is observed. The pyrometer is accurate to $\pm 13^{\circ}$ between 900° and 2000° .

A. R. POWELL.

Purification of gases. WASMUHT.—See VII.

PATENTS.

Furnace. J. F. BAKER, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. CO. (U.S.P. 1,705,704, 19.3.29. Appl., 27.7.26).—A continuously driven conveyor has a number of tiltable trays for receiving material which it moves through the annular heating chamber of a heat-treating furnace. The trays are periodically and successively caused, by the rotation of the conveyor, to

engage with a ramp so that they tilt and discharge their contents. J. S. G. THOMAS.

Dryer. R. RANSON, Assr. to FORD INSTRUMENT CO., INC. (U.S.P. 1,703,635, 26.2.29. Appl., 31.8.21).—The dryer consists of a rotary drum and a portable furnace, both having similarly shaped co-acting ends; the drum has a drying chamber, and the internal bore of the furnace is less in cross-sectional area than that of the chamber. Means are provided for removing the products of combustion. H. ROYAL-DAWSON.

Grinding, crushing, pulverising, mixing, and separating machine. D. J. REES (U.S.P. 1,706,254, 19.3.29. Appl., 6.1.27. U.K., 20.1.26).—See B.P. 264,324; B., 1927, 207.

Means for continuous drying or distillation of fine granular masses. L. HONIGMANN (U.S.P. 1,704,796, 12.3.29. Appl., 20.4.27. Ger., 5.11.25).—See B.P. 293,147; B., 1928, 658.

Separation of materials by [vacuum] flotation. F. E. ELMORE (U.S.P. 1,706,281, 19.3.29. Appl., 23.4.27. U.K., 16.7.26).—See B.P. 275,778; B., 1927, 800.

[Rotary drum] filter. H. NOTZ, Assr. to MASCHINEN-FABR. BUCKAU R. WOLF A.-G. (U.S.P. 1,705,226, 12.3.29. Appl., 8.3.28. Ger., 25.7.25).—See B.P. 301,541; B., 1929, 116.

Refrigerating apparatus of the absorption type. N. V. KODOWA REFRIGERATOR CO., and W. A. SLAGER (B.P. 308,898, 31.3.28).

Absorption refrigerating apparatus. Rectification of gases, vapours, or mixtures thereof in absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAGET (B.P. 282,770—1, 23.12.27. Ger., [A] 31.12.26, [B] 28.12.26).

[Floating cover-plate for] filter presses. J. TETLEY & SON, LTD., and G. COOPER (B.P. 308,901, 3.4.28).

Photochemical gas reactions (B.P. 307,521 and 307,406).—See II.

II.—FUEL; GAS; TAR; MINERAL OILS.

Sp. gr. of Alabama coals. B. W. GANDRUD and S. A. BRITTON (Alabama State Mine Exp. Stat. Bull. No. 2, 1928, 57 pp.).—Samples of Alabama coal were taken from 19 mines of the Warrior and Cahaba fields. These coals on analysis had d 1.26—1.37, ash 2—15%, moisture 2—4.5%. Ordinarily the sp. gr. increases uniformly with the percentage of ash; the discrepancies observed may be due to change in the character of the ash-forming material, to variation in the volatile matter content, or to their combined effect. In those cases

where the seams consist of benches of coal separated by partings of shale etc. of high ash content, crushing will be necessary to eliminate the ash-forming impurities, and the resulting finer sizes of coal will furnish the most difficult problem in effecting improvements in the washing of Alabama coal.

C. B. MARSON.

Microscopical examination of fine coal-cleaning products by the method of relief-polishing. E. STACH and F. L. KUHLEWITZ (Glückauf, 1928, 64, No. 25; Fuel, 1929, 8, 191—198).—The products obtained in the cleaning of fine coal have been examined microscopically by embedding the material in Schneiderhöhn's resin mixture (1 pt. of Venetian turpentine, 3 pts. of dammar resin, 2 pts. of shellac), which is then cast into small blocks, ground, and relief-polished. The characteristic appearance of the different constituents, vitrain, durain, fusain, and shale, is illustrated by photomicrographs. They can be recognised at once and their respective amounts determined. A. B. MANNING.

Properties of coking coals and their behaviour on coking. P. DAMM (Glückauf, 1928, 64, 1073—1080, 1105—1111; Fuel, 1929, 8, 163—177).—Six Silesian and Westphalian coals have been studied. The caking index (Meurice) increased with increasing oil bitumen content (Fischer, B., 1925, 656). More difficulty was experienced in correlating the solid bitumen content with the behaviour of the coals on carbonisation. The swelling and the swelling pressure, i.e., the pressure just necessary to prevent swelling during carbonisation, have been determined in an apparatus similar to that used by Korten (B., 1920, 713 A); the fusion temperatures and yields of volatile matter before, during, and after the plastic period, respectively, have also been determined. With a high initial yield of volatile products from the coal there is risk of a premature loss of part of the bitumen essential to coking. The yield of distillation products during the plastic period gives some indication of the swelling pressure. The yield during the final distillation has an important bearing on the quality of the coke produced; the greater the yield, the more fissured is the coke. On the other hand, a low yield results in a small contraction and causes the coke to adhere to the walls. The mechanism of coking is discussed in the light of the experimental results. The reactivity of the coke increased with diminution in the caking properties of the coal. A. B. MANNING.

Swelling and expansive force of coals. P. DAMM (Brennstoff-Chem., 1929, 10, 65—67).—It is important to differentiate between the swelling of a coal, which is its increase in volume when heated so that it is free to expand, and the expansive force, which is the force it exerts when heated at constant volume. The former depends on the degree of softening of the coal and on the quantity of volatile matter evolved during plasticity, whereas the true cause of the latter is uncertain, although the coal bitumen plays a part. The "sticking" of a fully carbonised charge in a coke oven may be favoured by the exertion of a high expansive force by the parent coal, but is primarily caused by a relatively small liberation of volatile matter, and hence only a slight contraction of the coke results during the later stages of carbonisation.

W. T. K. BRAUNHOLTZ.

Reducing action of various coals in an aqueous medium. V. S. KRYM and S. J. PANTSCHENKO (Brennstoff-Chem., 1929, 10, 63—65). G. STADNIKOV and N. PROSKURNINA (*Ibid.*, 65).—The diminution in concentration of aqueous solutions of ferric chloride in contact with coals or cokes, observed by Stadnikov and Proskurnina (cf. B., 1926, 729; 1927, 864), is not due to adsorption of ferric hydroxide, but to the reducing action of the fuel. The latter depends on such factors as concentration of the ferric chloride solution, time of reaction, particle size of the fuel, etc.

Stadnikov and Proskurnina suggest that the presence of bivalent iron in the solutions is due to the solvent action of hydrochloric acid on the sample and to base exchange between ferric chloride and ferrous compounds in the fuel.

In reply, the authors point out that the quantity of bivalent iron present may greatly exceed that of the total iron in the coal. The reducing action of coke is ascribed to the liberation of hydrogen sulphide in acid medium.

W. T. K. BRAUNHOLTZ.

Determination of degree of decomposition of peat. Determination of cellulose. W. I. KOMAROVSKY (Z. angew. Chem., 1929, 42, 336—338).—Cellulose is the only plant component found unaltered in peat, and the degree of decomposition which the cellulose has undergone is taken as a measure of the age of the peat. Dried and powdered peat was successively extracted with ether, alcohol-benzene mixture, ether again, and twice treated with 1% soda solution in a centrifugal. It was then repeatedly extracted with sodium sulphite and chlorine dioxide solution. The crude cellulose remaining was dissolved in Schweizer's reagent and precipitated with acetic acid and alcohol. The weight of pure cellulose can also be determined by difference, the weight of the insoluble residue being known. A series of tests show the decrease of cellulose content with increasing depth of origin of the peat. At 2 m. it does not exceed 8—9%. Values are lower than those obtained with Cross and Bevan's method, in which, nevertheless, it is considered that some cellulose is destroyed by the use of chlorine.

C. IRWIN.

Developments in gasworks carbonising plant, with special reference to refractory materials. G. M. GILL (Trans. Ceram. Soc., 1929, 28, 7—17).—Recent developments in carbonising plant in America, Germany, and England are briefly discussed. A description is given of intermittent vertical ovens and their advantages are outlined. Hot gases from producer-gas burning in the lower section of these ovens pass upward, round the ovens, descend through recuperators, and finally through a waste-heat boiler. Good silica material is necessary for the walls of the ovens and retorts and for the combustion chambers. For the producer linings the toughest hard-burnt firebricks are essential. Vertical installations lend themselves readily to thermal insulation. Tunnel kilns for burning silica refractories are advocated. Much loss would be avoided if the shapes of the refractories were properly standardised.

F. SALT.

[Control of] water-gas manufacture. W. SCHWEDER (Gas- u. Wasserfach, 1929, 72, 261—263).—On account

of contamination of crude water-gas by steam and by coke particles, and of the big fluctuations in make etc., careful analysis of the gas at different stages is difficult without dislocation of routine operation. An apparatus is described to meet these difficulties, which consists of a battery of burettes fitted with cooling jackets and connected to each other and to an analysis apparatus by suitable taps. The burettes are calibrated from 0–25 c.c. in 0.1 c.c., and from 25 to 100 c.c. in 0.5 c.c., and they are filled in turn with gas over known time intervals. The accumulated samples can then be analysed, and in this way it is particularly easy to follow the formation of carbon dioxide, even at 10 sec. intervals. The results of two short tests on a water-gas plant are given, showing a gas-making efficiency of 62 and 67% respectively; the former gave a thermal efficiency of 76%.

R. H. GRIFFITH.

Synthesis of higher hydrocarbons from water-gas. B. A. BUYLLA and J. M. PERTIERRA (*Anal. Fis. Quím. [Tecn.]*, 1929, 27, 23–38).—The low temperature of 254° has been maintained during the catalytic reduction by copper, cobalt, and manganous oxides of the carbon monoxide present in water-gas. Although the contraction is less at this temperature, a greater yield is obtained of liquid and solid hydrocarbons; thus by one passage over the catalyst 37.6 g. of organic products have been obtained from 1 m.³ of gas. The necessity of purifying the gas, in order that the catalyst may maintain its activity, has been demonstrated. H. F. GILLBE.

Commercial possibilities in the use of synthetic hydrocarbon processes in the gas industry. W. W. ODELL (*Fuel*, 1929, 8, 178–187).—The possibility of carburetting water-gas economically by means of hydrocarbons prepared synthetically from the water-gas itself is discussed as far as the available data permit. A desirable operating procedure for a gas plant would include the production of enriched water-gas in amounts equal to the demand for gas, using the off-peak water-gas for the production of condensable hydrocarbons, which can be used as solvents or motor fuel, or stored for future use in enriching gas without the deposition of carbon. Accurate estimates of cost of operation, equipment, etc. cannot yet be made, and the need for further investigation is emphasised. Data are required on such problems as the removal of traces of sulphur from water-gas, the production of suitable catalysts on a commercial basis, design of full-scale reaction chambers, etc.

A. B. MANNING.

Recovery of phenol from coke-oven gas liquor. P. HOENING (*Z. angew. Chem.*, 1929, 42, 325–331).—Recovery of phenols from the gas liquor produced at a group of coke-oven plants in the Ruhr has been introduced, primarily owing to complaints arising from the disposal of the effluents. The liquors containing originally 2–3 g./litre of phenol are reduced to a content of 0.6–0.8 g./litre, which is considered the economic limit. The liquor is washed with benzol before distillation for ammonia, and the benzol freed from phenols by distillation or alkali treatment, the latter being preferred. Several methods of washing are in use. A preliminary wash for tarry matters is used, and the main washing is carried out by counter-current at 65°. The phenols

from which benzol has been removed by distillation contain 65–75% of tar acids and 4–5% of pyridine, the remainder being hydrocarbons of high b.p. A further washing of the water with a lignite wash oil to remove benzol entrained with the water has been added. If the alkali process is used, two washers are required which are worked in series and fed with 30–35% caustic soda; continuous working is not practicable. The benzol used for washing has b.p. 85–120°. Details are given of variation in the degree of stripping with proportion of benzol used, temperature, and type of tower packing. With alkali washing, oils of higher b.p. can be used instead of benzol, provided they do not emulsify with water, a more complete extraction is possible, and steam consumption is less. Costs of working are discussed.

C. IRWIN.

Aluminium chamber oven for the [laboratory] low-temperature carbonisation of bituminous material. A. WEINDEL (*Brennstoff-Chem.*, 1929, 10, 67–69).—The apparatus comprises a horizontal chamber oven, made of aluminium of 15 mm. thickness, 60 cm. long, 10 cm. wide, and 16 cm. high (to crown). It is heated by means of three gas burners, and has a capacity of about 10 lb. of coal. One end is closed by a removable aluminium door, which is opened for filling and emptying the oven. Volatile products are led off by a delivery pipe at the top of the oven, tar being collected in a cooled flask, light oils adsorbed by activated carbon, and the gas burnt with or without previous sampling. Two thermometers are placed in holes bored in the wall of the oven. In general, 3 kg. of coal are carbonised, the temperature being raised rapidly to 400°, then more slowly so that 1½ hrs. are taken to reach about 500°, at which temperature the coal is maintained for a further 1½ hrs. Steam can be introduced into the oven during the test if required.

W. T. K. BRAUNHOLTZ.

Softening points of pitches and asphalts. A. SPILKER (*Z. angew. Chem.*, 1929, 42, 263–264).—In order to avoid the use of mercury, a modification of the Kraemer-Sarnow method is proposed, in which a smooth rod of lead-antimony alloy (80:20) with the lower end rounded, having a cross-section 5 mm. in diameter, and weighing 8.0 g., is employed. The results obtained by this "Kraemer-Spilker" method agree with those obtained by the Kraemer-Sarnow method within the limits of experimental error.

S. I. LEVY.

Cracking process. A. N. SACHANEN (SACHANOV) and M. D. TILITSCHÉEV (*Ber.*, 1929, 62, 658–677; cf. *B.*, 1929, 84).—The thermal decomposition of the following substances has been investigated under pressure in an iron autoclave over varied periods at temperatures about 425–450°: paraffin, m.p. 53°, diisomyl, naphthalene, anthracene, cymene, tetrahydronaphthalene, and naphthenes. Paraffins are cracked into unsaturated and methane hydrocarbons. If the operation is protracted, the unsaturated compounds may become partly polymerised and transformed into a mixture of hydrocarbons of higher b.p. which consists of naphthenes, unsaturated hydrocarbons, paraffins, and, possibly, aromatic hydrocarbons. Coke may ultimately be produced from the last-mentioned compounds. Polymerisation of the olefines is favoured by high pressure and long

duration of the process; if, however, the products are removed rapidly from the sphere of action polymerisation of olefines and their transformation into naphthenes is slight. Under the customary conditions of cracking the reaction is unimportant. Thermal decomposition of aromatic hydrocarbons is influenced mainly by the side chains. With increasing length of the latter the compounds become less stable. Conversely, the aromatic hydrocarbons with short side chains are markedly more stable. Methyl groups are most permanent, and are scarcely affected under the usual conditions of cracking. The presence of aromatic hydrocarbons in the petroleum is due partly to dehydrogenation of naphthenes, more particularly to decomposition of complex into simpler hydrocarbons. The complex compounds have side chains of varying weight which are eliminated in very varied manner, thus rendering very improbable the production of appreciable amounts of unsubstituted aromatic hydrocarbons. This is confirmed by the absence of decomposition products of naphthalene from the corresponding fractions. The behaviour of naphthenes depends on the size of the ring. Hexamethylene derivatives lose their side chains and become dehydrogenated and converted into aromatic hydrocarbons, thus explaining the almost complete absence of six-membered naphthenes from the cracked products. Naphthenes with five-membered rings are mainly decomposed by partial loss of side chains, and then pass into the benzine and petroleum. The most unstable substances are the paraffins and cyclic hydrocarbons with very long side chains. These reactions cause a concentration of the aromatic hydrocarbons with short alkyl groups during the cracking process, and hence a marked increase in sp. gr. Accumulation of naphthalene derivatives and possibly of other condensed aromatic hydrocarbons is to be expected, as is confirmed by the high sp. gr. of the oils obtained. As the concentration of aromatic hydrocarbons with short alkyl chains increases the rate of decomposition falls. As cracking proceeds a point is generally reached at which the main components of the initial material are decomposed and the products consist mainly of aromatic hydrocarbons with short side chains. Small amounts of more highly condensed products are present. This concludes the first stage of the change, which appears to be characterised by large yields of benzine and petroleum and high sp. gr. of the oils or residues. Coke is not generally formed at this stage. The first process is characterised almost entirely by decomposition reactions and incipient condensations. With increasing concentration of aromatic hydrocarbons the condensation reactions assume predominance. The condensation products of higher b.p. pass into tarry products and ultimately into coke. Since the aromatic hydrocarbons at this stage have only short side chains, much gas is produced, mainly hydrogen and gaseous hydrocarbons. Liquid products are formed only in small amount. The second stage of cracking is characterised by the conversion of the residue into coke and formation of much gas. H. WREN.

Composition of heavy Sakhalin crude oils and their working up. A. N. SACHANOV (*Neft. Choz.*, 1928, 15, 53—54).—The characteristics of an Okhinski crude (Sakhalin) oil are recorded, together with those of the

mazout and of the fractions obtained on its vacuum distillation. The results of cracking experiments are also recorded.

CHEMICAL ABSTRACTS

Determination of petrol or benzol vapour in air. P. ANDREJEV (*Chem. Fabr.*, 1929, 147—148).—Absorption methods for the determination of hydrocarbons in air scarcely permit of their subsequent analysis. This, however, can readily be carried out if condensation methods are used. A combined apparatus, in which after a preliminary condensation residual vapour is absorbed, consists of a glass tube (13 mm. diam.) containing two narrower inner tubes and surrounded by a glass worm. At the lower end is a collecting flask, and the air passes successively through the worm, the inner tubes, and the wide tube. The last-named is charged with absorbent. The whole is immersed in carbon dioxide snow or liquid air. The air is dried and freed from carbon dioxide, and if a liquid absorbent is used this must also be freed from moisture.

C. IRWIN.

See also A., April, 389, **Equilibria between benzene and (a) ferric oxide gel, (b) silica gel** (LAMBERT and CLARK). 403, **Inflammation of mixtures of olefines and air in a closed spherical vessel** (MAXWELL and WHEELER).

Hydrogen [from water-gas]. LARYUKOV. **Sulphur dioxide from waste gases.** WEISSENBERGER and PIATTI.—See VII. **Silica bricks for coke ovens.** KNUTH.—See VIII. **Insulating oils.** EDWARDS.—See XI. **Bleaching mineral oils.** ELAKOV.—See XII. **Decolorising carbons and molasses.** GARINO and REGÈ.—See XVII.

PATENTS.

Rotary retort. Distillation [of carbonaceous materials]. T. A. W. DWYER, Assr. to H. O. SCHUNDLER, W. H. BOTSFORD, and A. J. JAMES (U.S.P. 1,703,418—9, 26.2.29. Appl., [A] 15.8.24, [B] 20.1.26).—(A) A rotary retort of the horizontal type is formed of a casing in the shape of a frustum of a cone and is heated externally. The material is fed into the narrower end of the retort and discharged at the wider end. The vapours are drawn off through a conduit in the upper part of the discharge end of the retort. (B) The carbonaceous material is heated to successively higher temperatures as it progresses through the retort, evolving volatile matter of successively increasing density. The conditions within the retort are maintained as little disturbed as possible so that the vapours retain their stratified formation until they are withdrawn. A. B. MANNING.

Distillation and utilisation of carbonaceous materials, and manufacture of coke, briquettes, and solid fuel compositions. B. LAING and H. NIELSEN (B.P. 307,366, 4.10.27).—The solid residue obtained by distilling carbonaceous materials in a current of hot gas, preferably as described in B.P. 276,407, 287,037, and 287,381 (B., 1927, 867; 1928, 356, 395), is blended with coal or coke, and the mixture is carbonised or briquetted. A fuel of increased reactivity is produced, suitable for domestic or metallurgical purposes.

A. B. MANNING.

Utilisation of carbonaceous material. D. PYZEL, Assr. to SIMPLEX REFINING Co. (U.S.P. 1,699,989,

22.1.29. Appl., 25.8.20).—Asphalt is heated and sprayed into the top of a furnace stack down which inert non-combustible material in sufficiently large particles to allow ready passage of gases is continuously circulating. In the top, relatively cool zone the asphalt is subjected to distillation, and lighter constituents escape with gaseous products from an outlet at the top of the furnace. The heavier, less volatile material undergoes partial combustion in the middle zone, where a controlled air-blast is supplied and residual coke is finally converted into water-gas by steam-blast admitted through the base of the furnace. The inert material falls continuously over a grid to remove dust, and is returned to the charging chute. The water-gas escapes through the combustion and distillation zones, and after removal of distillation products is collected.

R. BRIGHTMAN.

Coke treatment and product. A. A. KOHR, Assr. to KOPPERS Co. (U.S.P. 1,705,020, 12.3.29. Appl., 25.9.25).—To enable coke to be identified when burned, it is quenched with a solution of a flame-colouring substance.

F. G. CLARKE.

Increasing the adsorptive power of charcoal. H. B. LEMON (U.S.P. 1,699,243, 15.1.29. Appl., 29.8.21).—Gaseous material is absorbed by the vegetable charcoal and removed by evacuating to low pressure at 450–900°. With permanent gases the absorption is preferably effected at low temperatures, *e.g.*, with liquid air; with steam, absorption at temperatures up to 450–900° is preferable.

R. BRIGHTMAN.

Gas-producing apparatus. H. F. SMITH, Assr. to GAS RES. Co. (U.S.P. 1,699,166, 15.1.29. Appl., 20.9.21).—Tar is extracted from producer gas and returned to the generating chamber either by uniform spraying over the fuel in the charging hopper, or by distribution on fuel discharging from a fuel conveyor, or by discharging through nozzles on to fuel in a mixing chamber provided with an agitating wheel and leading to a charging hopper operated by a rotating shaft. R. BRIGHTMAN.

Electrothermal gas producer. W. S. YARD and E. N. PERCY (U.S.P. 1,703,505, 26.2.29. Appl., 12.2.24).—The fuel bed in a gas generator is heated electrically, the generating chamber being provided with a number of downwardly extending legs, at the bottom of which are the heating electrodes. At the top of the chamber are inlets for air, steam, and oil, whilst gas offtakes are situated at the lower ends of the legs. A. B. MANNING.

Drying of fuel gases. CHEM. ENGINEERING & WILTON'S PATENT FURNACE Co., LTD., T. O. WILTON, and J. PARKER (B.P. 307,600, 10.2.28).—The gases are passed through hygroscopic substances and then through traps or packings, *e.g.*, pumice stone, magnesite, etc., adapted to retain any suspended liquid or deleterious gases produced in the drying process. Two different hygroscopic liquids, *e.g.*, calcium chloride solution and sulphuric acid, may be used in series, the diluted sulphuric acid being subsequently utilised for the manufacture of ammonium sulphate. A. B. MANNING.

Increasing the yield in photochemical gas reactions. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 307,521, 2.11.27).—Gas mixtures, in which

photochemical reactions are to be induced by the resonance radiation of metallic vapours, are circulated past the source of radiation in such a way that only relatively small yields of the required products are formed during each passage, and these are continuously withdrawn from the circulating gases. The reacting gases are preferably purified from all traces of deleterious impurities. Certain reactions, *e.g.*, the production of formaldehyde from water-gas, are advantageously carried out under increased pressures. A. B. MANNING.

Apparatus for carrying out photochemical gas reactions. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 307,406, 2.11.27).—In effecting gas reactions photochemically induced by resonance radiation of metal vapours, *e.g.*, mercury vapour, rays of resonance wave-length are radiated into the reaction chamber in maximum amount, and the spectral development of the linear width of the resonance lines is adjusted to the absorption capacity of the metal vapour mixed with the reacting gases by suitably arranging the radiating devices, and adjusting the current density and the vapour pressure in them. Thus, *e.g.*, the reacting gases may completely surround the radiating device.

J. S. G. THOMAS.

Distillation of tar. T. O. WILTON, and CHEM. ENGINEERING & WILTON'S PATENT FURNACE Co., LTD. (B.P. 307,577, 10.1.28).—The tar is circulated through a plant comprising a feed tank, an economiser, a coil still, and a vapour box. The vapours from the vapour box are condensed and the residual dehydrated tar is passed again to the feed tank into which crude tar is admitted as required. The temperature of the coil still is raised to 300°, or higher, until soft pitch can be withdrawn from the system, which thereafter functions continuously.

A. B. MANNING.

Treatment of low-temperature tar and its distillates. G. T. MORGAN and D. D. PRATT (B.P. 307,382, 6.12.27).—The low-temperature tar is treated with a solvent, *e.g.*, petrol, ether, or a low-boiling distillate of the tar itself, in order to precipitate the pitch, and the phenols are then extracted from the clarified solution by means of an alkaline solution saturated with common salt.

A. B. MANNING.

Still for use in oil refining and method of making same by electric arc welding. E. C. R. MARKS. From A. O. SMITH CORP. (B.P. 307,566, 24.12.27).—Annular sections, consisting of thick metal plates each bent to form a tube and electrically welded in the line of its meeting ends, are aligned longitudinally with heads at each end, and their abutting ends are electrically welded into an integral tubular structure.

H. S. GARLICK.

Apparatus for combining natural gas and hydrocarbon oil for the production of gasoline. L. S. WORTHINGTON, Assr. to C. L. THOMPSON (U.S.P. 1,700,556, 29.1.29. Appl., 22.1.24).—The oil is fed from a supply tank to a mixing chamber provided with constant-level control device. Oil drawn from the bottom of the mixing chamber is circulated through a heating coil and discharged through a series of atomising nozzles in manifolds communicating with the mixing chamber. The natural gas, independently preheated, is

discharged in opposition in a series of nozzles directly above the oil-spray nozzles. The mixture is sprayed through the manifolds into the mixing chamber, the liquid settling and vapours escaping through a discharge pipe to a heating coil and condenser. R. BRIGHTMAN.

Safety appliance [for oil-cracking apparatus]. L. C. HUFF and A. G. BOGARDUS, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,703,624, 26.2.29. Appl., 5.7.23).—Located above an outlet pipe adapted to be ruptured when the pressure exceeds a predetermined value is the funnel-shaped end of a steam line carrying a fusible plug. H. S. GARLICK.

Treatment of oil shale. M. J. TRUMBLE (U.S.P. 1,704,956, 12.3.29. Appl., 2.9.24).—Raw shale is preheated by indirect heat-exchange with spent shale and distilled by contact with highly superheated steam, the volatile products being recovered. Any residual carbon in the spent shale is burnt during the period of heat exchange, and the resultant products of combustion are passed through the raw shale. H. S. GARLICK.

Treating shale and other bituminous solids. W. H. HAMPTON (U.S.P. 1,703,192, 26.2.29. Appl., 30.12.21. Renewed 28.6.28).—The solid material is intimately mixed with an ammonia-liberating base and a mineral oil, and heated at not above 370°, the volatile products being recovered. H. S. GARLICK.

Preparation of hydrocarbons. F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,702,899, 19.2.29. Appl., 28.5.23).—Carbonaceous material, e.g., low-grade coal of high ash content, is ground to a slurry in water, levigated, agitated with 25–30% of hydrocarbon oil and additional water if necessary to improve the separation of ash particles from the plastic mass of oil and disseminated carbonaceous material, and the plastic mass is hydrogenated at high temperatures and pressures in presence or absence of hydrogenation catalysts. R. BRIGHTMAN.

Treatment of petroleum oils. J. C. BLACK, Assr. to PAN AMERICAN PETROLEUM Co. (U.S.P. 1,704,588, 5.3.29. Appl., 6.2.28).—Crude petroleum is prepared for distillation by being heated with sufficient of an alkaline solution to convert corrosive metallic salts into non-corrosive compounds. After separation under pressure the remaining water is removed by mixing with enough hot residual oil to raise the temperature to the b.p. of water under the pressure employed. J. A. SUGDEN.

Recovery of oil from emulsions. S. W. COLE (U.S.P. 1,700,627, 29.1.29. Appl., 17.12.24).—Crude petroleum emulsions, e.g., "cut oil," "bottom settlings," are broken up by agitation with calcium carbide and sodium and subsequent settling for 3–24 hrs. R. BRIGHTMAN.

Manufacture of by-products from inactive and inert oils or gases. C. S. PALMER (U.S.P. 1,699,627, 22.1.29. Appl., 28.9.21).—Inert oil or gas, e.g., saturated or paraffin hydrocarbons, is cracked, mixed with air or oxygen, and partly ignited in presence of a catalyst (platinised or vanadium asbestos) at 300–700°, preferably in presence of saturated or superheated steam, the partly oxidised products being passed over heat-

regulating surfaces in countercurrent with water for the steam supply, and after precipitation of the tar the products are recovered in a series of condensers. The tail gases are burnt for carbon-black, and the oxidising gas is preheated by heat from the cracking process. R. BRIGHTMAN.

[Anti-knocking] fuel and its manufacture. G. HAMMOND, Assr. to FUEL DEVELOPMENT CORP. (U.S.P. 1,699,355, 15.1.29. Appl., 6.8.25).—About 95% of liquid fuel, e.g., commercial gasoline, is heated in a retort with 4–7% of commercial alcohol at about 65° and 25 lb./in.² for $\frac{1}{2}$ –1 hr. On cooling, the product is decanted from separated water. R. BRIGHTMAN.

Automobile motor cooling oil. R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 1,700,392, 29.1.29. Appl., 21.4.25).—Hydrocarbon oil having flash point 93–99°, b.p. 230–290°, and viscosity below 36 sec. (Saybolt) at 38° is used. R. BRIGHTMAN.

Production of mineral lubricating oils. A. G. BLOXAM. From ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 307,649, 14.4.28).—A lubricating oil fraction is treated with liquefied sulphurous acid, and the insoluble portion is distilled over alkali under reduced pressure with or without the use of steam. H. S. GARLICK.

Lubricating composition and its manufacture. J. W. FINLAY, Assr. to PIERCE PETROLEUM CORP. (U.S.P. 1,699,961, 22.1.29. Appl., 13.4.27).—73.5% by wt. of mineral (cylinder) oil (viscosity [Saybolt] 150–200 sec./99°), 16.5% of animal fat, 2.5% of lime, 2.5% of sodium hydroxide, and 5% of asphaltum or heavy tar (viscosity [Saybolt] > 2000 sec./99°) are mixed and heated above 150° to give a grease, the m.p. of which may exceed 200°. R. BRIGHTMAN.

Refining of mineral oil. H. BLUMENBERG, JUN. (U.S.P. 1,700,347, 29.1.29. Appl., 12.1.27).—Cracked gasoline or mineral oil (*d* 0.829–0.8235) is circulated through a tank in which it is subjected to an electric current, e.g., 10 amp./ft.² at 3–7 volts, in presence of 2–7% of hydrochloric acid, using aluminium anodes. The anode may be surrounded with a layer of bauxite or other finely-divided aluminium compound. R. BRIGHTMAN.

Refining of mineral oil. A. S. RAMAGE, Assr. to GYRO PROCESS Co. (U.S.P. 1,702,313, 19.2.29. Appl., 3.11.24).—Mineral oil is mechanically agitated in succession with measured quantities of sulphuric acid, sodium hydroxide solution, and with water, and the mixtures are delivered to the lower part of settling tanks in which the sludge separates out and the clear oil overflows slowly and continuously to the next agitator in the series, and finally to the still or storage. R. BRIGHTMAN.

Refining of [hydrocarbon] oil. E. T. HESSLE (U.S.P. 1,702,540, 19.2.29. Appl., 12.3.26).—The preheated oil is directed at 200–350° under 3–8 atm. from a nozzle in countercurrent with hydrocarbon vapour under pressure from a similar nozzle to produce fog within the reaction vessel, and the fog is passed through a molten decomposition catalyst of tin containing a small percentage of antimony at 250–400°. The cracked vapours are passed through fractionating columns, the first of which contains iron and manganese

oxides or other desulphurising agents, and the tail gases uncondensed by compression are circulated to gas burners under the still and the opposition nozzle in the cracking vessel.
R. BRIGHTMAN.

Treating hydrocarbon oil. F. W. HALL, Assr. to TEXAS CO. (U.S.P. 1,700,479, 29.1.29. Appl., 23.2.26).—Absorbent clay is used to refine successively two or more hydrocarbon oils of increasing colour and viscosity, the exhausted clay from each treatment being partly revived by treatment with steam after solvent-washing from excess oil, and used at consecutively higher temperatures. The exhausted clay from the final treatment is fully revived and used again for treating the lightest coloured oil in the cycle.
R. BRIGHTMAN.

Splitting of coal, oils, and other hydrocarbons. A. DEBO, Assr. to INTERNAT. BERGHIN-COMP. VOOR OLIE EN KOLEN-CHEMIE (U.S.P. 1,704,792, 12.3.29. Appl., 15.5.25. Ger., 23.5.24).—See Can. P. 258,201; B., 1927, 273.

[Hydrocarbon oil] distilling apparatus. M. F. DE BAJLIGETHY (Re-issue 17,233, 12.3.29, of U.S.P. 1,542,864, 23.6.25).—See B., 1925, 749.

Bleaching of montan wax. W. PUNGS, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,699,250, 15.1.29. Appl., 8.3.28. Ger., 11.3.27).—B.P. 303,080; B., 1929, 198.

Means for [magnetically] cleansing or filtering lubricating oils. F. R. SIMMS and B. C. JOY (B.P. 307,547, 14.11.27).

Hydrogen and gas mixtures (B.P. 307,529).—See VII. Bituminous Emulsions (B.P. 308,051 and U.S.P. 1,699,536—7).—See IX. Conversion of hydrocarbons (B.P. 288,193).—See X.

III.—ORGANIC INTERMEDIATES.

Fusel oil reaction [of the German pharmacopœia] for absolute alcohol and spirit. K. R. DIETRICH and H. JEGLENSKI (Pharm. Ztg., 1929, 74, 436—437).—The coloration with sulphuric acid and salicylaldehyde obtained with alcohol containing fusel oil is not specific for the latter, but may be due to the presence of aldehydes. Previous treatment with hydroxylamine or *m*-phenylenediamine and distillation separates all substances likely to be present in alcohol, other than fusel oil, which give the coloration with salicylaldehyde.
S. I. LEVY.

[Preparation of] 2-aminoanthraquinone from chlorobenzene and phthalic anhydride. P. H. GROGGINS and H. P. NEWTON (Ind. Eng. Chem., 1929, 21, 369—375).—A detailed investigation of the effect of conditions on purity and yield in the preparation of 2-aminoanthraquinone from chlorobenzene and phthalic anhydride through *p*-chlorobenzoylbenzoic acid and 2-chloroanthraquinone. For details of the effects produced by stipulated changes in conditions the original must be consulted, but the technique outlined below gives almost theoretical yields of commercially pure 2-chloroanthraquinone with over 90% conversion into 2-aminoanthraquinone of 95—96% purity. The Friedel-Crafts condensation is effected with 3 pts. of chlorobenzene to 1 pt. of phthalic anhydride and at least 10% excess

of aluminium chloride at 50° for 5 hrs. with thorough agitation. Hydrolysis of the reaction mass is effected with dilute sulphuric acid below 50°, almost the whole of the excess of chlorobenzene being recovered by steam distillation. The cooled, filtered solution of the sodium salt of the crude acid, after further steam treatment, precipitates pure chlorobenzoylbenzoic acid (m.p. 147·8°) when slowly delivered under the surface of a large quantity of dilute sulphuric acid with violent (non-swirling) agitation. Ring closure to 2-chloroanthraquinone is best effected with a 10 : 1 ratio of 95% sulphuric acid at 135° for 6 hrs., followed by a 10% dilution with water after cooling. The primary product (90—95%) thus obtained has m.p. 210·5—210·8°, the remainder being obtained by further dilution of the mother-liquors. Increase in the proportion of sulphuric acid used is conducive to increase in gross yield, the purity of the primary product varying directly with the acid ratio and inversely with the dilution. Satisfactory results for the amination of the 2-chloro- to the 2-aminoanthraquinone are obtained either with 28·5% aqueous ammonia with addition of nitrobenzene (which considerably inhibits the formation of hydroxyanthraquinone) for 7 hrs. at 215° (which ensures the necessary removal of all the 2-chloro-compound) or with 40—50% ammonia, the latter procedure involving higher pressures, but generally producing a purer product in higher yields. The product is discharged from the autoclave into dilute sodium hydroxide solution. Purification of the 2-aminoanthraquinone from accompanying dianthraquinonylamine and hydroxyanthraquinone is effected by dissolution in 96% sulphuric acid at 125°, dilution with hot dilute (about 50%) sulphuric acid to the required degree of acidity, and vigorous agitation at 125° for a further 30 min. After cooling, the product is filtered by suction, the black, acid mother-liquors being completely removed by washing first with the acid used for dilution and finally with hot water. A product of 95—96% purity, suitable for the preparation of vat dyes, is thus obtained.

J. W. BAKER.

See also A., April, 405, Action of atomic hydrogen on hydrocarbons (VON WARTENBERG and SCHULTZE). 406, Catalysts for formation of alcohols from carbon monoxide and hydrogen (FROLICH and others). 436, Nitration of substituted anilines (RIEDEL and others). 439, 2-Substituted derivatives of *p*-cresol (COPISAROW). 448, *ms*-Alkylanthracenes (BARNETT and GOODWAY). 459, Analysis of easily carbonisable organic liquids (SEVAG). 460, Determination of formaldehyde (LIPPICH).

Phenol from coke-oven gas liquor. HOENING.—See II. Synthetic tannins. BERKMANN.—See XV.

PATENTS.

Manufacture of sulphonic acids of *N*-acetoacetylated arylamines [acetoacetic sulphonyl-arylamides]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 307,531, 7.12.27).—Acetoacetic arylamides are sulphonated with oleum containing more than sufficient sulphuric anhydride to combine with the water formed. *E.g.*, acetoacetanilide or acetoacetic *o*-chloroanilide is treated at about 10° with 3—5 pts. of 20% oleum.

C. HOLLINS.

Manufacture of 2-hydroxy-3-carboxynaphthalene [β -hydroxynaphthoic acid] and its metallic salts. E. SCHWENK (U.S.P. 1,700,546, 29.1.29. Appl., 1.6.27. Ger., 2.4.27).—Alkali salts of 2-hydroxy-1-naphthoic acid are converted (yield 90%) into the corresponding salts of β -hydroxynaphthoic acid by heating above 200°, e.g., at 260° for 6 hrs. under 4 atm. R. BRIGHTMAN.

Apparatus for refining raw carbon disulphide. P. SIEDLER and E. SCHULTE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,699,255, 15.1.29. Appl., 5.11.27. Ger., 24.10.25).—See B.P. 260,236; B., 1927, 907.

Production of acetic acid. H. DREYFUS (U.S.P. 1,704,965, 12.3.29. Appl., 21.5.24. U.K., 22.6.23).—See B.P. 226,248; B., 1925, 148.

Photochemical gas reactions (B.P. 307,406 and 307,521).—See II.

IV.—DYESTUFFS.

See A., April, 395, Reactions between colloids.
I. Dyes and proteins (PAULI and WEISS).

Testing antiseptic dyes. REDDISH.—See XX.

PATENTS.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,364, 3.9.27).—A halogenated *meso*-benz- or -naphtha-dianthrone (B.P. 303,184 and 303,095; B., 1929, 238, 251) is condensed with a primary or secondary amine, especially amino- and diamino-anthraquinones, to give vat dyes. Examples are: α -aminoanthraquinone with tribromo-, tetrabromo-, trichloro-, and dichlorodimethyl-*ms*-benz-dianthrone (violet), tribromo-, tetrabromo-*ms*-naphthadanthrones (red-violet), dichloro-*ms*-naphthadanthrone (claret), di- and tetra-bromo-*allo*-*ms*-naphthadanthrone (navy-blue), and dibromo-*ms*-anthradanthrone (violet); β -aminoanthraquinone with tribromo-*ms*-benz-dianthrone (copper-red), tetrabromo-*ms*-naphthadanthrone (yellow-brown), dichloro-*allo*-*ms*-naphthadanthrone (copper-red), and chloro-*ms*-anthradanthrone (brown); 1-amino-4-methoxyanthraquinone with tribromo-*ms*-benz-dianthrone (grey-blue), dibromodimethyl-*ms*-naphthabenz-dianthrone (olive-green), tetrabromo-*ms*-naphthadanthrone (dark-blue), dichloro-*allo*-*ms*-naphthadanthrone (black), and dibromo-*ms*-anthradanthrone (grey-blue); 1-amino-2-methylanthraquinone with tetrabromo-*allo*-*ms*-naphthadanthrone (dark blue); 1 : 5-diaminoanthraquinone with dichloro-*allo*-*ms*-naphthadanthrone (violet-black). Diamines which are violet vat dyes are obtained by condensing dichloro-*allo*-*ms*-naphthadanthrone and dibromo-*ms*-anthradanthrone with *p*-toluenesulphonamide and hydrolysing the products. C. HOLLINS.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,328, 3.9.27. Cf. B.P. 307,364, preceding).—A halogeno- or nitro-dibenzanthrone or -isodibenzanthrone is condensed with an aminoanthraquinone to give violet-grey to black vat dyes. Examples are: α -aminoanthraquinone with dibrominated dibenzanthrone (blue-black); 1 : 4-amino-methoxyanthraquinone with dichlorinated dibenzanthrone (blue-grey to blue-black), or dibrominated *iso*-dibenzanthrone (violet-grey). C. HOLLINS.

Manufacture of [azo] dyes containing chromium, and their application. SOC. CHEM. IND. IN BASLE (B.P. 282,783, 24.12.27. Switz., 24.12.26).—Pre-chromed dyes of the type *o*-aminophenols \rightarrow 3-methyl-5-pyrazolone, give level orange to red shades on wool and silk. As first components are mentioned 3-amino-*p*-cresol-5-sulphonic acid, 1 : 2 : 4-aminonaphtholsulphonic acid, *o*-aminophenol, and others. C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Elastic properties of wool in water at high temperatures. J. B. SPEAKMAN (Trans. Faraday Soc., 1929, 25, 169—176; cf. B., 1929, 277).—The ability of strained Cotswold wool fibres to return to their original lengths in water at 18° decreases continuously with increasing degree, temperature, and duration of the strain. Set realised in water below 90° is permanent in cold but not in hot water, whilst above 90° strained fibres still contract if reheated in the absence of tension, though the process of recovery is incomplete, and a truly permanent set, i.e., permanent at both high and low temperatures, is realised. The slow rate of recovery and the fact that it occurs to the same extent at 60° and 100° indicate that dissolution of the fibrillæ does not occur. The disappearance in hot water of set permanent in cold water is probably due to some form of internal rearrangement (recrystallisation) in fibres rendered more amorphous by plastic flow. J. GRANT.

Cellulose from cereal straws. S. D. WELLS (Ind. Eng. Chem., 1929, 21, 275—278).—Cereal straws are capable of purification by very mild processes; the action of dilute caustic soda, sodium carbonate with or without sulphur compounds, milk of lime, and chlorine on wheat straw is described. Milk of lime, which is commonly used in technical operations, yields only a crude product unless further purification with chlorine is employed. By alternate chlorine and soda treatments, yields of 43% of pentosan-, lignin-, and ash-free cellulose may be obtained. 6% caustic soda extracts about 15% of xylan from straw, whilst a further 9% of substances yielding furfuraldehydes are obtained by a second soda extraction after chlorination. The spent cooking liquors from the sodium carbonate and sulphur treatment yield appreciable amounts of lactic and acetic acids on hydrolysis and fermentation.

F. R. ENNOS.

Cellulose from corn [maize] stalks. H. A. WEBBER (Ind. Eng. Chem., 1929, 21, 270—275).—A summary of the economics of the production of maize stalks, their chemical composition, and methods of preparing cellulose therefrom. The stalks contain about 35% of cellulose; although bulkier than wood, they are more easily delignified. Commercial applications will probably develop along two distinct lines, viz., utilisation of the entire stalk as crude fibre for wall-board etc., and of the purified cellulose as raw material for paper, artificial silk, etc. F. R. ENNOS.

Pulping of flax straw. VI. Properties of flax-straw cellulose and its value in the cellulose industries. F. R. SCHAFER and M. W. BRAY (Ind. Eng. Chem., 1929, 21, 278—280).—Analyses are given of seed flax straw and its principal components—the bast

fibre and the shives—and also of a number of pulps suitable for paper-making which were obtained by various methods of cooking. Calculated on the oven-dry weight of material, the entire straw contains rather more than 50% of cellulose; the yield of dry pulp varies between 33 and 48%, the highest being obtained by digestion with caustic soda. F. R. ENNOS.

New materials for the manufacture of artificial silk. ANON. (Bull. Imp. Inst., 1929, 27, 1—9).—An investigation as to the suitability of bagasse, sulphite pulp prepared from Tasmanian stringy-bark, and *Phormium tenax* fibre as sources of cellulose for artificial silk. After a preliminary separation of the fibrous portion from the useless pithy material by mechanical and chemical means, bagasse yields on suitable treatment about 24% of a dry bleached pulp similar in chemical composition to wood pulp employed in the manufacture of artificial silk. From the results of analysis Tasmanian stringy-bark pulp and that from *Phormium tenax* fibre also appear suitable, but in all cases a manufacturing trial would be necessary to decide the question. F. R. ENNOS.

Viscose. XXII. Viscosity of viscose. G. KITA, S. IWASAKI, T. NAKASHIMA, S. MASUDA, and K. MATSUYAMA (J. Cellulose Inst., Tokyo, 1929, 5, 59—71).—It is generally acknowledged that the viscosity of viscose solution falls during the first stages of ripening, reaches a minimum, rises very slowly, and, shortly before coagulation, suddenly rises quickly. The authors, working with a viscose from cotton paper, did not, in general, observe the slow increase referred to. This variation in behaviour is ascribed to the fact that they used a diluted viscose solution instead of the usual concentrated solution, and measured its viscosity in an Ostwald viscosimeter instead of by the falling-sphere method. Under the latter conditions the normal behaviour is found. The ripening process was studied for different cellulose materials by means of both methods, and the fibre-forming capacity of the viscose was also measured. The change of viscosity as found by the falling-sphere method is always the same, whereas that found by means of the Ostwald viscosimeter varies with the kind of cellulose used. The change in the fibre-forming capacity follows the viscosity as measured by the former method, and Schuster's theory (B., 1926, 398) as to the fall of viscosity during ripening is not upheld; it is thought more likely to be due to the increased dispersion produced by the dissolution of the cellulose xanthate. From the present work it is concluded that the change of viscosity is due to two factors, viz., dispersion and structure formation, and that the former effect is completed during the first days of ripening. This would explain the differences obtained when the viscosity is measured in the above two ways. Measurements of the viscosity of viscose under a range of pressures in the Ostwald viscosimeter have been carried out, and the results may be tolerably well expressed by means of the Ostwald equation. Results of fibre thickness measurements for different pressures also show good agreement with this equation. It is deduced that fibre thickness is not directly proportional to the pressure—which is actually the case. B. P. RIDGE.

Viscose silk. (MISS) J. C. MEISS (Chem. Weekblad, 1929, 26, 170—176).—An account of the preparation of the viscose solution and the spinning for silk.

S. I. LEVY.

Influence of air in the manufacture and preparation of pure cellulose for high-quality viscose rayon. W. A. DYES (Chem.-Ztg., 1929, 53, 185—186).—Whilst the process of mercerisation changes only the physical characteristics of cellulose, the presence of air or oxygen during this treatment influences the decomposition of the cellulose and the pure cellulose content of the final product. Various patents are quoted in which precautionary measures are described for avoiding the decomposition of the cellulose during the manufacture of alkali-cellulose, and others are described in which advantage is taken of the use of air or oxygen to accelerate the ripening. With definite quantities of oxygen the reaction may be so controlled as to give the required decrease of viscosity of the viscose without causing a considerable reduction in the α -cellulose content of the product. The importance of the content of chemically unchanged pure cellulose in viscose rayons and of maintaining as high a value as possible for this content in high-quality materials is discussed.

B. P. RIDGE.

Influence of liquids on manufactured cellulose hydrate. W. LÜDKE (Kolloid-Z., 1929, 47, 341—351).—The problem considered is that of increasing the tenacity of moist artificial threads of cellulose hydrate by the addition of another substance to the thread. This was investigated by measuring the tenacity of the thread under various organic liquids. Reproducible results were obtained when the liquids were dried by sodium acetate, and for each liquid a characteristic alteration in the tenacity was found. The results show that the tenacity under most liquids is not only greater than that under water or aqueous electrolytes, but is also greater than that of the air-dried thread containing the normal amount of moisture. The swelling of cellulose hydrate in organic liquids is less than in water. The tenacity falls rapidly on addition of small quantities of water to the organic liquid, soon reaching the value obtained under water itself. Films of cellophane show a similar behaviour to the threads of cellulose hydrate. In general, the influence of a given liquid depends on its water content, its behaviour towards water, and its swelling effect. As a first approximation, the lowering of tenacity produced by a liquid is parallel with its surface tension. The greatest tenacity is therefore obtained with liquids such as ethyl ether, ethyl alcohol, and acetone, and the least with water. E. S. HEDGES.

Acetolysis of cotton cellulose. C. C. SPENCER (Cellulosechem., 1929, 10, 61—73).—The influence of temperature, duration of treatment, and sulphuric acid content of the reaction mixture on the yield of cellobiose octa-acetate in the acetolysis of cotton has been investigated. For each concentration of sulphuric acid used there is an optimum temperature which corresponds with the greatest yield. The maximum yield was obtained by the treatment of 2 g. of cotton (98.6% of α -cellulose) with 0.2 c.c. of sulphuric acid and 8 c.c. of acetic anhydride at 50.4° for 14 days; the mean yield

from nine experiments was 42.3% of the theoretical. The product had $[\alpha]_D +41^\circ$ to $+41.8^\circ$ and m.p. $227.5-228^\circ$. With the exception of a 50% yield referred to by Hess and Friese (A., 1927, 44), the present is the highest value so far recorded in the literature. Hess and Friese's method was also used in this investigation, but the yield obtained was only 37% of the theoretical. On the contrary, in place of the cellobiose octa-acetate mentioned by these authors, under certain conditions a quantity of glucose penta-acetate is obtained.

B. P. RIDGE.

Plasticity and solvation of cellulose esters. S. E. SHEPPARD, E. K. CARVER, and R. C. HOUCK (5th Coll. Symp. Mon., 1928, 243-252).—A discussion.

CHEMICAL ABSTRACTS.

Rags and their preparation for papermaking. R. H. CLAPPERTON (Proc. Tech. Sec. Papermakers' Assoc., 1928, 9, 34-48).—The various grades of rags used in the papermaking industry are described and an outline is given of the preliminary treatment which each grade requires and the classes of paper for which the resulting bleached pulp is most suitable.

D. J. NORMAN.

Dissection of wood fibrils by chemical means. G. J. RITTER (Ind. Eng. Chem., 1929, 21, 289-290).—Treatment of delignified spruce and elm fibres with 68-77% phosphoric acid at $60-65^\circ$ for 15-20 min. causes separation of the fibrils (cf. A., 1928, 1162) into spindle-like bodies termed "fusiform bodies," which appear to be the smallest building units visible with the microscope.

F. R. ENNOS.

Bleaching of wood pulp. P. K. BAIRD and R. H. DOUGHTY (Pulp & Paper Mag., 1929, 27, 223-225).—A study of the effect of varying rates of agitation on the bleaching of wood pulp at consistencies of 2, 5, and 7% indicates that the rate of agitation, providing that it is sufficient to ensure a uniform reaction mixture, does not appreciably affect the total time of bleaching. The final whiteness of the pulp is, however, slightly lower at higher rates of agitation at the lower consistencies. It is concluded that the best results in the bleaching of wood pulp would be obtained if the pulp and bleaching solution were rapidly and efficiently mixed at the beginning of the process and were subsequently agitated but slightly or even not at all.

D. J. NORMAN.

Testing of wood pulps for strength. J. L. A. MACDONALD and G. A. CRAMOND (Proc. Tech. Sec. Papermakers' Assoc., 1928, 9, 130-146).—The problems associated with the evaluation of the strength of wood pulp are discussed and apparatus is described which permits of the preparation of test sheets from pulps both in the beaten and unbeaten states under standard conditions. Duplicate tests show a sufficiently close agreement for the purposes of mill control.

D. J. NORMAN.

Retention of clay in paper. F. W. BAILEY (Proc. Tech. Sec. Papermakers' Assoc., 1928, 9, 80-84).—A series of mill tests made during the manufacture of medium-sized super-calendered printing papers containing 50% of mechanical wood pulp indicate that clays of widely different degrees of "fatness" (as indicated by the bulk of a 5 or 10% aqueous suspension

of the clay after settling for varying periods of time) show no appreciable differences in retention.

D. J. NORMAN.

Quality control in the sulphite pulp industry. A. LAMPEN (Proc. Tech. Sec. Papermakers' Assoc., 1929, 9, 4-33).—See B., 1928, 564.

Evaluation of wood pulp. J. W. BERRIMAN (Proc. Tech. Sec. Papermakers' Assoc., 1929, 9, 114-119).

See also A., April, 383, **Deformation of fibrous materials** (HERZOG and JANCKE). 394, **Effect of temperature on viscosity and ease of precipitation of cellulose acetate** (WHITBY and GALLAY). 430, **Lignin and cellulose** (FREUDENBERG). **Alkylcelluloses** (SAKURADA). **Celluloseglycollic acid** (SAKURADA). **Cellulosexanthoacetic acid** (NAKASHIMA). **Cellulosexanthamides** (NAKASHIMA).

Cellulose in peat. KOMAREVSKY.—See II.

PATENTS.

Treatment of stem fibres. C. E. BAHRÉ (B.P. 307,128, 14.12.27).—Flax straw or other stem fibres are treated in the field by mechanical means, with or without previous drying or fermentation, so as to remove the woody or foreign material covering the fibrous layer; the latter is then baled and subsequently treated by chemical means or by natural retting to obtain the 50% of spinning tow corresponding to 40% of cellulose fibre which it contains. [Stat. ref.] F. R. ENNOS.

Operation of pulp digestors. E. MORTERUD (U.S.P. 1,703,745, 26.2.29. Appl., 25.2.27).—In order to utilise economically the heat from the hot gases liberated in the digestion of wood pulp under pressure, the digesting liquor is conducted to a separate gas- and steam-liberating vessel, or a series of these, under reduced or gradually decreasing pressure, in which the gases are liberated in stages and the heat therefrom is accumulated in suitable apparatus such as a series of surface condensers; the digesting liquor is then returned to the digester.

F. R. ENNOS.

Production of [white sodium-]cellulose. E. HÄGGLUND (B.P. 292,534, 15.6.28. Ger., 21.6.27).—In order to increase the yield and strength of the cellulose produced, the raw material (wood) is first incompletely opened up by boiling with alkali so that separation of the fibre is just possible. It is then treated with chlorine and afterwards with bleaching powder, the bleaching being completed after washing with water and alkali by a further addition of bleaching powder. F. R. ENNOS.

Isolating cellulose. J. O. PEIRCE and W. T. REDDISH, ASSRS. to TWITCHELL PROCESS Co. (U.S.P. 1,703,830, 26.2.29. Appl., 14.10.26).—Used paper is re-pulped, treated with a sulphonated mineral oil reagent (sodium salt) to remove foreign matter such as ink, size, etc., washed, and made into new paper.

F. R. ENNOS.

Production of artificial threads by the cuprammonium stretch-spinning process. J. P. BEMBERG A.-G. (B.P. 283,923, 9.12.27. Ger., 20.1.27).—While under treatment in the acid channels, the threads are conducted through one or more V-shaped thread guides, which are adjustable in position in the channels, and are

made of wire or strip material, so that they can be produced accurately to a predetermined size and shape.

F. R. ENNOS.

Treatment of sulphite-pulp waste. J. T. TRAVERS, ASSR. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,699,258, 15.1.29. Appl., 21.6.27).—The sulphite-pulp waste-liquor is settled from suspended matter, passed through porous calcium carbonate, and treated with a mixture of either 1.6 lb. of calcium sulphate, 2.4 lb. of slaked lime, and 0.6 lb. of ferrous sulphate, or of 4.2 lb. of "solid waste" (from alkali plant), 1.5 lb. of calcium sulphate, 1.3 lb. of slaked lime, and 0.5 lb. of ferrous sulphate per 1000 gals. Waste dust from the precipitators in cement manufacture may be substituted for "solid waste" or for calcium sulphate in the first mixture, and dicalcium phosphate may replace the ferrous sulphate.

R. BRIGHTMAN.

Treatment of waste sulphite[-cellulose] liquor. G. C. HOWARD (U.S.P. 1,699,845, 22.1.29. Appl., 22.11.26).—The sulphite liquor is treated with an alkaline reagent to afford successively an inorganic precipitate practically free from organic matter, and a precipitate comprising the major part of the lignin content, and is finally treated with fresh lime or pulverised ash from combustion of the lignin precipitate, the fluid sludge separated at this stage being used in treating a fresh quantity of sulphite-liquor.

R. BRIGHTMAN.

Manufacture of paper. J. A. DE CEW (U.S.P. 1,704,728, 12.3.29. Appl., 21.2.28).—The paper stock is freed from gases before it is converted into paper.

F. G. CLARKE.

Treatment of waste [sulphite-cellulose] liquor. E. L. RINMAN (U.S.P. 1,699,808, 22.1.29. Appl., 15.4.26. Swed., 17.4.25).—See F.P. 627,752; B., 1929, 50.

[Continuous] treatment of [unretted] flax for spinning. M. WADDELL and H. C. WATSON (B.P. 308,667, 17.9.27).

Manufacture of [subdivided] artificial fibres. F. FERRAND (B.P. 308,645, 24.12.27).

Spinning pump for conveying viscose or other liquids. G. ARENDT and O. WEICHER (B.P. 293,801, 11.7.28. Ger., 12.7.27).

Insulating material (B.P. 293,293).—See XIII. **Fibres from skins.** (B.P. 290,154). **Fabrics resembling wash leather** (B.P. 307,189).—See XV. **Yeast** (U.S.P. 1,703,272).—See XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Colouring of small glass beads and of Christmas tree ornaments with aniline dyes. W. HANNICH (Chem.-Ztg., 1929, 53, 265—266).—The colouring mixture used consists of an emulsion of gelatin, gum arabic, gum tragacanth, or Senegal gum, with or without starch, in a solution of an aniline dye. Application of the mixture may be made by spraying or dipping.

A. R. POWELL.

Bleaching of wood pulp. BAIRD and DOUGHTY.—See V. **Tizerah extract.** VOGEL.—See XV.

PATENTS.

Dyes containing chromium (B.P. 282,783).—See IV. **Coloured building material** (B.P. 307,448).—See IX.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Vapour pressure of some salt solutions of importance in the ammonia-soda process. B. NEUMANN, R. DOMKE, and E. ALTMANN (Z. angew. Chem., 1929, 42, 279—283).—The vapour pressures at 0—40° of saturated solutions of ammonium hydrogen carbonate alone and in admixture with sodium hydrogen carbonate, or ammonium chloride, or both, have been determined. In solutions containing all three salts the vapour phase in equilibrium at 30° contains 12.9 mg./litre of ammonia in air and 6.9 mg./litre in carbon dioxide. At temperatures below 35° in air and 30° in carbon dioxide the vapour phase contains ammonium hydrogen carbonate which is completely dissociated above 40°. Under a pressure of 1.2 atm. of carbon dioxide there is a well-marked point at 25.9° at which the solution contains only sodium hydrogen carbonate and ammonium chloride; under 2.5 atm. pressure this point is at 27.8°. Below these points sodium chloride, and above them ammonium hydrogen carbonate, is found in the solution. Theoretically, no ammonia should exist in the gas phase below these points, but actually there is also a minute amount which is shown to be due to dissociation of ammonium chloride. The partial pressure of ammonia over saturated ammonium chloride solution at 20° is 7.5 mm. of mercury. The results of this work indicate that the most favourable temperature for conducting the ammonia-soda process is 30°.

A. R. POWELL.

Iodometric determination of chromic oxide in potassium chromium alum. J. E. S. HAN (J. Amer. Leather Chem. Assoc., 1929, 24, 124—129).—Sodium peroxide (2 g.) is added gradually, with stirring, to 20 c.c. of a 2% solution of the sample mixed with 125 c.c. of water; the mixture is boiled for 30 min., filtered to remove ferric hydroxide and insoluble matter, and the filtrate titrated with 0.1N-sodium thiosulphate after adding potassium iodide and excess of acid. The ferric hydroxide may be dissolved in hot dilute sulphuric acid and determined colorimetrically or volumetrically.

D. WOODROFFE.

Composition of fluorides and fluosilicates sold as insecticides. R. H. CARTER and R. C. ROARK (J. Econ. Entomol., 1928, 21, 762—774).—Samples of commercial sodium fluoride contained: total F 40.3—43.9, NaF 89.2—98.8, NaHF₂O—0.04, Na₂SiF₆O—2.17, Na₂CO₃ O—4.39, moisture 0.004—1.02, impurities 0.4—6.5%, p_H of 2% solution 6.8—10.5, cub. in. per lb. 21.9—51.2. Samples of commercial sodium fluosilicate contained: total F 57.1—60.3, Na₂SiF₆ 94.4—99.8, NaHF₂ O—0.02—0.14, moisture 0.04—0.21, impurities 0.14—5.5%, p_H of 0.5% solution 3.1, cub. in. per lb. 22.6—28.5. The p_H of a 0.5% solution of commercial magnesium fluosilicate (100%) was 3.0—3.1. Results are also given for calcium fluosilicate, copper fluoride, barium fluoride, cryolite, and various

mixtures and dusting powders. The acidity of fluosilicate solutions may account for the burning of foliage. The addition of a small quantity of lime or sodium carbonate to fluosilicate to decrease the acidity converts some of the soluble fluosilicate into fluoride without increasing the p_H of the solution. The effect of crystal size on biological action is considered. CHEMICAL ABSTRACTS.

Influence of shaking on various precipitation reactions. G. THANHEISER and P. DICKENS (Arch. Eisenhüttenw., 1928—9, 2, 575—581; Stahl u. Eisen, 1929, 49, 430—431).—The use of the shaking apparatus previously described (cf. Bardenheuer and Dickens, Stahl u. Eisen, 1927, 47, 762) increases the grain size of barium sulphate and calcium oxalate precipitates, thereby increasing the rate of filtration, and accelerates the rate of precipitation of magnesium ammonium phosphate. In all cases shaking for 10 min. is sufficient. The grain size and rate of precipitation of ammonium phosphomolybdate are unaffected by agitation.

A. R. POWELL.

Wet purification method for hydrogen. I. LARYUKOV (Masloboino Zhir. Delo, 1928, No. 2, 6—7).—Hydrogen sulphide is removed from hydrogen obtained from water-gas by scrubbing with 5—10% sodium or potassium carbonate solution. The carbonate is regenerated by blowing hot air through the liquid.

CHEMICAL ABSTRACTS.

Purification of gases, especially chlorine, by repeated liquefaction. R. WASMUHT (Chem. Fabr., 1929, 145—147, 158—159).—No laboratory method exists for the preparation of chlorine, quite free from oxygen, on a reasonably large scale, or for its purification by chemical methods. Commercial chlorine containing 0.8% of impurities is best purified by liquefaction at -70° under atmospheric pressure using a mixture of solid carbon dioxide and acetone. The gas is first passed through sulphuric acid and further dried by freezing at -15° ; it then passes through a short steel cooling coil into a steel collecting vessel, these two being surrounded by the freezing mixture. Uncondensed impurities are drawn off by a filter pump. The apparatus is filled with chlorine gas before immersion in the freezing mixture. It is then suspended within a cooled Dewar flask against a counterpoise. The apparatus described yields 2.2 litres of liquid chlorine in 10 hrs. with a consumption of 20 kg. of carbon dioxide.

C. IRWIN.

Behaviour of carbon dioxide under pressure, and its possible industrial applications under moderate pressure. N. W. KRASE and J. B. GOODMAN (Chem. Met. Eng., 1929, 36, 162—163).—The reaction $\text{Ca}(\text{OAc})_2 + \text{H}_2\text{CO}_3 \rightleftharpoons \text{CaCO}_3 + 2\text{HOAc}$ was studied to ascertain whether higher pressures of carbon dioxide would move the equilibrium further to the right. Four experiments were undertaken at 800 and 2400 lb. pressure at temperatures varying from 20° to 200° on solutions with a fixed concentration of calcium acetate. The precipitated calcium carbonate was filtered off without releasing the pressure, and the amount of acetic acid liberated titrated after eliminating dissolved carbon dioxide. The experiments indicated that the concentration of acetic acid increased with temperature and

pressure, reaching 10% at 2400 lb. and 115° . Thus it may be possible to employ carbon dioxide under pressure in many chemical industries where a low concentration of hydrogen ions is required; the use of such a process would obviate neutralisation and subsequent filtration. Its possible application in, e.g., the conversion of starch into glucose is indicated. C. B. MARSON.

Recovery of sulphur dioxide from waste gases. G. WEISSENBARGER and L. PIATTI (Chem.-Ztg., 1929, 53, 245—247, 266—267).—The quantity of sulphur dioxide absorbed by methylcyclohexanone depends on the partial pressure of the gas in the waste gases and on the temperature. At 25° a maximum absorption of 0.25% of the weight of the absorbent is obtained from gases containing 0.3% of sulphur dioxide; with increasing concentration of sulphur dioxide the maximum absorption rises sharply up to 10%, then more slowly. With very dilute gas mixtures the presence of mercury accelerates absorption below and decreases it above 20° . Practically all the absorbed gas is expelled at 80° . The presence of sulphur trioxide in the gas mixture results in a slow sulphonation with the formation of a large proportion of lower-boiling compounds and some compounds of very high b.p., the liquid becomes dark brown in colour and fluorescent, and, on heating, a black flocculent precipitate forms. cycloHexanone behaves similarly to its methyl derivative, but the action of sulphur trioxide on it is much more severe. A. R. POWELL.

See also A., April, 399, Two forms of crystalline beryllium hydroxide (FRICKE and HUMME). 400, System NaNO_3 — Na_2SO_4 — MgCl_2 — H_2O at 0° , 10° , 25° , 75° , and 100° (LEIMBACH and PFEIFFENBERGER). 404, Decomposition of nitrous oxide in the silent electric discharge (JOSHI). 406, Quantitative electrolytic reduction of nitric acid (NIETZ). Formation of hydrazine from ammonia by electrical discharge (BREDIG and others). 407, Electrolysis of cyanogen halides (CLARK and STREIGHT). 411, Nitrosylsulphuric acid (JONES and others). Preparation of pure hydrogen fluoride (FREDENHAGEN and CADENBACH). 412, Quantitative analysis with the spectrograph (NITCKIE). 413, Detection of bromide and iodide (MURMANN). 414, Micro-determination of iodine in organic materials (REITH). Determination of nitrous oxide gasometrically (MENZEL and KRETZSCHMAR). Determination of nitrite ion (GERMUTH). 431, Synthesis of cyanamide (KADECFLECK). 473, Fixation of atmospheric nitrogen by *Azotobacter* (MEYERHOF and BURK).

Washing powders. PHILLIPS and others.—See XIX.

PATENTS.

Manufacture of a colloidal absorption [product] of hydrogen chloride. V. A. LAPENTA (U.S.P. 1,699,596, 22.1.29. Appl., 17.8.27).—An infusion of agar-agar, e.g., 20 g. in 1 litre of hot water, is autoclaved for 25 min. at 30 lb. and filtered warm through asbestos wool. 250 c.c. of hydrochloric acid are added and kieselguhr or purified siliceous earth containing 5% of tricalcium phosphate is stirred in to give a stiff paste. The resulting cake is pulverised when dry, the free hydrochloric acid content being determined by extraction with 0.1N-sodium hydroxide. R. BRIGHTMAN.

Production of hydrocyanic acid. M. J. BROWN, Assr. to PACIFIC R. & H. CHEM. CORP. (U.S.P. 1,702,761, 19.2.29. Appl., 11.1.24).—Sulphuric acid and cyanide solution or slurry are run continuously into a shallow bowl with propeller agitator. Hydrogen cyanide is evolved through the heat of the reaction and dilution and escapes through a liquid trap to the receiver or absorbent. The liquid is immediately thrown over the edge of the bowl into the conical container, down which it passes into a vessel heated by a live-steam manifold. Spent liquor is discharged at one end of the heater and hydrogen cyanide escapes by a pipe through the liquid trap to the receiver. Liquid from the trap is by-passed to the mixing vessel.

R. BRIGHTMAN.

Complex hydrofluoric acids. M. BUCHNER (Austral. P. 1355, 8.4.26).—Hot complex fluorides or mixtures thereof are treated either with hydrogen or hydrogen compounds or with mixtures yielding hydrogen or hydrogen compounds.

J. S. G. THOMAS.

Manufacture of alkali cyanides. H. B. KIPPER (U.S.P. 1,699,362, 15.1.29. Appl., 7.5.26).—An intimate mixture of an alkali metal salt, carbon, and iron or iron and manganese, *e.g.*, manganese ore, as catalyst is charged into a rotating drum heated at 1000–1500°, to which air or producer gas, preheated to 500–1000°, is simultaneously admitted. The alkali cyanide is discharged and falls through a housing box into an externally heated stationary drum with rotating beating and pulverising and movable scraper bars wherein the conversion is completed in a nitrogen atmosphere. The reaction products are discharged to a closed chamber from which the gases escape; the cyanide, after cooling, is removed. Gases from the rotating drum escape upwardly from the housing box, and, after scrubbing, the carbon monoxide is mixed with air and used to preheat the air or producer gas supply. R. BRIGHTMAN.

Production of water-glass solutions. I. G. FARBENIND. A.-G. (B.P. 299,763, 1.8.28. Ger., 31.10.27).—Alkali lyes containing alkali chlorides, obtained by the electrolysis of alkali chloride solutions, are heated in autoclaves under a pressure of, *e.g.*, 20–30 atm., with silica or siliceous material.

L. A. COLES.

Manufacture of sodium hydrogen pyrophosphate. J. N. CAROTHERS and C. F. BOOTH, Assrs. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,699,093, 15.1.29. Appl., 13.1.26).—61% phosphoric acid solution, containing less than 0.6% of iron and aluminium phosphates, is neutralised to methyl-orange with sodium carbonate, and the solution (d_{25}^{20} 1.56) is filtered and crystallised, the sodium dihydrogen phosphate being dehydrated with air at 150° and converted into sodium hydrogen pyrophosphate by heating at 240° for about 8 hrs.

R. BRIGHTMAN.

Manufacture of metallic nitrates. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 306,998, 1.12.27).—A nitrite obtained by the action of nitrous gas on the metal oxide or carbonate is treated with oxygen or gases containing oxygen in the presence of water at a pressure of 15 atm. or higher and above 150°, a catalyst, *e.g.*, an alkali hydroxide, being employed.

W. G. CAREY.

Manufacture of anhydrous metallic chlorides. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,524, 7.11.27 and 14.7.28).—Material containing metal oxides, *e.g.*, bauxite or kaolin in the manufacture of aluminium chloride, is treated in a well-insulated shaft furnace with a current of carbonyl chloride freshly prepared by the action of chlorine on carbon monoxide in the presence of a catalyst and heated to about 550° by its heat of formation. Coal is preferably mixed with the furnace charge, so that the issuing gases contain a high proportion of carbon monoxide and are thus suitable for the manufacture of fresh supplies of carbonyl chloride.

L. A. COLES.

Production of calcium molybdate. A. KISSOCK (B.P. 280,240, 7.11.27. U.S., 6.11.26).—Molybdenum ores, concentrated to contain about 80–90% of molybdenum sulphide, are roasted to convert the sulphide into the trioxide and, on completion, sufficient lime is added to convert the trioxide into calcium molybdate. The two processes are preferably effected in a single roasting furnace, *e.g.*, of the multiple-hearth type, the lime being fed on to one of the lower hearths and the temperature during the formation of the molybdate being maintained at about 760°.

L. A. COLES.

Production of cobalt carbonyl. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,112, 3.12.27).—Increased yields are obtained in the manufacture of cobalt carbonyl from cobalt and carbon monoxide by excluding oxidising agents during the preparation of the metal and by passing the carbon monoxide over a catalyst of an oxide of manganese or copper or a mixture of these oxides to remove free oxygen.

W. G. CAREY.

Production of hydrogen and gas mixtures containing the same. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,529, 2.12.27).—Mixtures of air, oxygen, or carbon dioxide, and steam, if desired, with gaseous, liquid, or solid carbonaceous material, *e.g.*, methane, gases obtained in the distillation of coal, tars, oils, etc., but excluding products obtained in the conversion of saturated into unsaturated hydrocarbons, are subjected to the action of the electric arc, and the gas mixtures obtained are treated with steam to convert carbon monoxide into carbon dioxide and hydrogen, the carbon dioxide and other impurities being removed from the product. A mixture of nitrogen and hydrogen suitable for the synthesis of ammonia is obtained by using air as the oxidising gas, and subsequently adding hydrogen, prepared, *e.g.*, by a similar process using carbon dioxide instead of air, to adjust the mixture to the correct proportion.

L. A. COLES.

Production of hydrogen sulphide. R. F. BACON (U.S.P. 1,700,578, 29.1.29. Appl., 5.5.27).—Hydrogen is passed through molten sulphur at 250–300° under 5–10 atm. with thorough agitation, volatilised sulphur being subsequently condensed.

R. BRIGHTMAN.

Sulphur composition and its manufacture. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,699,694, 22.1.29. Appl., 4.4.25).—Sulphur is dispersed with starch, gum tragacanth, and highly colloidal clay; *e.g.*, 9 pts. of sulphur, 1 pt. of bentonite, and 20 pts. of water in a ball mill give a dispersion 40 pts. of which

incorporated with 60 pts. of wood flour and hot-moulded at 130–140° and 1000 lb. gives a product of 35–65% greater transverse strength than is obtained from undispersed sulphur. R. BRIGHTMAN.

Manufacture of alkali nitrate. W. WILD and C. BECK, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,699,643—4, 22.1.29. Appl., 12.5.27. Ger., 8.7.26).—See B.P. 283,771—2; B., 1928, 230.

Purification of solutions of metal [zinc] salts. A. VOHL and W. WACHTENDORF (U.S.P. 1,706,196, 19.3.29. Appl., 3.1.27. Ger., 24.12.25).—See B.P. 263,809; B., 1928, 124.

Disintegrated alumina. C. VON GIRSEWALD, H. SIEGENS, and M. MARSCHNER, Assrs. to METALLGES. A.-G. (U.S.P. 1,704,599, 5.3.29. Appl., 28.5.27. Ger., 8.5.26).—See B.P. 284,131; B., 1928, 231.

Production of salt from brines and solutions. F. B. DEHN. From MARTIN-COLVIN Co. (B.P. 308,746, 28.12.27).—See U.S.P. 1,657,633; B., 1928, 230.

Treatment of iron pyrites (B.P. 307,188 and 307,190). Separation of metals from cyanide solution (U.S.P. 1,699,350).—See X. Granular fertilisers (B.P. 307,575). Non-hygroscopic phosphates (U.S.P. 1,706,101).—See XVI.

VIII.—GLASS; CERAMICS.

Devitrification of glass. P. VILLARD (Compt. rend., 1929, 188, 969–972).—Devitrification of glass is not produced by the individual action at 750–950° of any one of the furnace gases (illuminating gas, air, hydrogen, carbon dioxide, etc.), and in certain cases these gases may restore devitrified glass to its polished state. Hydrogen containing 1% of oxygen, however, produces devitrification owing to the removal of sodium or potassium by reduction of the silicate by atomic hydrogen, and the flame of the emergent gas is coloured by the alkali metal. Devitrification may therefore be avoided by the use of gases burnt as completely as possible, e.g., by addition of air or oxygen at a high temperature.

J. GRANT.

Kilns and kiln firing. I. S. R. HIND (Trans. Ceram. Soc., 1929, 28, 26–52).—A general summary is presented of the results of an extensive investigation of a number of industrial kilns firing ceramic building materials, refractory materials, and pottery. Exhaustive numerical data are given in tabular form. The characteristics of the different types of kilns—round down- and up-draught, continuous chamber, continuous ring, continuous tunnel (muffle and open-fire)—are discussed in detail. Attention is directed to the effect of setting density on the fuel consumption, and to the relative merits of gas firing and direct coal firing.

F. SALT.

Effect of substituting high-silica sand for some grades in lime-bonded silica bricks. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1929, 28, 62–64).—Three series of bricks bonded with 2% of lime were prepared, the first containing only Topley ganister, the second 9.6% of Lynn sand between 20- and 100-mesh and 90.4% of ganister, of which 51% passed through a 100-mesh sieve, and the third, in which all the ganister

grades smaller than a 30-mesh were replaced by sand. The bricks were fired to cone 16, and the sp. gr., porosity, and mechanical properties were determined. The sand was more readily inverted than the ganister, and its use, up to 10%, may be advantageous. The third series of bricks was unsatisfactory; they showed a lower bulk density and a higher porosity. F. SALT.

Properties and specifications of silica bricks for coke ovens. H. KNUTH (Feuerfest, 1929, 5, 21–26).—The requirements which silica bricks for coke ovens are expected to fulfil are outlined, and the physical and chemical properties and methods of testing which determine or give an indication of the quality of the bricks are critically reviewed. Theoretical considerations and practical experience indicate that sufficient criteria of the quality of a brick are given by tests determining the refractoriness under load, the sp. gr., and the porosity. Ten buyers' specifications are quoted and discussed in detail. On the basis of the data presented, it is suggested that a specification prescribing a minimum silica content of 93%, a minimum refractoriness of cones 32/33, a refractoriness under a load of 2 kg./cm.² of 1500–1600°, a maximum sp. gr. of 2.38, and a total porosity of 26–28% by vol. (maximum) would suffice for industrial purposes. F. SALT.

Application of colloid chemistry to the study of clays. I. A. E. J. VICKERS (Trans. Ceram. Soc., 1929, 28, 91–100).—A bibliographical review is presented, the object of which is to re-state problems confronting the student of clays in terms of colloid chemistry. Modern views on the nature of colloids are outlined.

F. SALT.

Selenium-red as a ceramic colour. J. H. POLGREAN (Trans. Ceram. Soc., 1929, 28, 87–90).—The selenium-red colour was produced successfully with a mixture of 20% of selenium oxide and 80% of cadmium sulphide. The colour was destroyed at temperatures above 800°. A suitable borax flux which fused at about 700° was developed. The best results were obtained on a biscuit surface and on a matte glazed surface. It was not successful as an on-glaze colour.

F. SALT.

See also A., April, 400, **Reduction of fused silicates by carbon monoxide (BOGITCH)**. 411, **Action of silica, alumina, and kaolin on barium sulphate (MARCHAL)**. 419, **Collapsing temperature of laboratory glass tubing (LAUBENGAYER)**. 420, **Pink kaolin, and ruthenium as minor constituent of Tanokami kaolins (IMORI and YOSHIMURA)**.

Heat-conduction problems. GRIFFITHS.—See I. **Gasworks carbonising plant.** GILL.—See II. **Colouring of glass beads etc.** HANNICH.—See VI.

PATENTS.

Sealing or jointing of glass and quartz. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 307,994, 22.12.27).—Use is made of a glass free from alkali metals and containing at least 75% SiO₂, more than 6% B₂O₃, and small amounts of alumina, lime, or magnesia. J. A. SUGDEN.

Ovens for ceramic products. O. MOLLE, and ATELIERS J. HANREZ, Soc. ANON. (B.P. 307,815, 14.12.27).

—A labour-saving kiln operation is described. The products are set on a slab, which is then conveyed to and from its position in the chamber or gallery by a system of special trucks. J. A. SUGDEN.

Refractory material. J. G. DONALDSON and H. L. COLES, Assrs. to GUARDIAN METALS Co. (U.S.P. 1,703,662, 26.2.29. Appl., 4.2.26).—A mixture of zirconia, silica, and an excess of graphite is fused and cooled. This material is then heated and molten metal poured round it. A union is effected between the two.

J. A. SUGDEN.

Refractory coating material for furnace installations. C. LEAN. From VEREIN F. CHEM. U. MET. PROD. (B.P. 307,580, 16.1.28).—Finely-divided material such as alumina, zirconia, zirconium silicate, thoria, etc. is mixed with a plastic refractory clay or bauxite together with small quantities of water-soluble salts (e.g., sulphates, phosphates, fluorides, borates). The product is plastic, burns on at low temperatures, and, being very dense, resists slag attack. Hydraulic binders may also be used, and give great strength in the unburnt state.

J. A. SUGDEN.

Refractory building material. G. KNUDSEN, V. M. GOLDSCHMIDT, and R. KNUDSEN (B.P. 307,391, 3.9.27. Addn. to B.P. 260,298; B., 1928, 232).—Hydrated magnesium silicates, more especially serpentine, may replace the talc in the process whereby the material is heated with magnesia in the presence of small amounts of accelerators to form magnesium orthosilicate. (Cf. also B.P. 301,547; B., 1929, 131.)

J. A. SUGDEN.

Manufacture of solid bodies of high density [from refractory materials]. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 292,997, 14.6.28. Ger., 29.6.27).—Powdered material (first compressed) is sintered at high temperature. The porous body is then compressed at a lower temperature between members which offer no resistance to lateral expansion. A body is obtained with a density 85% of that attained by fusion. A suitable press is described.

J. A. SUGDEN.

Polishing and grinding material. B. SZILARD (U.S.P. 1,704,308, 5.3.29. Appl., 14.6.24. Fr., 18.6.23).—A colloidal solution prepared from titanium oxide and a titanium salt is precipitated to obtain a very fine powder.

J. A. SUGDEN.

Treating glass plates and the like to prevent moisture affecting their transparency. O. DEMARET (U.S.P. 1,705,453, 12.3.29. Appl., 19.4.27. Belg., 27.4.26. Renewed 12.10.28).—See B.P. 267,428; B., 1927, 365.

[Refractory linings in] furnace construction. W. RORN, Assr. to VACUUM-SCHMELZE GES. M.B.H. (U.S.P. 1,704,902, 12.3.29. Appl., 12.12.24. Ger., 24.12.23).—See B.P. 226,801; B., 1925, 850.

Manufacture of splinterless glass. G. F. BASTER (B.P. 308,482, 3.4.28).

Enamelling metal (U.S.P. 1,704,586). Castings to be enamelled (B.P. 307,674).—See X.

IX.—BUILDING MATERIALS.

Hydrated Portland cement as a colloid. A. H. WHITE (5th Coll. Symp. Mon., 1928, 349—360).—Only the exterior surfaces of cement particles react with water; on drying and re-wetting, fresh colloid is formed before swelling of the gel previously formed prevents the penetration of more water. The swelling and shrinking of cement blocks which were alternately wetted and dried have been followed over a number of years. It is computed that the water is compressed to about half its volume. CHEMICAL ABSTRACTS.

Density of wood substance, adsorption by wood, and permeability of wood. A. J. STAMM (J. Physical Chem., 1929, 33, 398—414).—The apparent density of wood substance in water, organic liquids, and various aqueous solutions has been determined at 25° by means of a Gay Lussac pyknometer and compared with the true density at 25° determined by displacement of helium. The heartwood of seven different soft woods and two hard woods in the form of wood meal, dried to constant weight at 105°, was the material used. Particle size is without effect on density. The values of d obtained by means of helium are slightly less than those obtained with water, and are greater than those obtained with non-polar organic liquids. Alaska cedar wood gives values of d which increase with an increase in polarity of the displaced liquid, the density, viscosity, and compressibility of which are without effect on the apparent density. The true density of the heartwood of the Sitka spruce in helium, and the apparent densities in water and benzene, decrease with a rise in temperature over the range 10° to 40°, the coefficient of cubic expansion being $3.7 \times 10^{-4}/1^\circ$, approximately three times the expansion in external dimensions shown by a block of the same wood. The densities of the constituents of wood substances are also compared. The presence of solutes such as glycerin, acids, alkali, and chlorides in the displacement medium depresses the apparent density, the depression increasing exponentially with an increase in concentration. This effect with potassium hydroxide is abnormally large, and a slight permanent depression remains after its removal. Hydrogen, nitrogen, and oxygen are only slightly adsorbed by wood; chlorides are selectively and apparently hydrolytically adsorbed to a slight extent from aqueous solution. Potassium hydroxide is considerably adsorbed. The finer structure of wood is apparently impermeable to non-polar liquids. L. S. THEOBALD.

Compressive and transverse strength of brick. J. W. MCBURNEY (J. Amer. Ceram. Soc., 1929, 12, 217—229).

See also A., April, 388, Solubility of calcium sulphate from 0° to 200° (PARTRIDGE and WHITE). 410, Reactions between colloidal silica and lime (JOLIBOIS and CHASSEVENT).

Heat-conduction problems. GRIFFITHS.—See I.

PATENTS.

Roasting cement in a rotary kiln. O. LELLEP (B.P. 288,192, 2.4.28. Ger., 2.4.27).—Raw pulverised material is granulated by sprinkling with water while

in motion (cf. B.P. 292,987; B., 1929, 306), and falls upon a travelling grate. Here it is dried and partially sintered by the waste gases from the rotary kiln which are drawn through the grate. The material then falls into the rotary kiln, which may be much shorter than is usual. The thermal efficiency is much increased.

J. A. SUGDEN.

Manufacture of cement and the like. A. C. DAVIS (B.P. 307,763, 11.10.27).—A new type of kiln is described. A vertical kiln is heated by four burners (coal, oil, etc.) at the bottom. The raw material in the form of powder or slurry is projected from jets placed below the burners through and upward with the flames. The velocity of the hot gases is adjusted so that the spray is kept suspended until drying and clinkering is complete. The consequent increase in density causes the particles to fall back through the flames and collect in the bottom of the kiln. The waste heat of the gases and clinker is utilised by boilers or for preheating the air. The flame temperature and gas velocity are adjusted so that the material is suspended sufficiently long to attain the clinkering temperature of 1400°. The flames may be given a swirling motion to concentrate them at the bottom of the kiln. The clinker produced is already in a fine state of division and facilitates subsequent grinding.

J. A. SUGDEN.

Cementitious material. E. H. S. BROWNE and A. M. SARGINT (B.P. 307,625, 5.3.28).—Powdered glass is mixed with zinc sulphate and sufficient potassium silicate solution to give working consistency. With choice of suitable zinc salt it is acid-resistant. After setting, treatment with certain solutions (e.g., calcium chloride) promotes further hardening.

J. A. SUGDEN.

Packing of cement. K. CHRISTENSEN (B.P. 307,970, 16.12.27).—The dry cement is compressed into a solid block and wrapped in paper.

J. A. SUGDEN.

Manufacture of moulded pieces by using hydraulic binding material. J. C. SEAILLES and SOC. LAP (B.P. 307,638, 23.3.28).—Liquid hydraulic mixture is poured into the mould which is rapidly vibrated. Excess of water collects on the surface and is run off. The material is then in such a state that it may be removed immediately (before it has set) and allowed to set apart from the mould.

J. A. SUGDEN.

Insulating material and its manufacture. C. E. HITE, ASSR. to UNIVERSAL GYPSUM & LIME CO. (U.S.P. 1,702,729, 19.2.29. Appl., 21.4.24).—Starch material is converted into an aqueous paste by agitating and heating, e.g., to 87°, and the paste is mixed with 0.5% of sulphuric acid and transferred to a lead-lined storage tank, whence it is mixed continuously with comminuted calcined gypsum containing 10–12% of carbonates, silicates, or similar impurities. The plastic mixture may be discharged direct on to the surface of paper liners for the production of wall-board.

R. BRIGHTMAN.

Manufacture of bricks. F. BANDINI (B.P. 307,550, 12.12.28).—The wet clay containing not less than 20% of moisture is heated to about 95°, extruded through a heated machine, and cut up into bricks. These may then be dried in a humidity dryer in

10 hrs. Abrupt changes of temperature are avoided throughout.

J. A. SUGDEN.

Coloured building material. G. H. HADFIELD, and SAND & SHINGLE, LTD. (B.P. 307,448, 8.12.27 and 4.9.28).—Sand or finely-divided stone, granite, etc. is mixed with a colouring matter (minerals such as glauconite or aniline dyes) together with sufficient Portland cement to bind to a weak, friable mass. This is then broken down again. The colour-retaining properties of this material may be enhanced by the incorporation of a small amount of oily matter.

J. A. SUGDEN.

Plaster. J. E. UNDERWOOD and C. A. CABELL, ASSRS. to NAT. LIME ASSOC. (U.S.P. 1,703,125, 26.2.29. Appl., 21.4.26).—A mixture of hydrated lime, hydrated calcium aluminate, a carbonate, a sulphate, and a retarder is claimed.

H. ROYAL-DAWSON.

Manufacture of marble plaster (artificial marble). L. E. CHASSEVENT (U.S.P. 1,703,097, 26.2.29. Appl., 26.4.27. Fr., 7.5.26).—A mixture of plaster and water is compressed so that the amount of water remaining in the product corresponds to that necessary for the conversion of plaster into gypsum.

H. ROYAL-DAWSON.

Fireproofing and preservation of wood. F. S. VIVAS, ASSR. to INTERNAT. FIREPROOF PRODUCTS CORP. (U.S.P. 1,702,285, 19.2.29. Appl., 11.3.27).—Green wood is treated with 5% sodium hydroxide solution at 100° for about 1½ hrs. to remove resins. The container is evacuated and the wood is treated at 150 lb./in.² and 80° with an aqueous solution of, e.g., 2.5 pts. of calcium chloride, 0.5 pt. of ammonium chloride, and 0.5 pt. of boric acid in 10 pts. of water. The wood is dried with hot air, treated at 150 lb./in.² with a solution of, e.g., 2.5 pts. of zinc sulphate and 2.5 pts. of aluminium sulphate in 100 pts. of water, and finally treated for 6 hrs. with hot water.

R. BRIGHTMAN.

Bituminous or like emulsions. J. A. MONTGOMERIE (B.P. 308,051, 16.2.28).—A small amount (1–5%) of fatty acid pitch of vegetable and/or animal origin is added to the material, which is poured into a hot dilute alkaline solution. In some cases a solution of the material in a suitable solvent (e.g., naphtha) is used. A soap (e.g., 0.25% of sodium laurate) may be added to the emulsion to increase the spreading or wetting power.

J. A. SUGDEN.

Manufacture of water pastes of bituminous emulsions. L. C. JONES, C. C. LOOMIS, and H. W. BANKS (U.S.P. 1,699,536—7, 22.1.29. Appl., 8.8.24).—(A) Disintegrated bituminous material, e.g., 150 pts. of crude Trinidad asphalt, is ground with 135 pts. of water in a ball mill and 15 pts. of gasoline or other volatile hydrocarbon solvent for the bitumen are added, grinding being continued to produce a smooth paste. A small quantity of soap or other alkaline dispersing agent may be added. (B) The bituminous material is dispersed, e.g., by melting 54 pts. of blown asphalt (softening pt. 55°) containing 7% of soft coumarone resin, and pouring intermittently into the thin paste obtained from 10 pts. of China clay and 13.5 pts. of denatured alcohol, further alcohol being added to maintain the creamy viscous condition of the mix.

R. BRIGHTMAN.

Wood-preserving means. K. H. WOLMAN (U.S.P. 1,700,010, 22.1.29. Appl., 9.11.23. Ger., 25.9.23).—See B.P. 229,179; B., 1925, 284.

Production of floor, roof, and wall coverings [of fabric, rubber, and bitumen]. M. H. TATE (B.P. 308,504, 1.5.28).

Drying and/or otherwise treating timber. C. GOODALL (B.P. 308,743, 28.12.27).

Treatment of sulphite-pulp waste (U.S.P. 1,699,258).—See V. Refractory building material (B.P. 307,391).—See VIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Properties of Thomas rail-steel. E. H. SCHULZ and A. WIMMER (Stahl u. Eisen, 1929, 49, 385—388).—The mechanical and wearing properties of Thomas steel are shown by a review of the literature and by comparative tests on two steels to be at least equal to those of Siemens-Martin steel for the manufacture of rails.

A. R. POWELL.

Brittleness in mild steel. G. R. BOLSOVER (Iron and Steel Inst., May, 1929. Advance copy. 15 pp.).—Mild steel rods which have been extended 15% or more become brittle on tempering at about 250°, whereas undeformed rods after similar heat-treatment remain ductile. At the room temperature the impact resistance of tempered deformed rods falls from 90 to 10 ft.-lb., but increases again with rise of testing temperature to a maximum at 25° with steel quenched and tempered prior to cold work, at 80° with normalised steel, and at 100° with steel very slowly cooled from 950°. With increasing phosphorus content the extent to which the impact resistance is decreased in any of the above conditions is increased, especially if the steel has been reheated after cold-working.

A. R. POWELL.

Experiences with the Lurgi sinter apparatus. M. BLAU (Stahl u. Eisen, 1929, 49, 388—392).—Experiments on the briquetting of iron ore from the Geier mine containing 25—27% Fe, 16—17% Mn, 13—15% SiO₂, 8% Al₂O₃, 8—10% of combined water, and 24% of moisture are described. The ore is ground to 10 mm., without drying, in a special mill and mixed with 10% of coke in an Erko mixer. The mixture is conveyed by mechanical means to a charging bin where it is moistened with 4—5% of water and discharged on to a layer of previously sintered ore in the Lurgi sinter apparatus. Ignition is effected by means of gas from an anthracite gas-producer, and sintering proceeds by drawing a current of air through the mass the thickness of which should not exceed about 30 cm. Full working details and cost data are given.

A. R. POWELL.

Magnetic roasting of iron ores. W. LUYKEN and E. BIERBRAUER (Arch. Eisenhüttenw., 1928—9, 2, 531—543; Stahl u. Eisen, 1929, 49, 466—467).—Reduction of ferric oxide ores at 500° for 30 min. or at 700° for 10 min. in coal gas or producer gas affords a highly magnetic ferrosiferrous oxide, but for good results on a commercial scale close grading of the particle size is essential. By continuing the reduction

to obtain ferrous oxide and then roasting this product at 500° in the air a highly magnetic form of ferric oxide is obtained which is readily separated from the gangue by means of a weak magnetic separator; in this case no grading of the ore is necessary, and there is no danger of oxidation or reduction proceeding too far as is possible in the first case.

A. R. POWELL.

Dimensions, working conditions, and efficiency of German electric steel furnaces. S. KRIZ (Stahl u. Eisen, 1929, 49, 417—425).—The results of a questionnaire to German steel smelters are summarised, and data are given for numerous types of electric steel furnaces.

A. R. POWELL.

Economics of various refining agents in the Siemens-Martin process. S. SCHLEICHER (Stahl u. Eisen, 1929, 49, 458—462).—The efficiencies and costs of various types of iron ore and of rolling-mill scale in reducing the carbon content of iron during the refining process in the open-hearth furnace are compared. The rate of decarbonisation of the iron is not affected by oxygen content of the ore, which is of importance only in determining the amount of ore required. A high content of silica in the ore necessitates a larger addition of lime and results in greater losses of iron in the slag. On the basis of the analysis of the ore, the cost of ore and lime, and the loss of iron in the slag the costs of the various types of ore for removing 100 kg. of carbon from the iron are calculated.

A. R. POWELL.

Case-hardening [of steel] in cyanide baths. F. RAPATZ (Stahl u. Eisen, 1929, 49, 427—429).—Steel takes up both nitrogen and carbon from a fused cyanide bath, the outer surface layers being richer in nitrogen than in carbon, but the rate of diffusion of carbon into the interior is much greater than that of nitrogen. The best results are obtained at 850°, the depth of penetration being about 0.6 mm. in 2 hrs.; at 950° the amount of carbon taken up is only about half that absorbed at 850° in the same time, but the nitrogen absorption is only slightly less. The process is particularly adapted to the treatment of small articles and for replacing carbon lost from the surface layers during heat-treatment.

A. R. POWELL.

Determination of hydrogen in steel. T. E. ROONEY and G. BARR (Iron and Steel Inst., May, 1929. Advance copy. 8 pp.).—An apparatus is described in which the determination is made by heating the metal in pure nitrogen in an electrically heated silica tube and measuring the volume of hydrogen evolved by means of the Shakespear katharometer.

A. R. POWELL.

Molybdenum steels and their use in permanent magnets. A. F. STOGOFF and W. S. MESSKIN (Arch. Eisenhüttenw., 1928—9, 2, 595—600; Stahl u. Eisen, 1929, 49, 429—430).—Steel with 0.9—1% C and 2—2.5% Mo has considerably better magnetic properties than the usual tungsten and chromium magnet steels, but the decrease in the magnetic moment by ageing is approximately the same. The highest values for the remanence and coercivity are obtained by quenching in water from 800°; a slightly lower temperature gives much lower values for these properties, whereas a slightly higher temperature has little effect. Complete

dissolution of the molybdenum carbide into solid solution does not occur below 1100°; at 800° the alloy has a granular pearlitic structure. A. R. POWELL.

Tungsten in the chemical industry. H. ALTERTHUM (Z. angew. Chem., 1929, 42, 275—278, 308—314).—A review of recent journal and patent literature showing the many new uses of tungsten in alloys resistant to wear and corrosion and in various products for the chemical industry. A. R. POWELL.

Rise of the break in the tensile strength curve of metals due to strain and ageing. GALIBOURG (Compt. rend., 1929, 188, 993—995).—The curves representing the variation of the rise for extra-soft steel as a function of time are hyperbolæ of the form $x = ay/(b - y)$, where x is the time of ageing, y the difference between the final load of the first strain and the load corresponding with the break in the second strain curve, and a , b are constants. The rate of rise increases with the temperature, the temperatures of ageing between the first and second strains being 12.5—14.5°, 50—53°, and 175—180°. Pure nickel aged at 175—180° also showed the phenomenon.

J. GRANT.

Influence of the dimension of the test pieces in viscosity tests of metallurgical products. J. Cournot (Compt. rend., 1929, 188, 995—997; cf. B., 1926, 161).—Experiments at 15—200° and 200—350° on aluminium (99.9%) and commercial duralumin wire, respectively, annealed at 350°, showed that the practical limit of viscosity increases with the diameter (1—2.8 mm.) at a rate which is rapid for the fine wires, but appears to approach an asymptotic value for the thicker wires. In the latter case the temperature has only a slight influence on the rate of increase. The phenomena are more marked for duralumin.

J. GRANT.

See also A., April, 384, **Properties of copper-antimony alloys** (STEPHENS and EVANS). **Recrystallisation of aluminium** (VAN ARKEL and VAN BRUGGEN; BURGERS and BASART). 388, **Properties of iron-copper alloys** (KUSSMANN and SCHARNOV). **Influence of rate of cooling on structure of eutectics** (TAMMANN and BOTSCHWAR). 398, **Activities of molten alloys of thallium with tin and with lead** (HILDEBRAND and SHARMA). **System magnesium-zinc** (HUME-ROTHERY and ROUNSEFELL). **Cadmium-rich alloys of system cadmium-gold** (DURRANT). 399, **Silver-copper eutectic** (LEROUX and RAUB). 406, **Catalysis by metals of platinum group** (LEVI). 412, **Quantitative analysis with the spectrograph** (NITZKIE).

Heat-conduction problems. GRIFFITHS.—See I. **Metals and brewing.** MATTHEWS.—See XVIII.

PATENTS.

Furnace. T. T. SCOTT, ASSR. to SCOTT FURNACE Co. (U.S.P. 1,703,460, 26.2.29. Appl., 21.8.26).—In a furnace of the reverberatory type air conduits are formed between the bridge and the end wall, this space being partially arched to the level of the bridge walls. Fuel is blown into the end of the furnace to meet the air passing over the bridge. C. A. KING.

Furnace treatment of metal and metalliferous material. AMER. RADIATOR Co., Assees. of D. H. MELOCHE (B.P. 289,007, 26.3.28. U.S., 19.4.27).—The bottom of a metal-melting furnace of cupola type is packed with broken refractory material which supports the charge to be melted. Powdered fuel is burned in one or more tubular combustion chambers, which are attached to the cupola and downwardly inclined so that the combustion gases pass through the bed of refractory material and upwards around the metal in the furnace.

C. A. KING.

Apparatus for melting and casting. H. L. COLES and J. G. DONALDSON, ASSRS. to GUARDIAN METALS Co. (U.S.P. 1,703,658, 26.2.29. Appl., 3.7.26).—An electric melting furnace comprises a heating chamber provided with a passage way through which a series of material-conveying troughs pass continuously in such a way that their contents are brought below a single series of arc electrodes which are positioned to melt simultaneously all of the material in each trough as it passes below them.

A. R. POWELL.

Annealing furnace. T. CARTWRIGHT (B.P. 307,522, 3.11.27).—A tunnel furnace having two lines for trucks is divided by arches and baffle walls into six chambers of which only the two middle chambers are heated to maximum temperature. Material for annealing, e.g., metal plates, is conveyed in opposite directions so that heat transference will take place in the end portions of the furnace. The furnace is suitable also for roasting ores.

C. A. KING.

Sheet-steel annealing. P. A. MEEHAN, ASSR. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,703,630, 26.2.29. Appl., 11.8.27).—A layer of diatomaceous material is placed between each pair of superposed layers of sheet metal.

H. ROYAL-DAWSON.

Reduction of iron ores. M. LAMBOT (B.P. 301,011, 22.3.28. Belg., 23.11.27).—A mixture of iron ore and excess of carbonaceous material is fed into a rotary chamber heated externally so as to reduce the ore and partially carbonise the reduced metal. The mixture is discharged continuously into a vertical furnace chamber in which carbonisation is completed, and from the lower end of which the outlet of material to a fusion furnace can be regulated by means of a slide valve. C. A. KING.

Obtaining iron from ores. VEREIN. STAHLWERKE A.-G. (B.P. 297,097, 29.5.28. Ger., 16.9.27).—Iron ores are chlorinated with ammonium chloride with or without calcium or magnesium chloride in a current of hydrogen chloride, and the product is subjected to fractional sublimation to obtain pure ferric chloride, which is reduced to iron by heating in a current of hydrogen, the hydrogen chloride so formed being used for the regeneration of ammonium chloride by interaction with the ammonia liberated in the first stage.

A. R. POWELL.

Reduction of ore and conversion of hydrocarbons. W. H. SMITH (B.P. 288,193, 26.8.27. U.S., 4.4.27).—Iron ore is passed in fine streams through a retort into which a stream of heavy hydrocarbon oil is injected preferably above the reduction zone. Cracking takes place with the deposition of carbon on the ore particles and the production of vapours of lower hydrocarbons.

and a gas relatively rich in methane and hydrogen. As the carbon-coated particles fall through the hot zone of the retort they are reduced to sponge iron, in the presence of which the excess of hydrogen and carbon react to produce methane. Several modifications of the process are described.

A. R. POWELL.

Treatment of iron pyrites. S. I. LEVY and G. W. GRAY (B.P. 307,188 and 307,190, 10.2.28).—(A) Iron pyrites containing copper, lead, and zinc is heated at 700° out of contact with the air so as to expel sulphur and leave a residue of ferrous sulphide, which is digested with hydrochloric acid to obtain ferrous chloride solution, hydrogen sulphide, and an insoluble portion with a high copper content. The filtered solution is electrolysed to remove lead and evaporated to recover ferrous chloride crystals, which are dehydrated and heated in air and/or steam to obtain ferric oxide and hydrochloric acid. This acid is mixed with the mother-liquors from the crystals and the mixture used for decomposing further quantities of ferrous sulphide, the cycle being repeated until the mother-liquors are sufficiently rich in zinc to repay treatment for its recovery. The copper residues are roasted and leached to extract copper and zinc, and the sulphur dioxide formed is allowed to react with the hydrogen sulphide produced earlier, the excess of the latter being burnt in a Claus burner to obtain sulphur. (B) Pyrites is heated in chlorine under conditions which ensure expulsion of sulphur and arsenic and sublimation of the zinc and lead as chlorides. The residual copper and ferrous chloride are extracted with boiling water and the filtered solution is cooled to obtain ferrous chloride crystals. The mother-liquor and washings from the crystals are treated with scrap iron to separate the copper, then electrolysed to remove lead, and returned for the extraction of further quantities of the chlorinated ore. The ferrous chloride crystals are dissolved in water and the solution is electrolysed to obtain pure iron and chlorine for further chlorination.

A. R. POWELL.

Roasting of pyritic ores. NAT. PROCESSES, LTD.; and S. ROBSON (B.P. 307,439, 8.11.27).—Raw pyrites is mixed with 3—5 times its weight of previously roasted ore, and the mixture is roasted in a Dwight-Lloyd blast-roasting apparatus to obtain a gas rich in sulphur dioxide.

A. R. POWELL.

Manufacture of lined metal containers. W. E. BALLARD (B.P. 307,573, 7.1.28).—Iron or steel drums are lined with lead by the usual method; the bottom is also provided with a lead lining, then fixed into position and, after filling the container with a cooling liquid, is welded on so that, by maintaining the liquid under slight pressure, a water-tight joint is obtained.

A. R. POWELL.

Enamelling metal. W. J. BECK and J. A. AUPPERLE, Assrs. to AMER. ROLLING MILL Co. (U.S.P. 1,704,586, 5.3.29. Appl., 16.11.23).—A steel very similar to mild steel, but containing an amount of silicon which, though insufficient to spoil the deep drawing and spinning property, forms a bond with the enamel, is used in the process.

J. A. SUGDEN.

Treatment of castings to be enamelled. W. ECKHOFF and L. DEUSSENER (B.P. 307,674, 23.6.28).—

Development of pinholes is avoided by mixing the dry enamel or treating the surface with a highly inflammable material such as petrol, thus preventing moisture from entering pores in the metal surface.

J. A. SUGDEN.

Ingot mould. D. J. GILES, Assr. to LATROBE ELECTRIC STEEL Co. (U.S.P. 1,703,442, 26.2.29. Appl., 6.12.26).—A mould for casting steel ingots is formed from a cast-iron alloy containing 0.25—3% Cr.

H. ROYAL-DAWSON.

Separation of copper from nickel. I. W. WILENCHIK (U.S.P. 1,703,329, 26.2.29. Appl., 16.4.28).—Copper-nickel alloys are fused with sulphur, sodium sulphate, calcium sulphate, and coke, whereby, on settling, two layers are obtained the lower of which consists of nickel sulphide and the upper of sodium copper sulphide.

A. R. POWELL.

Separation of precious metals and copper from cyanide solution. L. H. DUSCHAK, Assr. to OLIVER CONTINUOUS FILTER Co. (U.S.P. 1,699,350, 15.1.29. Appl., 1.10.25).—The solution is treated with an amalgam, e.g., zinc or sodium amalgam, containing a metal electropositive with regard to copper till most of the precious metals are removed. The copper is then removed by further treating with a fresh portion of amalgam and the solution used in treating fresh ore.

R. BRIGHTMAN.

Smelting of manganese [ore]. A. G. BETTS (U.S.P. 1,703,657, 26.2.29. Appl., 23.11.25).—Manganese silicate ore is smelted with a sulphide of iron to obtain metallic iron, manganese sulphide, and a ferrous silicate slag. The manganese sulphide layer is converted into oxide, which is reduced to metal in the usual way.

A. R. POWELL.

Anti-friction metal. R. J. SHOEMAKER, Assr. to S. & T. METAL Co. (U.S.P. 1,703,212, 26.2.29. Appl., 23.12.27).—A lead alloy containing 1—5% Sn, 0.1—1% Ca, 0.02—0.3% Mn, and 0.02—0.1% Al is claimed.

H. ROYAL-DAWSON.

Preparation of refractory metals. J. W. MARDEN, T. P. THOMAS, and J. E. CONLEY, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,704,257, 5.3.29. Appl., 12.2.23. Renewed 8.2.27).—The oxides of metals such as uranium, thorium, etc. are reduced by heating with a mixture of an alkaline-earth metal and halide.

J. A. SUGDEN.

Refractory [metal] article. C. F. LORENZ, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,704,256, 5.3.29. Appl., 24.4.22).—A crucible is composed of tungsten powder sintered to tungsten wool.

J. A. SUGDEN.

Compound for hardening metals. W. MORRELL (B.P. 307,233, 5.4.28).—A carburising mixture consists of equal parts of rock salt and potassium ferrocyanide, together with naphthalene or camphor.

C. A. KING.

Manufacture of lead-antimony alloys for use in sheathing electric cables etc. STANDARD TELEPHONES & CABLES, LTD. From MATÉRIEL TÉLÉPHONIQUE (B.P. 307,543, 10.12.27).—An alloy containing 98.5—99% Pb, 1.2%—0.8% Sb, and 0—0.5% Cu is claimed. The copper and antimony are added in the form of an alloy containing 86% Pb, 13% Sb, and 1% Cu.

A. R. POWELL.

Production of grey cast iron. H. HANEMANN, Assr. to EDELGUSSVERBAND G.M.B.H. (U.S.P. 1,705,972, 19.3.29. Appl., 27.5.26. Ger., 25.11.25).—See B.P. 262,043; B., 1927, 819.

Production of zinc. C. MATZEL, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,705,128, 12.3.29. Appl., 13.7.26. Ger., 20.7.25).—See B.P. 255,482; B., 1927, 448.

Hydrometallurgically treating material containing lead and zinc values. W. H. CORBOULD (U.S.P. 1,706,143, 19.3.29. Appl., 31.3.26. Austral., 28.7.25).—See B.P. 273,420; B., 1927, 658.

[Gyratory] fore-hearths for cupola furnaces. L. F. C. GIRARDET (B.P. 286,295, 21.2.28. Fr., 4.3.27).

Electrodeposition of chromium (U.S.P. 1,705,954).—See XI.

XI.—ELECTROTECHNICS.

Behaviour and effects of chlorine as an impurity in the lead accumulator. J. T. CRENNELL and A. G. MILLIGAN (Trans. Faraday Soc., 1929, 25, 159—165).—The addition of hydrochloric acid corresponding with 0.8 g. of chlorine to a lead accumulator results in the reduction of the lead peroxide of the positive plate to lead sulphate. The chlorine liberated then diffuses to the negative plate, the spongy lead of which is oxidised to lead sulphate while the chlorine is reduced to hydrochloric acid and is available for another cycle (cf. B., 1927, 528). The rate of self-discharge thus produced was calculated approximately from Fick's diffusion law on the above assumptions, but the value obtained (0.03 amp.-hr./hr.) was only half that observed, probably on account of the capacity loss due to the formation of lead sulphate on the surface of the plates. The chlorine is removed slowly by evaporation, or rapidly by charging for about 3 cycles. Wooden sheet separators may be bleached or disintegrated, but the effect on the life of the plates is small though perceptible. J. GRANT.

Effect of copper as an impurity in the lead accumulator. J. T. CRENNELL and A. G. MILLIGAN (Trans. Faraday Soc., 1929, 25, 165—168).—Additions of up to 0.1 g. of copper (as copper sulphate dissolved in 7.5*N*-sulphuric acid) to the lead accumulator have only negligible effects on the rates of sulphation and capacity loss. Comparison of the hydrogen overvoltage at a pure copper surface with the *E.M.F.* of the lead-lead sulphate reaction indicates that local action at the negative plate in the presence of copper is negligibly slow. J. GRANT.

Interfacial tension measurements in the examination of insulating oils. J. C. EDWARDS (J. Sci. Instr., 1929, 6, 90—95).—A semi-automatic device is described for obtaining relative values of the interfacial tension between oils and dilute acid solutions. Drops of acid, formed under a constant-pressure head, at the end of a capillary tube dipping under the surface of the oil, fall through the oil, making contact on the way between a pair of lead electrodes. The electrodes are connected, through a relay, to an automatic counter, so that the number of drops of the acid formed from a given volume may be determined, and hence the interfacial tension between the oil and acid solution. It was

found that the electrical stress between the electrodes was without effect on the results, which were reproducible to within 1.5%. Subject to the elimination of kinetic energy effects in the formation of the drops, the pressure head was not critical. The method has been applied to a study of the deterioration of oils through heating to 115° in an open vessel. The interfacial tension falls in a curve roughly hyperbolic over a period of 7 days. F. G. TRYHORN.

See also A., April, 385, **Superconductors** (DE HAAS). 388, **Magnetic properties of iron-copper alloys** (KUSSMANN and SCHARNOV). 402, **Potential of the nickel electrode** (HARING and BOSCHE). **Hydrogen overvoltage of alloys** (RAEDER and EFJESTAD). 404, **Decomposition of nitrous oxide in the silent electric discharge** (JOSHI). 406, **Formation of hydrazine from ammonia by electrical discharges** (BREDIG and others). **Quantitative electrolytic reduction of nitric acid** (NIETZ). 413, **Simple reference electrode for potentiometric titrations** (WILLARD and BOLDYREFF).

Steels for magnets. STOGOFF and MESSKIN.—See X.

PATENTS.

Electric furnaces. J. C. WOODSON [A], and O. A. COLBY [B], Assrs. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,705,697 and 1,705,717, 19.3.29. Appl., [A] 20.10.27, [B] 7.11.27).—(A) Means for handling a sinuous resistance heating element for insertion into or removal from an electric furnace are described. (B) Resistance elements covering the floor and side walls are supported upon a frame having wheels running upon a track, so that the resistors can be removed as a unit from the furnace. J. S. G. THOMAS.

Electric furnace. R. J. WALKER (U.S.P. 1,706,010, 19.3.29. Appl., 11.6.28).—A grid, having adjacent plane sides at right angles and opposite plane sides parallel, is arranged with its sides inclined to the furnace wall. J. S. G. THOMAS.

Electric resistance furnace. BIRMINGHAM ELECTRIC FURNACES, LTD., and A. G. LOBLEY (B.P. 308,009, 11.1.28).—Zig-zag or flat-coil resistance units are directly supported by integral or attached ribs or beadings projecting from the faces of the lining bricks or blocks of the furnace, and form channellings or housings to contain the resistance units, in such manner that practically the whole surface of the resistance units radiates heat. J. S. G. THOMAS.

Temperature-responsive magnetic material. I. F. KINNARD, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,706,172, 19.3.29. Appl., 11.11.25).—The temperature-permeability characteristic of a copper-nickel alloy containing 40—20% Cu, 60—80% Ni, and a small amount of iron approaches unity at about 100° and increases almost uniformly with decrease of temperature below 100°. J. S. G. THOMAS.

Electrical insulating material. W. S. SMITH, H. J. GARNETT, and H. C. CHANNON (B.P. 307,390, 2.9.27).—Cable insulation is prepared by dissolving gutta-percha and/or balata in a suitable solvent and removing dirt by decantation or filtration. If desired, resins may be

extracted by means of a solvent, and an antioxidant, *e.g.*, tannin, may be added to the purified product.

J. S. G. THOMAS.

Insulating materials [for under-water cables etc.] and their production. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 307,966, 15.12.27).—Rubber is treated with water at above 100°, *e.g.*, 125°, for sufficient time to cause at least 99.9% of the nitrogenous matter in the rubber to be removable by subsequent washing. A sample of the heat-treated material, 1.27 mm. thick, after immersion for 4 weeks in a 3.5% solution of sodium chloride, should have a dielectric constant not less than 3 and a specific conductance not more than 20×10^{-6} microhm⁻¹ cm.⁻¹ when subjected to an alternating current of 1000 cycles/sec.

J. S. G. THOMAS.

Preparation of luminescent tubes. P. F. J. LEBRUN (U.S.P. 1,704,981, 12.3.29. Appl., 26.8.27).—A high-tension current is passed through a rarefied mixture of air and a conducting alcoholic vapour before the tube is filled with luminescent gas.

J. S. G. THOMAS.

Röntgen tube. F. ECKERT, Assr. to SENDLINGER OPTISCHE GLASWERKE G.M.B.H. (U.S.P. 1,703,391, 26.2.29. Appl., 5.8.24. Ger., 13.8.23).—To enable Röntgen tubes to be easily fashioned, they are made of easily fusible glass containing a rare earth.

F. G. CLARKE.

Process for securing good electrical contact with crystalline cuprous oxide. A. K. CROAD. From HANOVIA CHEM. & MANUF. CO. (B.P. 307,962, 14.12.27).—Finely-divided surfacing metal or metals, *e.g.*, silver, copper, nickel, is deposited on the surface of the crystal from a solution or an anode composed of the metal to be deposited. Thus, *e.g.*, the crystal covered with iron dust is placed in a solution of copper sulphate whereby copper is deposited on the crystal. Alternatively, metallic nickel may be deposited on the electrolytically reduced surface of the crystal to form a nickel bronze.

J. S. G. THOMAS.

Electrolytic deposition of chromium. R. AUERBACH, Assr. to CHROMEPLATE, INC. (U.S.P. 1,705,954, 19.3.29. Appl., 6.1.27).—Freshly made silicic acid is added to a solution of chromic acid and the solution electrolysed.

J. S. G. THOMAS.

Electrolytic cell. W. E. KERSHAW and J. L. WOODRIDGE (B.P. 307,452, 8.12.27).—Electrodes of nickel or nickel alloy, devoid of active material, are immersed in a solution of an alkaline hydroxide, *e.g.*, sodium hydroxide, to which a soluble chromate may be added.

J. S. G. THOMAS.

[Cleaning electrodes of] electrolytic devices. H. O. SIEGMUND, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,705,944, 19.3.29. Appl., 2.9.26).—Electrodes, after immersion in an unsaturated solution of potassium hydroxide and rinsing with water, are immersed in a bath composed of equal parts of dilute nitric and hydrofluoric acids and rinsed in water.

J. S. G. THOMAS.

Preparing lead electrodes for electric storage batteries. I. G. FARBERIND. A.-G. (B.P. 291,032, 23.5.28. Ger., 24.5.27).—Admixed metal is eliminated from plates consisting of lead alloy supported in a lead

grating, by electrolysis at ordinary temperatures in sulphuric acid of *d* 1.10, employing a current density of about 1.2 amp./dm.²

J. S. G. THOMAS.

Preventing explosive gas mixtures from being formed in the casings of electrical apparatus enclosed in an insulating medium. M. BUCHHOLZ (B.P. 307,570, 2.1.28).—Vapours or gas mixtures formed within the casings actuate electrical devices which control plant for supplying air to, or exhausting air from, the casing, switch off the current, and actuate a visible or audible signal.

J. S. G. THOMAS.

[Apparatus for] lead-covering of electric cables etc. by extrusion. W. T. HENLEY'S TELEGRAPH WORKS CO., LTD., and E. E. JUDGE (B.P. 308,521, 2.6.28).

Electric discharge tubes. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 307,982 and 308,879, 21.11.27 and 5.3.28).

Electric luminous tubes. Devices for production of ultra-violet radiation. N. V. INTERNAT. OCTROOIBUREAU, Assees. of F. MEYER, H. J. SPANNER, and E. GERMER (B.P. 282,064, 6.12.27, and 285,068, 7.12.27. Ger., [A] 10.12.26, [B] 11.2.27).

Negative secondary battery plates. D.P. BATTERY CO., LTD., and J. WADDELL (B.P. 308,283, 21.9.27).

Gas producer (U.S.P. 1,703,505). Photochemical gas reactions (B.P. 307,406 and 307,521). Refining mineral oils (U.S.P. 1,700,347).—See II. Apparatus for melting (U.S.P. 1,703,658). Treatment of iron pyrites (B.P. 307,188 and 307,190). Alloys for cables etc. (B.P. 307,543).—See X. [Liquid] insulating material (B.P. 293,293).—See XIII.

XII.—FATS; OILS; WAXES.

Splitting of fats. G. PETROV and N. SOKOLOV (Oil Fat Ind., Russia, 1928, No. 1, 28—30).—After agitation with steam in presence of sulphuric acid (5%, 25% of the fat) and zinc, aluminium, and sodium sulphates (total, 10% of acid), linseed oil, sunflower oil, animal fat, and hydrogenated vegetable oil were saponified with "kontakt" (2%) and sulphuric acid (0.2%), the emulsions being broken up by adding 0.2—0.5% of calcium sulphate and agitating with steam for 10—15 min. For linseed and sunflower oils the transparency is increased by the preliminary treatment, but the colour is not improved; for animal fat and hydrogenated vegetable oil the colour was improved.

CHEMICAL ABSTRACTS.

Bleaching vegetable and mineral oils and animal fats with clay. I. S. ELAKOV (Trans. State Exp. Inst. Silicates, Moscow, 1927, No. 21, 67—72).—The adsorptive power of clay is unconnected with the presence of magnesia. Crystalline clays are less efficient than colloidal clays; fine clays have a high decolorising power. Clays suspended in water are more efficient than the original clays. The water in clays is a hindrance in their use as decolorising agents.

CHEMICAL ABSTRACTS.

Fats. X. Thiocyanometric examination of fats containing linolenic acid. Analysis of linseed oil. H. P. KAUFMANN and M. KELLER (Z. angew. Chem.,

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1929, 42, 73—76; cf. B., 1929, 135).—Addition of thiocyanogen takes place at only two of the three double linkings of linolenic acid, the end-point being, however, sharp, and unaffected by excess of thiocyanogen or prolonged reaction. For the analysis of linseed oil, the sample is hydrolysed in absence of oxygen, and after removal of unsaponifiable matter the pentane solution of the fatty acids is dried and the iodine value (I) of the total fatty acids is determined by the bromometric method using 2 hrs.' and 24 hrs.' reaction. The thiocyanogen value (T) is similarly determined, using 0.1—0.2 g. and a large excess (200%) of acetic acid-thiocyanogen solution but no solvent. For fats containing linolenic acid $N/7$ -5-thiocyanogen solution is recommended. The proportion of saturated acids (S) is now determined by Bertram's method, and the composition of the total fatty acids is calculated from the approximation equations: Oleic acid (O) = $(100 - S) - 1.104(I - T)$; linoleic acid (L) = $(100 - S) - 1.104 \times (2T - I)$; linolenic acid (Ln) = $-(100 - S) + 1.104T$, in which $S + O + L + Ln = 100$. Similarly, the proportion of saturated acids in a mixture of oleic acid, linoleic acid, and saturated acids is given by $S + 100 - 1.108T$, or $S = 100 - 1.120T + 0.008I$, and the oleic and linoleic acids are given by $O = 1.112(2T - I)$, $L = 1.104(I - T)$. The mixture of linoleic acid and linolenic acid obtained from Calcutta linseed oil after separating oleic acid as lithium oleate from the liquid fatty acids and fractionation of the remaining lithium salts gave iodine value 235 and thiocyanogen value 146, from which the composition of the mixture was calculated as linolenic acid 58.2%, linoleic acid 41.8%, and linolenic acid 60.3%, linoleic acid 39.7%, respectively; isolinolenic acid also appears to combine with only 2 mols. of thiocyanogen. R. BRIGHTMAN.

Rapid determination of water in fats and glycerin. V. TSCHERNUISHEV (Oil Fat Ind., Russia, 1928, No. 1, 17—18).—The fat is pressed on a weighed filter paper, when the water is absorbed; the sample and paper are dried at 105°. Glycerin is poured on to a weighed filter paper, dried at 80°, powdered sodium sulphate is added, and the whole is extracted in a thimble with dry acetone. CHEMICAL ABSTRACTS.

Iodine value in relation to origin and age of olive oil. E. DE'CONNO and D. RAGO (Annali Chim. Appl., 1929, 19, 98—107).—Of olive oils from three different sources, those from Lucca have higher iodine values (103) than those from Sorrento (102), and still higher than those from Bari (97). Thus the proportion of glycerides of unsaturated acid in the oil varies with the quality of the soil and with the climatic conditions. The iodine value falls if the oil is kept, the diminution being marked during the six months following extraction, there being a tendency to assume a stable limiting value. T. H. POPE.

Fruits and seeds of *Aleurites Fordii* from Kenya Colony. ANON. (Bull. Imp. Inst., 1929, 27, 10—12).—The seeds produced in Kenya from those of Chinese origin closely resemble the latter and give a similar or slightly higher yield of tung oil of the same character. F. R. ENNOS.

Fruits and seeds of *Hydnocarpus Woodii* from

North Borneo. ANON. (Bull. Imp. Inst., 1929, 27, 12—16).—The oil from *Hydnocarpus Woodii* seeds contains the glycerides of both hydnocarpic and chaulmoogric acids, in this respect resembling the oil from the seeds of *H. Wightiana*. F. R. ENNOS.

Oil from *Carthamus tinctoris* (safflower oil). J. ZUKERVANIK (Acta Univ. Asiæ Med., 1928, 6, 3—19).—The unshelled seeds from *C. tinctoris* grown in Mid-Asia contain up to 25% of oil having the following composition: linolenic and isolinolenic (0.0—0.5%), linoleic (39—50%), oleic (34—37%), palmitic (5—6%), and stearic (3—4%) acids, glyceryl residue (C_3H_7) 4—6%, and unsaponifiable matter (0.5—1.0%). The physical and chemical constants differ only slightly from those of safflower oils from other parts of the world, and the oil can be put to the same economic uses. Oil from the shelled seeds is suitable for edible purposes. Detailed experiments on exposure of the oil to air and light under varying conditions showed that the iodine value decreased; the acetyl value, acid value, and refractive index increased; the viscosity increased; and the colour was bleached. Oxidation is slower in the dark, in the absence of air, and at low temperatures. The Meigen-Neuberger thallium salt method for the separation of fatty acids gave much better results than the Varrentrapp process. E. H. SHARPLES.

Antirachitic factor in burbot-liver oil. B. CLOW and A. MARLATT (Ind. Eng. Chem., 1929, 21, 281—282).—Burbot-liver oil is an excellent source of the antirachitic vitamin, being eight times as potent as cod-liver oil. It is not known whether this difference is due to the inherent property of the oils or to a variation from one sample to another. F. R. ENNOS.

Application of castor oil in soap manufacture. N. SPASSKI (Masloboino Zhir. Delo, 1928, No. 2, 34—35).—The use of castor oil increases the lathering power of soap and its solubility in cold water. The necessary increase in the concentration of the salting-out electrolyte decreases the proportion of water in the soap. CHEMICAL ABSTRACTS.

Oxidation and polymerisation of ethyl esters from linseed acids. G. PETROV and N. SOKOLOV (Oil Fat Ind., Russia, 1928, No. 2, 35—36).—Ethyl esters of the products of the splitting of linseed oil with "kontakt" were distilled with steam. The fraction 210—230° had acid value 14.0, iodine value 194.7, saponif. value 188.44, and d 0.8921. Comparative drying experiments were performed with linseed oil, fatty acids, and ethyl esters. The formation of a linoxyn skin is due to the presence of glycerol and unsaturated acids. CHEMICAL ABSTRACTS.

Sulphonated oils. II. Hydrolysis of ricinoleo-sulphuric acid or its sodium salt. K. NISHIZAWA and K. WINOKUTI (Chem. Umschau, 1929, 36, 97—102).—The hydrolysis of the pure sodium ricinoleo-sulphate (cf. B., 1929, 363) was systematically studied. 0.1M-Aqueous solutions of the salt are hydrolysed to the extent of 2.86% and 18.54% after 5 hrs. at 75° and 100° respectively. 0.1M-Aqueous solutions were quite stable for 24 hrs. in the presence of equal volumes of solutions of hydrochloric acid of 0.1N—10N at 25°;

physical differences in appearance, viscosity, etc. were, however, noticed. At 100° the salt is fairly rapidly attacked by hydrochloric acid, the rate depending on the concentration; complete hydrolysis was effected by equal volumes of *N*- and 2*N*-hydrochloric acid in 35 and 20 min. respectively; the corresponding times of hydrolysis by sulphuric acid were 50 and 30 min., and the use of this acid for the determination of the organic sulphate by the volumetric method is recommended as satisfactory provided suitable concentrations are chosen and the boiling is sufficiently prolonged. Solutions of the sodium ricinoleosulphate are stable to aqueous alkali at the ordinary temperature; at 100° the stability decreases with dilution of the alkali; the degree of saponification, however, produced by alcoholic caustic soda increases with increasing concentration of the reagent.

E. LEWKOWITSCH.

Determination of organically combined sulphur in sulphonated oils. K. H. BAUER (Chem. Umschau, 1929, 36, 102). Further experiments confirm that determinations of organic sulphate in sulphonated oils by the American volumetric method agree with those made by Herbig's gravimetric method; sulphuric acid effects complete hydrolysis, but the boiling must be more prolonged than when hydrochloric acid is used, corresponding to the difference in hydrogen-ion concentration of the two acids (cf. Nishizawa and Winokuti, preceding abstract).

E. LEWKOWITSCH.

Determination of neutral fat in sulphonated oils. R. HART (J. Amer. Leather Chem. Assoc., 1929, 24, 120—121).—The neutral fat may be calculated from the saponification value determined in the usual way. The alcoholic solution of a weighed amount of the sulphonated oil is rendered neutral to phenolphthalein before saponification. The amount of organically combined sulphur trioxide eliminated by this saponification is negligible. The neutral fat is expressed as oleic acid. Good agreement is shown by comparative tests on 10 samples using the saponification method and the difference method.

D. WOODROFFE.

See also A., April, 417, **Extraction apparatus** (GAŠPERÍK). 423, **Products of partial hydrogenation of higher mono- and poly-ethylenic esters** (HIL-DITCH and VIDYARTHI). 440, **Reactions of cholesterol** (BAHL). 476, **Oxidative capacity and peroxidic-oxygen content of cod-liver oil** (DELORE). 477, **Jute seed oil** (SEN). [Fat from] **rind of California oranges** (MATLACK). 478, **Microscopical differentiation of neutral and hydrolysed fat** (HEUPKE).

Sunflower seed as feeding-stuff. WEISER and ZAITSCHEK.—See XVI. **Germicidal soaps.** HAMPIL.—See XXIII.

PATENTS.

Treatment of heavy vegetable oil. H. S. ADAMS and L. MEUSER, Assrs. to NAUGATUCK CHEM. CO. (U.S.P. 1,697,337, 1.1.29. Appl., 13.1.26).—Castor oil flows slowly and continuously in a thin stream through an inclined nichrome tube heated at 450—500°. Yields of 25% of crude 85% heptaldehyde are obtained. Other material inert to heptaldehyde at 450° may be used for the cracking tube. Undecenoic acid is separated by steam-distillation of the condensate. R. BRIGHTMAN.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Nitrocellulose finishes. B. CAMPBELL (J. Oil & Colour Chem. Assoc., 1929, 12, 61—81).—A comprehensive dissertation on nitrocellulose lacquers, their historical development, raw materials used therein, considerations involved in their formulation, and the effects on related chemical fields of their development.

S. S. WOOLF.

Relation of composition to properties of lacquer solvents. R. CALVERT (Ind. Eng. Chem., 1929, 21, 213—215).—Nitrocellulose solvents are usually oxy-compounds containing carbonyl, hydroxyl, and other groups, and these are increasingly incompatible with resins with increase in oxygen content. Blending with hydrocarbon resin solvents and oxidation of the resin are expedients adopted to surmount this difficulty. The hydrolysis of esters is discussed from the viewpoint of the liberation of undesirable acid content in lacquers, and it is pointed out that an ester of a strong acid may be expected to hydrolyse more rapidly in a lacquer than an ester of the same alcohol with a weaker acid.

S. S. WOOLF.

Lifting of varnishes by lacquer solvents. H. E. HOFMANN and E. W. REID (Ind. Eng. Chem., 1929, 21, 247—249).—The lifting effects of one-, two-, and three-component solvent and diluent mixtures on varnish films of various types and ages were noted. In the hydrocarbon group, petroleum distillates cause least lifting, whilst of alcoholic solvents the lower members are the most desirable from this point of view, but small quantities of less volatile members of lifting tendency have nevertheless to be used. Short-oil varnishes are less affected by lacquer solvents than are long-oil varnishes. Little difference was noted between the lifting of linseed oil and tung oil varnishes by lacquer solvents, although linseed oil varnishes were more readily lifted by alcoholic solvents and less readily affected by hydrocarbons and ester solvents than tung oil varnishes. Over a period of three months, ageing was not found to have the anticipated effect of decreasing lifting by lacquer solvents, but more prolonged tests are deemed advisable. A brushing lacquer incorporating a solvent mixture of very little lifting tendency is formulated.

S. S. WOOLF.

Pyroabietic acid from French rosin. E. FONROBERT and A. GRETH (Chem. Umschau, 1929, 36, 93—97).—French rosin was heated at 250° for 168 hrs. and then distilled *in vacuo* (cf. Dupont and Dubourg, Bull. Inst. Pin, 1928, 51, 181), giving a 50% yield of crude pyroabietic acid (distilling at 200—225°/2—2.5 mm.) in the form of a pale yellow resin, which crystallised from acetone in white prismatic needles, m.p. 172—177°, rising continuously on recrystallisation. By careful repeated recrystallisations from acetone and alcohol this product was separated into two isomerides (about 90% of *d*-acid). The less soluble acid (*l*-pyroabietic acid) showed the properties of a monocarboxylic acid, and had m.p. 194°, $[\alpha]_D^{25}$ —15.2°, saponif. value 184.3, and a constitution corresponding to $C_{20}H_{30}O_2$. The more fusible, more soluble dextrorotatory "*d*-pyroabietic acid" corresponded closely with Dupont and Dubourg's pyroabietic acid and had m.p. 156—158°, $[\alpha]_D^{25}$ +37.5°.

saponif. value 184.4. The iodine value (Hanus) of a mixture of the acids (m.p. 168°) was 137.4. The m.p. of mixtures of the *d*- and *l*-acids lie between the values for the pure individuals, and since only very small, or no, depression of the m.p. was observed in mixtures of these acids with ordinary abietic acid from French rosin, these three acids must have similar structures. Examination of specimens of Dupont's acid and of the crystalline deposit from old rosin oils (French) revealed the presence of some *l*-pyroabietic acid.

E. LEWKOWITSCH.

Turpentine oil from larch turpentine. SCHIMMEL & Co. (Ber. Schimmel, 1928, 101; Chem. Zentr., 1928, ii, 1827).—Turpentine from *Larix decidua*, Miller, yielded on distillation in steam an oil (14.6%) having d^{15}_4 0.8661, $\alpha_D - 9^\circ 32'$, n^{20}_D 1.47220, acid value 0, ester value 7.5. A larch turpentine oil from the Tyrol had d^{15}_4 0.8637, $\alpha_D - 12^\circ 3'$, n^{20}_D 1.46717, acid value 0, ester value 1.9.

A. A. ELDRIDGE.

Elemi oil. SCHIMMEL & Co. (Ber. Schimmel, 1928, 24; Chem. Zentr., 1928, ii, 1827).—The oil (15.9%) from Columbian Carana-elemi resin had d^{15}_4 0.9683, $\alpha_D + 18^\circ 34'$, n^{20}_D 1.50448, acid value 5.0, ester value 22.4, ester value after acetylation 42.9. The first runnings of the oil contained phellandrene.

A. A. ELDRIDGE.

Nature and constitution of shellac. I. W. H. GARDNER and W. F. WHITMORE (Ind. Eng. Chem., 1929, 21, 226—229).—The best solvents for shellac are the alcohols, organic acids, and ketones, indicating the presence of hydroxyl, carboxyl, and carbonyl groups in the resin. The apparent tendency to give colloidal solutions in these solvents and the lack of solubility in most other solvents suggest a complex molecular aggregation in the solid state and a high mol. wt. The colloidal nature of shellac and its solutions is manifested by swelling that accompanies preliminary dissolution with many solvents, the rapid rise in viscosity with concentration of solution, its adhesive properties, etc.

S. S. WOOLF.

See also A., April, 449, **Indian turpentine from *Pinus longifolia*, Roxb.** (GIBSON and SIMONSEN).

Selenium-red as ceramic colour. POLGREAN.—See VIII. Hop resins. WINDISCH and others; also WÖLLMER.—See XVIII.

PATENTS.

Manufacture of varnish. B. F. WILLIAMSON and W. H. BEISLER (U.S.P. 1,699,646, 22.1.29. Appl., 30.12.25).—The strained gum thus, e.g., 100 pts., is mixed with 3 pts. of glycerol and 150 pts. of tung oil or other drying oil and heated in about 3 hrs. to 150°. Steam or other non-oxidising gas is then passed through the mixture and heating is continued at 275—335° until the proper consistency is obtained, the passage of steam being maintained until the temperature reaches 200°, when 200 pts. of turpentine or other solvent or thinner are added and any desired drier.

R. BRIGHTMAN.

Removal of paint and varnish. J. H. GRAVELL (U.S.P. 1,700,491, 29.1.29. Appl., 15.4.26).—A mixture, by vol., of 55 pts. of cresylic acid and 10 pts. of sulphuric acid, alone, or mixed with 10 pts. of butyl alcohol or

with 10 pts. of butyl alcohol and 25 pts. of tetrachloroethane, is used at 120—125°.

R. BRIGHTMAN.

Insulating material [resistant to heat, moisture, and electricity]. ELEKTRA-LACK-WERKE G.M.B.H., SYSTEM DR. KRONSTEIN (B.P. 293,293, 3.1.28. Ger., 2.7.27).—Substantially liquid insulating material, e.g., oil varnish, rubber solution, celluloid varnish, artificial resin, is applied to a solid carrier of transparent, regenerated cellulose obtained from viscose and the coating is dried and solidified at any desired temperature.

J. S. G. THOMAS.

Synthetic resins. GRINDLEY & Co., LTD., and R. L. YEATES (B.P. 306,924, 30.11.27).—A mixture of polyhydric alcohols, polybasic acids or their anhydrides, acid resins, and/or fatty acids derived from non-drying oils, drying oils, or fats is heated, e.g., at 200—270°. If products of low acid value are required, basic materials, e.g., oxides, hydroxides, or suitable salts of aluminium, calcium, zinc, etc., are added.

S. S. WOOLF.

Production of condensation products from phenols and aldehydes. KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 288,228 and 290,963, [A] 15.11.27, [B] 3.11.27. Austr., [A] 6.4.27, [B] 21.5.27).—(A) The condensation of phenols and aldehydes is carried out in the presence of protective colloids, e.g., gum arabic, gelatin, at a dilution favourable to the formation of a powdery precipitate. (B) The colloidal resin solution prepared in accordance with B.P. 267,901 (B., 1928, 720) is freed from water by distillation in the presence, if desired, of organic solvents, e.g., glycerin, glycol, to such an extent that a transparent, opalescent material results. Glass-clear materials may be obtained if the bases in the initial condensation are neutralised by suitable acids, e.g., polycarboxylic acids, hydroxy-acids, sulphonic acids, of dissociation constant 10^{-2} — 10^{-5} and having alkali salts easily soluble in water.

S. S. WOOLF.

Manufacture of phenolic condensation products. A. J. WEITH, ASSR. to BAKELITE CORP. (U.S.P. 1,699,727, 22.1.29. Appl., 25.5.23. Renewed 11.4.28).—Fusible phenolic-methylene resin is incorporated with phenol to give a ratio of 9—12 phenol groups to 6 methylene groups, and formaldehyde or other substance containing methylene (with preferably 0.1% of ammonia or other basic catalyst) is added to establish a phenol/methylene ratio of 1:1.

R. BRIGHTMAN.

Manufacture of white lead pigment. S. W. KENDALL (U.S.P. 1,706,301, 19.3.29. Appl., 24.9.24).—See B.P. 240,424; B., 1925, 999.

Production of condensation products of [hydroxymethyl] methylol compounds of a urea. M. LUTHER and C. HEUCK, ASSRS. to I. G. FARBERIND. A.-G. (U.S.P. 1,699,245, 15.1.29. Appl., 2.3.27. Ger., 27.2.26).—See B.P. 266,752; B., 1928, 762.

Preventing discoloration of canned foods (U.S.P. 1,699,274).—See XIX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

See also A., April, 394, **Effect of temperature on viscosity and ease of precipitation of rubber** (WHITBY and GALLAY). 415, **Determination of barium**

sulphate by solutions of sodium carbonate (WOLESENSKY). 434, Polymerisation of α -methylstyrene (STAUDINGER and BREUSCH).

PATENTS.

[Rubber] mucilage for uniting bodies or pieces of material of the same or of different kinds. H. O. BRUHN (B.P. 306,864, 24.11.27 and 29.6.28).—A solution of rubber in a chlorinated hydrocarbon is incorporated with a perfume and sufficient of a saturated solution of an inorganic or an organic salt, e.g., aluminium sulphate or sodium propylnaphthylsulphonate, to increase the viscosity of the paste. F. R. ENNOS.

Insulating material (B.P. 307,390 and 307,966).—See XI.

XV.—LEATHER; GLUE.

Tizerah wood and extract. W. VOGEL (Collegium, 1929, 68—74).—Tizerah (*Rhus pentaphylla*) is a thick bushy tree about 10 ft. high and 16—20 ft. in circumference, found chiefly in Northern Africa. Both bark and leaves are used by the natives for tanning and dyeing morocco leathers, but their tannin content is less than that of the wood derived from the upper parts of the root and the main branches. About 65,000 tons of tizerah wood are produced annually. The flesh portion is dark reddish-brown and the heartwood leather-brown in colour. On analysis, the wood and liquid and solid extracts, respectively, gave (%): tans 22.4, 32.1—41.3, 69.9; non-tans 2.2, 4.0—4.7, 5.4; insoluble matter 60.9, 0.3—1.0, 2.7; water 14.5, 53.3—62.7, 22.0; ash 1.8, —, 1.0. Bright coloured root wood contains most tannin. Tizerah is a pyrocatechol tannin, containing dark brown phlobaphens and closely resembling quebracho in its reactions (cf. B., 1927, 853). It yields a slightly darker coloured leather than quebracho. Tizerah liquors are not so readily clarified as those of quebracho, and should not be sulphited. Tizerah can be used to replace quebracho.

D. WOODROFFE.

South African tanning materials. Black wattle. C. O. WILLIAMS (Union S. Africa Dep. Agric. Sci. Bull., 1928, No. 63, 68 pp.).—The composition and properties of the bark of *Acacia mollissima* have been investigated. Bark from the larger branches of mature trees or the top portion of the main stem contains about 30%, whilst the twigs and leaves contain only about 4%, of tannin. The effects of drying, exposure to rain, and moulds are described. Solid wattle extract contains about 60% of tannin. The ash of spent wattle bark contains approx. 70% CaCO_3 , 4% K_2O , and 1% P_2O_5 .

CHEMICAL ABSTRACTS.

Adsorption and tanning properties of synthetic tannins. J. BERKMANN (Collegium, 1929, 49—54).—Synthetic tannins contain sulphonic acids (completely absorbable), mineral salts (non-absorbable), and salts of sulphonic acids which are freely and characteristically absorbed by hide powder, the adsorption of which varies with different products and depends on the metal present in the following order of increasing adsorption: sodium, potassium, ammonium, barium, calcium, and magnesium. Iron, chromium, aluminium, and copper salts are adsorbed to a very much greater degree almost

equal to that of the free sulphonic acids. Solutions of salts of condensed aromatic sulphonic acids are semi-colloidal and do not readily ionise. Hide substance shows selective adsorption for the organic part of the sulphonic acid salt. The adsorptivity is almost proportional to the complexity of the product from which the synthetic tannin is derived. The actual condensation process has a greater effect on the adsorptivity of products derived from raw materials of high mol. wt. The adsorption by hide powder increases to a maximum and then diminishes as the amount of aldehyde used for the condensation process increases. Very little formaldehyde is required to produce maximum adsorption with the salts of sulphonated higher hydrocarbons. The adsorption diminishes more rapidly with dilution of condensed tannins than of uncondensed. Judged by the irreversibility of the tannage, synthetic tannins are good tanning materials. The adsorbed sulphonic acids cannot be washed out and render the leather quite stable, but their salts are more easily, though incompletely, removable by washing. Since synthetic tannins tan in acid solution only, it is evident that the desired mechanical properties are imparted by the sulphonic acids and not by their salts.

D. WOODROFFE.

Determination of insoluble matter in tanning extracts. A. TURNBULL (J. Amer. Leather Chem. Assoc., 1929, 24, 121—124; cf., B. 1928, 534).—A non-plastic clay is prepared by grinding white fireclay brick to pass 100-mesh, then digesting it with a mixture of 90 pts. of hydrochloric acid and 10 pts. of nitric acid. After thorough washing it is dried, and 90 pts. are mixed with 10 pts. of fine acid-washed natural kaolin. This mixture affords a suitable filtering layer on the porous septum previously described. An improved filtering apparatus is described in which a Schott funnel 17G with porous septum is used. To prepare the filtering layer, a mixture of 2 g. of the non-plastic clay with 75 c.c. of tan liquor is poured on to the septum and filtered till clear. The Schott funnel is fitted into the filtering funnel, tan liquor filtered through the latter into the flask until clear, and then 60 c.c. are collected. Comparative figures are given for insoluble matter determined by the porous septum and the old and new official methods respectively.

D. WOODROFFE.

Sanio's potassium dichromate test for tannins. C. M. FEAR (Analyst, 1929, 54, 227).—No reliance is to be placed on this test (cf. Botan. Ztg., 1863, 17).

D. G. HEWER.

Tannery waters. E. MEZEY (Collegium, 1929, 55—68).—Titration curves have been determined for five different waters, viz., (a) distilled, (b) river, (c) conduit, (d) well, and (e) water containing nitrogenous matter. Samples (b)—(e) were of waters used by tanneries, and of which (b) and (c) contained only 5° of hardness, respectively. The suitability of a water for tannery purposes can be judged from its buffering effect, as shown by these titration curves. The flatter are the curves, the greater is the buffering effect. The greatest buffering effect was shown by sample (e).

D. WOODROFFE.

Hydration of animal skin by the volume-change method. E. R. THEIS and H. A. NEVILLE (Ind. Eng. Chem., 1929, 21, 377—379).—Pieces of corium of fresh

and salted steer hide were placed in a dilatometer, covered with 0.1*N*-, 0.01*N*-, and 0.001*N*-hydrochloric acid and water, respectively, and the changes in volume noted over a period of 48 hrs. and also the time required to establish equilibrium. The order of increasing contraction of volume of fresh hide was 0.01*N*-acid, water, 0.001*N*-acid, and 0.1*N*-acid. There was an initial increase in volume with 0.1*N*-acid and then a gradual contraction. With cured hide there was first a contraction in volume with water and all acids (except 0.1*N*), then an apparent increase for 36 hrs., and finally a further decrease in volume. With 0.1*N*-acid an initial increase was followed by a gradual decrease in volume. The initial contraction in volume of cured hide is attributed to hydration. After several hours the proteins in cured hide degenerate, bacterial action sets in, and the hydration capacity is less than before, hence the increase in volume. Acid solutions of $p_H < 2$ diminish the bacterial action. Cured hide when soaked for 24 hrs. in water and then placed in milk of lime gave an initial contraction in volume in the soak, with subsequent increase. This diminution in volume was caused by the lime liquor checking bacterial action; equilibrium was attained in 24 hrs. These results show that *post mortem* changes and the effects of cure and of various anions on the hide can be studied by means of their hydration effects. D. WOODROFFE.

Properties of shoe leather. VII. Temper and break. J. A. WILSON (J. Amer. Leather Chem. Assoc., 1929, 24, 112—119).—Methods of measuring "temper" (flexibility and recovery after bending) and break (cracking by bending) are described. The flexibility was much less in the belly than in the butt, very low for kangaroo, kid, and suede leathers, and highest for heavy chrome hide. The percentage recovery varied from 45% to 72%. Splitting the leather reduced the flexibility very much, but had little effect on the recovery factor. "Break" values (number of wrinkles produced) for the belly regions were one half to two thirds of those for the butt regions. Vegetable and chrome-tanned calf skins had the greatest "break" figures 26—28, glazed kid 14, and patent 0. D. WOODROFFE.

Effect of neutralisation of chrome[-tanned] leather on fat absorption. H. B. MERRILL and J. G. NIEDERCORN (Ind. Eng. Chem., 1929, 21, 364—366).—Samples of one-bath chrome-tanned calfskin were neutralised with different amounts of borax and sodium bicarbonate solutions, the p_H value of each solution being noted; the samples were then washed, fat-liquored with soap and neat's-foot oil, dried out, and analysed. In both cases the percentage of fat absorbed decreased with increasing neutralisation. Neutralisation did not affect the penetration of the fat. The fat absorbed by the leather did not depend on the final p_H value of the neutralising solution, but decreased with increasing removal of sulphuric acid from the leather. The effect of the neutralisation on the fat absorption was less pronounced when the time of fat-liquoring was prolonged. D. WOODROFFE.

Consistency of casein glue. F. L. BROWNE and D. BROUSE (5th Coll. Symp. Mon., 1928, 229—242).—Casein solutions containing no lime have a more complex

flow (as determined with a modified Herschel consistometer) than jelly-forming glues containing lime. With the latter no change from viscosity to plasticity after mixing was observed. CHEMICAL ABSTRACTS.

Casein and some of its applications. L. L. VAN SLYKE (N.Y. Agric. Exp. Sta. Tech. Bull., 1928, No. 139, 1—41).—A summary of researches. CHEMICAL ABSTRACTS.

See also A., April, 413, **Colorimetric hydrogen-ion determination** (CAMERON). 458, **Nitrogen distribution of gelatin** (DAFT). **Fractionation of gelatin** (KUNITZ and NORTHROP).

PATENTS.

Manufacture of spinning and weaving fibres [from skins etc.]. R. TANDLER (B.P. 290,154, 6.3.28. Austr., 7.5.27).—Skins etc. are chemically treated with, e.g., lime liquor followed by a salt-hydrochloric acid pickle to loosen and disintegrate the fibres, then rolled or stamped, and/or further treated with chemical substances, e.g., two-bath chrome tanning liquors, aluminium sulphate, etc., greased, the epidermis or grain layer removed, and the product disintegrated by carding, tearing, or combing into the separate fibres. D. WOODROFFE.

Treating fabrics resembling wash leather. H. W. and F. H. TRELEAVEN and W. JANVIER (B.P. 307,189, 10.2.28).—Cloth or fabric, which may or may not have been previously impregnated or painted with a solution of albumin and afterwards treated with a solution of tannic acid, is steeped for 15 min. in a cold mixture of oil of *Pinus Siberica*, glycerin, and water, and then dried. D. WOODROFFE.

Manufacture of hardened casein products. I. G. FARBENIND. A.-G. (B.P. 279,863, 26.10.27. Ger., 26.10.26).—Casein is mixed with glyoxal or one of its homologues or a derivative capable of setting free the glyoxal under the conditions of treatment, to which a colouring or filling material and a clarifying or softening agent may be added. D. WOODROFFE.

Manufacture of mineral acid-free synthetic tanning material. J. SCHÄFER, Assr. to J. R. GEIGY Soc. ANON. (U.S.P. 1,706,325, 19.3.29. Appl., 8.8.27. Ger., 16.8.26).—See B.P. 276,014; B., 1928, 420.

Treatment of leather. A. O. T. BEARDMORE (U.S.P. 1,704,718, 12.3.29. Appl., 24.3.28).—See B.P. 300,077; B., 1929, 30.

XVI.—AGRICULTURE.

Phosphoric acid requirements of Czechoslovakian soils. E. G. DOERELL (Superphosphate, 1928, 1, 128—132).—A study of acidity, and of lime and phosphate requirements. CHEMICAL ABSTRACTS.

Lime and phosphoric acid in the soil. M. A. EGOROV (Trans. Sci. Inst. Fertilisers, Moscow, 1926, No. 34, 37—45).—The greater the amount of carbon dioxide in the soil, the smaller is the effect of acid phosphate. In certain loam soils the phosphate was parallel with iron, and not with calcium. CHEMICAL ABSTRACTS.

Action of water-soluble mono- and di-phosphates on permutite, in connexion with the fixation

of phosphoric acid in the soil. E. BERL and P. SCHMITTNER (*Z. angew. Chem.*, 1929, 42, 351—355).—Permutite takes up phosphoric acid readily from primary (dihydrogen) phosphates, to the extent of 25% of its own weight (as P_2O_5); the action is less in the case of secondary (monohydrogen) salts. The phosphoric acid is taken up mainly as aluminium phosphate, insoluble in water, but completely soluble in citric acid.

S. I. LEVY.

Does frost exert a "solubilising" effect on the phosphate and potash of soils? O. ENGELS (*Forts. Landw.*, 1928, 3, 96; *Bied. Zentr.*, 1929, 58, 52).—Examination by Neubauer's method of soils frozen once, and repeatedly, showed little or no increase in root-soluble phosphate or potash as a result.

A. G. POLLARD.

Actual acidity of the podzol soils and the influence of liming. N. P. REMEZOV (*Trans. Sci. Inst. Fertilisers, Moscow*, 1927, No. 42, 5—26).—Variations in pH in the same field depend on meteorological and biological factors; the fallow plot was more acid. In unlimed plots the acidity increased with increase of nitrate; in limed plots the reverse held. In podzol soil the acidity increases with the depth of the horizons.

CHEMICAL ABSTRACTS.

Causes of plant suffering from over-liming. E. V. BOBKOV, B. A. GOLUBER, and A. F. TYNLIN (*Zhur. Opitn. Agron.*, 1927, 23, 165—202).—Over-liming of mineral soils, especially light forest virgin soils, may cause plant injury owing to intensified biological processes resulting in an increase of hydrogen carbonate, ammonia, nitrite, and nitrate. Similar effects were not observed with heavy soils.

CHEMICAL ABSTRACTS.

Soil respiration and fertility. E. H. REINAU (*Festschr. Stoklasa*, 1928, 305—318; *Bied. Zentr.*, 1929, 58, 107—108).—The intensity of the assimilation of carbon dioxide by green leaves from the air immediately surrounding them is such that practically the whole of the carbon dioxide produced within the soil can be absorbed. The interchange of layers of air at the soil surface with higher layers is small. Hence the view is propounded that carbon dioxide production in soils must control crop yields.

A. G. POLLARD.

Determination of the moisture equivalent of soils and the rôle of soil colloids on this moisture equivalent. G. J. BOUYOUCOS (*Soil Sci.*, 1929, 27, 232—241).—Moisture equivalents are determined by placing the air-dried soil sample in a small Buchner funnel previously covered with a filter paper and connected by way of an ordinary filter flask with a filter pump. The soil is thoroughly moistened and then allowed to drain under suction for (say) 10 min. The funnel is covered with moist cloth to prevent evaporation by suction. Results so obtained are closely related to the colloidal content of the soils as determined by the hydrometer method. No relationship exists between the coarse silt and sand contents and the moisture equivalent. The hydrometer method may be used for the indirect determination of the moisture equivalent, and can give "single-value" factors concerning the physical properties of soils.

A. G. POLLARD.

Determination of the weight of carbon and combined water in the soils of the world. V. AGAFONOV (*Compt. rend.*, 1929, 188, 1000—1001).—Calculations based on Glinka's map of the total weight of carbon and combined water in the soil of various zones give the following figures (in millions of tons), the figures in parentheses being for water: Tundras 73 (314), podzolic 306 (1326), yellow-(Ramann)-brown soils 46 (158), tchernozem 286 (179), reddish-brown soils 80 (135), Sierozem 10 (42), red soil of the Steppe deserts 33 (46), red earth (warm temperate zone) 2 (21), laterites and lateritic soil 126 (15132), mountain soil 87 (377).

R. BRIGHTMAN.

Relation of temperature to the amount of nitrogen in soils. H. JENNY (*Soil Sci.*, 1929, 27, 169—188).—In the soils of the semi-acid, semi-humid, and humid regions of the United States there is a relationship between mean annual temperature and average total nitrogen content of upland prairie and timber soils and of terrace and bottom land soils. Nitrogen contents decrease with increasing temperature according to an exponential law. For every 10° decline in mean annual temperature there is an increase of 200—300% in average nitrogen content. The carbon : nitrogen ratio narrows with rising temperature.

A. G. POLLARD.

Determination of replaceable sodium in alkali and non-alkali soils. R. H. BRAY (*J. Amer. Soc. Agron.*, 1928, 20, 1160—1166).—Barber and Kolthoff's method is satisfactory. A test for replaceable sodium is proposed.

CHEMICAL ABSTRACTS.

Determination of barium in soils. G. BERTRAND and L. SILBERSTEIN (*Ann. sci. agron. franç.*, 1928, 45, 223; *Bied. Zentr.*, 1929, 58, 97).—20 g. of fine soil are ignited at dull red heat to destroy organic matter and then decomposed by heating with three times its weight of sodium potassium carbonate in two nickel crucibles. The melt is extracted with hot distilled water, filtered, and the residue containing alkaline earths, iron, and aluminium washed with sodium carbonate solution. After drying and igniting the filter paper the residue is treated with hydrochloric acid and evaporated to remove silica, again treated with hydrochloric acid, filtered, and washed. The residue, still containing some barium, is again treated with sodium potassium carbonate as before. The final acid filtrates from the two extractions are united and the barium is precipitated by the addition of 5—10 drops of 10% sulphuric acid. After keeping for two days with periodical shaking the barium sulphate is removed, treated twice with hydrochloric acid, and weighed. Results show soils to contain 0.083—1.717 g. Ba/kg. This is equivalent to 10—35% of the total sulphur of the soil.

A. G. POLLARD.

Flow of clay pastes through narrow tubes. G. W. S. BLAIR and E. M. CROWTHER (*J. Physical Chem.*, 1929, 33, 321—330).—A description is given of a modified plastometer of the Bingham and Green type (B., 1920, 495 A) suitable for use with small amounts of clay and soil pastes over a range of applied pressure equivalent to 0.05—50 cm. of mercury, and with rates of flow of 0.0002—2.0 cm.³/sec. The flow curves of suspensions prepared from a heavy Rothamsted clay

show four stages, viz., (i) no flow occurs, (ii) dv/dP is constant, (iii) dv/dP increases rapidly, and (iv) dv/dP becomes constant again. The corresponding movements are plug flow, plug flow with stream-line flow in an outer sheath, and stream-line flow. The ratio of the constants derived from the slopes of the plug and stream-line stages is found to be constant for a given clay over a wide range of concentration. Small, but probably significant, variations in this ratio are discernible in clays of different geological origin. L. S. THEOBALD.

Repair of soil filter tubes. G. J. LARSINOS and A. B. BEAUMONT (Soil Sci., 1929, 27, 243).—Breaks in Pasteur-Chamberland filter tubes are effectively repaired by sheathing the crack with rubber tubing.

A. G. POLLARD.

Drift in potential of the quinhydrone electrode [for soil measurement]. L. D. BAVER (J. Amer. Soc. Agron., 1928, 20, 1125—1140).—Unless stirred, the lower part of soil suspensions tended to be more acid than the upper part. The deviations in p_H obtained with different electrodes were not influenced by passing air free from carbon dioxide, or hydrogen, through the suspensions, or by keeping them overnight. The reading is taken preferably 1 min. after immersion of the electrodes in the soil suspension.

CHEMICAL ABSTRACTS.

Changes produced in plants on emasculation. V. G. TARANOVSKAYA (Zhur. Oputnoi Agron., 1927, 23, 127—164).—Experiments with barley, lupins, maize, buckwheat, mustard, hemp, poppy, and soya beans showed that in general the grain crops suffered decrease in dry matter. Increase in leaf area causes an increased intensity of carbon dioxide assimilation, and there is a more successful utilisation of mineral substances of the soil. The nitrogen content decreased, but the percentage of protein-nitrogen increased. Most of the calcium did not combine with the oxalic acid. There was an increase in carbohydrates, especially sugars, and a decrease in cellulose.

CHEMICAL ABSTRACTS.

Disappearance of nitrate under timothy. L. G. JONES (J. Amer. Soc. Agron., 1928, 20, 1167—1175).—Nitrate disappears fairly rapidly when added to timothy sod; except for the largest application, the disappearance was accounted for by the nitrogen taken up by the crop. At certain stages of growth the crop contained considerably more nitrogen than was contained in the added nitrate.

CHEMICAL ABSTRACTS.

Relation between concentrations of potassium in culture solutions and optimum plant growth. R. P. BARTHOLOMEW and G. JANSSEN (Soil Sci., 1929, 27, 189—203).—The intake of potassium by oats, lucerne, Hubam clover, cow peas, soya beans, Sudan grass, and cotton was examined by means of water cultures in solutions having varying concentrations of potassium salts. The potassium requirements of the different plants varied considerably, optimum growth being attained at concentrations of 0.5 p.p.m. in the case of Hubam clover and 3 p.p.m. for Sudan grass. All plants made reasonably good growth, however, with 0.5 p.p.m. of potassium in the nutrient. No relationship existed between the total potash requirements of the plants and the optimum concentrations. The

bearing of this on the varying ability of different species of plants to utilise soil potash is discussed. Plants are able to take up more potash than is required for optimum growth.

A. G. POLLARD.

Influence of soil type on the calcium and magnesium content and other physiological characters of the lucerne plant. J. F. FONDER (Soil Sci., 1929, 27, 205—232).—Analyses of lucerne plants in varying stages of growth on seven different soils are recorded. The inter-relationships between the calcium and magnesium contents of the stems and leaves and the expressed juices of these, of the moisture content, and the sp. gr. of expressed juices are recorded and discussed from the point of view of their dependence on soil type. Changes during the period of the day are included.

A. G. POLLARD.

Copper content of the leaves of beet treated with copper preparations. M. FRIGERIO (Giorn. Chim. Ind. Appl., 1929, 11, 67—69).—The amount of copper salts retained by the leaves and tops of beet after lifting varies widely, being influenced not only by the number and intensity of the applications, but also by the weather conditions. The oxalates present in the material, which are also a source of danger to the cattle, were approximately constant in all un-siloed samples examined. About one third of the total copper present is dissolved by the gastric and pancreatic juices, this ratio being independent of the nature of the copper preparation administered. Preservation of the leaves and tops in silos does not sensibly influence the solubility or toxic activity of the copper compounds present, but results in almost complete destruction of the oxalates.

T. H. POPE.

Response of oats and soya beans to manganese on some Costal Plain soils. L. G. WILLIS (N.C. Agric. Exp. Sta. Bull., 1928, No. 257, 1—13).—Chlorosis in soya beans (but not stunted growth in corn) is remedied by application to the unproductive area of manganous sulphate. Injury to oats by lime or calcium phosphate applications to a soil deficient for soya beans in manganese can be prevented by addition of manganous sulphate.

CHEMICAL ABSTRACTS.

Calcium in lemon and orange leaves. A. R. C. HAAS and F. F. HALMA (Citrograph, 1928, 13, No. 3, 10).—The calcium content of the ash of lemon and valencia leaves is about 33%; of this, 18% in the former and 48% in the latter is soluble in water. The ratio of insoluble to soluble ash is 11:4 and 1:1, respectively. Correlation between the results and the sensitiveness of the leaves to freezing is suggested.

CHEMICAL ABSTRACTS.

Hexamethylenetetramine and formaldehyde as nutrients for the haricot bean. L. MARRASSÉ (Compt. rend., 1929, 188, 1006—1007; cf. Nicolas, A., 1923, i, 77, 427; 1924, i, 128; 1925, i, 623).—When separated from their cotyledons and deprived of starch by 5 days in darkness at 24° and 4 days in distilled water the embryos are nourished by contact with 0.02% hexamethylenetetramine and 0.016% formaldehyde solutions. The formation of the starch commences where the embryo is in contact with the impregnated paper and does not follow the reverse order of its disappearance in

hydrolysis. The starch granules are smaller than normal and yield violet to yellowish colorations with iodine, possibly in consequence of the transitory formation of dextrins.

R. BRIGHTMAN.

Press- and extraction-processes in the treatment of sunflower seed [for feeding-stuff]. S. WEISER and A. ZAITSCHECK (Forts. Landw., 1928, 3, 926; Bied. Zentr., 1929, 58, 63—67).—In experiments with sheep fed with the residue from the solvent-extraction of sunflower seed, the digestibility of protein and fat appeared satisfactory, but the crude fibre and nitrogen-free extract not only showed poor digestibility itself, but markedly reduced the digestibility of hay mixed with it. The starch value of this residue was 35, the figure being higher if the proportion of husk was reduced during manufacture. With goats the press-cake from sunflower seed gave much greater live-weight increase than the solvent-extracted material. For the production of a satisfactory feeding material the removal of husks followed by pressure-extraction is necessary.

A. G. POLLARD.

Intervarietal chemical differences in the mature potato tuber. T. P. MCINTOSH (Scottish J. Agric., 1928, 11, 304—311).—A preliminary study of the value of various chemical tests for distinguishing between different varieties of mature potato tubers. In the alkali test, sections were immersed in *N*-potassium hydroxide solution and then laid in dishes, a yellow colour developing. For the oxidase test, sections are brushed with a 0.5% solution of benzdine in 50% alcohol and dried for 1 hr. at 5°, when a light brown to dark purplish-brown colour is produced. The blackening test is based on the oxidation of tyrosine to give a black coloration due to the formation of melanin. The tyrosinase reaction is based on the formation of a red colour due to the oxidation of tyrosine or of *p*-cresol by tyrosinase.

CHEMICAL ABSTRACTS.

Examination of the "effect factors" of the three principal nutrients of cultivated plants and of the determination of the nutrient content of soils according to Mitscherlich. M. NOACK (Kühn-Arch., 1928, 19, 412—553; Bied. Zentr., 1929, 58, 109—114).—Numerous trials are recorded designed to examine the alleged constancy of the "effect factors" as applied to different plants and to different forms of fertilisers. In many cases the nature of the fertiliser and the kind of crop did not markedly affect the "effect factors." There were, however, notable exceptions in which different crops indicated different contents of assimilable nitrogen and phosphorus in identical soils. The Mitscherlich "effect factor" for potash in the presence of sodium varied considerably.

A. G. POLLARD.

Phosphate nutrition of plants and soil acidity. Rock phosphate fertilisation on chernozem soils. E. V. BOBKOV and A. L. MASLOVA (Trans. Sci. Inst. Fertilisers, Moscow, 1926, No. 39, 5—38).—A forest loam (p_H 6.8) gave up phosphate soon after the addition of small quantities of hydrochloric acid; chernozem and podzolised loam (p_H 5.9—6.8) did not afford phosphate until p_H 4.0 was reached, and sandy soil only at p_H 2.5. All the soils released more calcium as the acidity increased, but practically no iron was

released by addition to 10 g. of 6 c.c. of 0.1*N*-hydrochloric acid. The soils which released the phosphorus most readily did not respond to phosphorus fertilisers. The phosphorus content of a soil, as indicated by the titration curves, may be an index of the phosphorus requirements of the soil.

CHEMICAL ABSTRACTS.

Fertiliser requirement of the onion. M. GÓRSKI and M. KOZTOWSKA (Rocz. Nauk. Rolnicz., 1928, 20, 1—14; Chem. Zentr., 1928, ii, 1891).—The onion reacts chiefly to fertilisation with potassium, that to nitrogen and phosphate being very small. Rye, oats, and potatoes react chiefly to nitrogen; only potatoes reacted to potassium. The large potassium requirement of the onion is due to its small ability to utilise soil potassium.

A. A. ELDRIDGE.

Absorption of nutrients by the onion. M. KOZTOWSKA (Rocz. Nauk. Rolnicz., 1928, 20, 15—24; Chem. Zentr., 1928, ii, 1891).—Two phases in the life of the onion are differentiated: that of absorption of mineral substances, and that of the transference of nutrient materials from the tops into the bulbs.

A. A. ELDRIDGE.

Swedish fertiliser trials in 1927. G. SUNDELIN, C. LARSON, and E. MANELL (Med. Centralanst., försöks. jordbruks, No. 340; Bied. Zentr., 1929, 58, 114—115).—Summaries of the results of numerous field trials are recorded and classified according to crop, soil type, and reaction.

A. G. POLLARD.

Citrus chlorosis as affected by irrigation and fertiliser treatments. P. S. BURGESS and G. G. POHLMAN (Ariz. Agric. Exp. Sta. Bull., 1928, No. 124, 183—232).—Following a study of the soil, fertiliser treatments have been applied, but irrigation with intermediate aeration, following soil-moisture studies, was more successful.

CHEMICAL ABSTRACTS.

Comparing various quantities of mixture of "ammophos" and ammonium sulphate plus filler to make a 10:10 formula (10% N and 10% P_2O_5). C. L. LOCSIN, F. TABHAN, and E. PUNZALAN (Rep. Comm. Cane Var., Dis., Fert., 6th Conv. Philippine Sugar Assoc., 1928, 83—86).—Yields are correlated with fertiliser values.

CHEMICAL ABSTRACTS.

Testing various quantities of "ammophos" 20:20 (or 16.5% N and 20% P_2O_5). C. L. LOCSIN (Rep. Comm. Cane Var., Dis., Fert., 6th Conv. Philippine Sugar Assoc., 1928, 86—89).—Yields are correlated with fertiliser values. Sufficiency of phosphate is a factor in the development of maturity of the sugar cane.

CHEMICAL ABSTRACTS.

Comparative pot experiments with superphosphate, "reform phosphate," "plutophos," "moor-phos," and two Polish raw phosphates. C. DREYSPRING and C. KRÜGEL (Superphosphate, 1928, 1, 62—72, 77—90).—Only superphosphate afforded an increase in grain and straw of oats grown on lowland moor soil. With barley on sandy loam the relative yields were: 100, 15, 10, 25, (a) 15, (b) 35. There was a close relation between the yield and the quantity of phosphate assimilated by the plants from the different phosphates.

CHEMICAL ABSTRACTS.

Effect on soil reaction of nitrogenous fertilisers under the anaërobic conditions of rice production.

R. P. BARTHOLOMEW (J. Amer. Soc. Agron., 1928, 20, 1305—1313).—Nitrogenous fertilisers are differentiated as follows: (a) acid-producing: ammonium sulphate, Leuna saltpetre, "ammophos," and urea, (b) alkali-producing: sodium and calcium nitrates and calcium cyanamide, (c) those producing no change in hydrogen-ion concentration: cottonseed meal and blood meal. Increase or decrease in hydrogen-ion concentration is injurious, group (b) being the more harmful.

CHEMICAL ABSTRACTS.

Microbiology of soils in relation to liming and phosphate applications. E. E. UPENSKI (Trans. Sci. Inst. Fertilisers, Moscow, 1926, No. 34, 65—71).—Determinations of nitrate, nitrite, and other products of microbial metabolism are insufficient for the evaluation of the rôle of microbes. In a study of liming and phosphorus application, *Azotobacter*, denitrifiers, nitrifiers, and *Bacillus mycoides* are specially important. Protozoa are not important under field conditions. Liming beyond p_H 7.6 increases the activities of denitrifiers.

CHEMICAL ABSTRACTS.

Biological and chemical examination of stall manures. IV (Pt. 2). G. RUSCHMANN (Zentr. Bakt., 1928, 75, 405—426; Bied. Zentr., 1929, 58, 56—62).—Horse manure alone was used for the hot fermentation (Krantz) process. Less satisfactory results were obtained than with ordinary mixed yard manures. The compression of the stack did not check the initial fermentation sufficiently, and there was a relatively heavy loss of dry matter. Physically, too, the product was inferior. The active organisms in the fermenting stack were fewer than in the case of mixed manure. Details of the micro-flora are recorded. No definite relationships exist between the ammonifying power of hot-fermented manure and the nature of the micro-flora or the method of fermentation. The presence of nitrates and nitrites in the finished stack is accounted for by the number of the corresponding organisms contained therein. Yard and stall manure is normally free from these. In some cases the absence of nitrate in manure results from the activity of denitrifying organisms. In well-prepared hot-fermented stacks very few denitrifying organisms are found. Organisms surviving the hot-fermentation process have an enhanced vitality. Butyric fermentation may occur in manure stacks and lead to soil injury when applied. This is minimised by careful attention to the temperature of the fermenting stack, the bacteria concerned being largely reduced in number at higher temperatures. "Cold" manure stacks characterised by poor rotting-down of the litter have relatively high numbers of "butyric" bacteria. Satisfactory humification in the manure stack appears to correspond with the presence of small numbers of starch-splitting organisms. In the hot fermentation of horse manure the humification occurs relatively late in the process, and in this connexion the exclusion of oxygen, maintenance of sufficiently high temperature, and moisture content are important. (Cf. B., 1927, 395; 1928, 870.) A. G. POLLARD.

Hot fermentation of manure and weeds. C. FRUWIRTH (Forts. Landw., 1928, 3, 832; Bied. Zentr., 1929,

58, 83—84).—The vitality of seeds surviving the hot-fermentation process for manure was examined. Daily additions of weeds and manure were made to the stack in a concrete pit and seed samples inserted in fine-meshed packets. A small number of seeds germinated after the "cold" fermentation of the ordinary yard manure, but in the hot-fermentation process none survived. Similar results were obtained with undigested seed entering the stack with the dung. A. G. POLLARD.

Value of peat as a material for the preparation of composts and peat manure. D. N. PRIANISHNIKOV (Trans. Sci. Inst. Fertilisers, Moscow, 1927, No. 34, 72—75).—Peat, employed with potassium and phosphates, supplied nitrogen to plants. The phosphorus of raw phosphates, when the latter are composted with peat, becomes water-soluble. Alkaline composting of peat rendered 12% of nitrogen soluble in 72 days.

CHEMICAL ABSTRACTS.

Use of dextrin in the isolation or identification of *Azotobacter chroococcum*. C. E. SKINNER (Soil Sci., 1929, 27, 245—246).—A dextrin-nitrate-agar medium renders the dissolution of *A. chroococcum* more easy than Ashby's or similar media poor in nitrogen. The appearance of apparently non-chromogenic strains is also avoided. A. G. POLLARD.

Carbon dioxide, a chemical accelerating the penetration of respiratory insecticides into the tracheal system by keeping open the tracheal valves. E. H. HAZELHOFF (J. Econ. Entomol., 1928, 21, 790).—A small quantity of carbon dioxide will probably accelerate the penetration of hydrogen cyanide, carbon disulphide, etc. into the tracheal system of insects.

CHEMICAL ABSTRACTS.

Magnesium and calcium chlorates as substitutes for sodium chlorate for killing field bindweed. W. L. LATSHAW and J. W. ZAHNLEY (J. Amer. Soc. Agron., 1928, 20, 1328; cf. B., 1928, 102).—The above substances are equally effective; the magnesium and calcium salts possess certain advantages over the sodium salt.

CHEMICAL ABSTRACTS.

Experiments with lime and phosphates on the Kriukovo and Volokolamsk experimental plots in 1925. S. V. SHCHERBA (Trans. Sci. Inst. Fertilisers, Moscow, 1927, No. 41, 39—84).

Determination of the nutrition requirement of the vine by chemical examination of the leaves. A. JACOB (Z. angew. Chem., 1929, 42, 257—262).

See also A., April, 420, Calcareous soils of Bavaria (NIKLAS and others). Weathering of sandstone, limestone, and basalt in red-earth areas (REIFENBERG). 431, Synthesis of cyanamide (KADLEC-FLECK). 478, Sodium nitrate and development of chlorosis in rice (METZGER and JANSSEN).

Fluorides and fluosilicates as insecticides. CARTER and ROARK.—See VII. Germicidal soaps. HAMPIL.—See XII. Tobacco. HALEY and others.—See XX.

PATENTS.

Production of granular fertilisers. W. J. WORBOYS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 307,575, 9.1.28).—Gelatinous phosphates, e.g., colloidal iron and/

or aluminium phosphates, which may be added to an aqueous paste of the fertiliser or formed during the manufacture of, *e.g.*, ammonium phosphates from rock phosphate containing iron and aluminium compounds, are used as binding agents in the production of fertilisers. Solidification in granular form is effected by compressing the pasty mixture, or by extrusion, cutting off in small lengths, and drying. L. A. COLES.

Production of non-hygroscopic phosphates [fertiliser]. H. BLUMENBERG, JUN., ASSR. to STOCKHOLDERS SYND. (U.S.P. 1,706,101, 19.3.29. Appl., 30.4.25).—Crude, ground phosphate rock is mixed with sufficient phosphoric acid to give acid calcium phosphate and up to 20% excess acid, calculated on the acid phosphate. The product is fused at 600°, cooled, and ground.

F. G. CLARKE.

Artificial fertiliser. H. HEIMANN, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,698,793, 15.1.29. Appl., 27.8.21. Ger., 29.1.18).—Non-deliquescent fertiliser is obtained by mixing hot ammonium nitrate solution, *e.g.*, 85–88%, with potassium chloride or sulphate, ammonium sulphate, or other fertilising salt, the product being recovered in the solid state by evaporation of the water through the heat present.

R. BRIGHTMAN.

Manufacture of fertiliser material. G. P. WALTON and R. F. GARDINER, ASSRS. to U.S.A. (U.S.P. 1,703,504, 26.2.29. Appl., 19.7.27).—Perishable organic material is mixed with sufficient of an acid salt to preserve the mixture, which is then air-dried. A. B. MANNING.

Manufacture of [high-grade] mixed fertiliser. W. GAUS, R. GRIESSBACH, and O. SCHLIEPHAKE, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,699,234, 15.1.29. Appl., 17.1.28. Ger., 21.1.27).—See B.P. 295,477; B., 1928, 765.

[Manufacture of] mixed fertiliser. E. SCHWARTZ, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,699,254, 15.1.29. Appl., 15.1.26. Ger., 19.1.25).—See B.P. 255,726; B., 1926, 843.

XVII.—SUGARS; STARCHES; GUMS.

Viscous fermentation of frozen [sugar] beet. H. COLIN and M. SIMONET (Compt. rend., 1929, 188, 943–945).—A Gram-negative *Coccus* has been isolated from frozen beet, which is probably responsible for the typical fermentation. From the juice of such beet, alcohol precipitates a white carbohydrate, darkening at 200° and softening at 250°, which is slowly hydrolysed by hot acids to afford dextrose exclusively.

G. A. C. GOUGH.

Colouring matters of beet molasses. M. GARINO, A. REGÈ, and F. RUBINO (Giorn. Chim. Ind. Appl., 1929, 11, 61–63; cf. B., 1929, 372).—Caramelan, caramelen, glucic and apoglucic acids all impart to their solutions brown colours, which are difficult to distinguish by the naked eye, but may be differentiated with the help of Wood's light (3650 Å.) if the materials are prepared from pure sugars. With molasses, however, this is difficult owing to the marked luminescence characteristic of the molasses itself. The colouring

power varies from a minimum of about 800 units (Stammer) for caramelan to a maximum of 18,000 for caramelen. The colouring substances formed by the action of lime on invert sugar are acid, and may form salts with the lime or with ammonia derived from the aminic groups of the nitrogenous constituents of the beet and so give rise to substances of high colouring power. T. H. POPE.

Decolorising carbons and their action on the colouring matters of beet molasses. M. GARINO and A. REGÈ (Giorn. Chim. Ind. Appl., 1929, 11, 64–67).—Results are given of the action of a number of active carbons on the colouring matters of beet molasses, both singly and mixed, in presence and in absence of sugar. The coefficient of adsorbability diminishes with increase in the sugar content of the solution, and the adsorption of mixtures of the colouring matters cannot be calculated beforehand. The coefficients of adsorbability of these colouring matters depend principally on their surface tensions, but specific affinities for carbons activated in different ways are also observed. Of the carbons examined, carboraffin adsorbs the most caramelan and the most calcium glucate, whilst appula adsorbs most caramelen and polycarbon most calcium apoglucate. Indications are obtained that not only the extent, but also the quality, of the surface of the active carbon exerts an influence on the adsorption. Replacement of the mineral framework of animal charcoal by water by treatment with hydrochloric acid results in increase in the surface of contact between the carbon and the solution, so that the carbon becomes more active towards the colouring matters investigated, the activity increasing with the proportion of water left in the material. Much of the decolorising power is lost if the carbon is dried to such an extent that air is able to penetrate the pores. T. H. POPE.

Inversion of sucrose in beet-house syrups. R. J. BROWN and H. W. DAHLBERG (Ind. Eng. Chem., 1929, 21, 282–285).—A method for the determination of small quantities of invert sugar in beet syrups is described, a process based on the work of Quisemburg and Thomas (B., 1921, 399 A) being used. Although the invert sugar content of a high-purity syrup may be taken as a measure of the inversion of the sucrose, that of low-purity products is of no value in calculating the loss of sucrose owing to destruction of invert sugar during heating. The rate of destruction of the invert sugar depends on the concentration of the impurities, variations in p_H value between 6 and 9 having no appreciable effect. The rate of loss of sucrose by inversion increases with fall in p_H value and with rise of temperature, decreases with decreasing purity, and is constant with beet syrups from different sources under the same conditions or with solutions of varying concentration. The observed rate of inversion of sucrose in these syrups is higher than that calculated from the inversion constant of sucrose with hydrochloric acid. This discrepancy may be due either to the influence of other ions, or most likely to the difference between the hydrogen-ion concentration of the cold solutions in which the p_H is measured and of syrups heated to the temperature at which inversion is allowed to proceed. F. R. ENNOS.

Unknown losses of sugar during saturation. L. O. SNAJDMAN (Nauch. Zapiski, 1928, 5, 322—328).—If calcium carbonate is precipitated in an aqueous alkaline sugar solution, adsorbed sugar and alkali are returned to the solution as the amorphous precipitate becomes crystalline. CHEMICAL ABSTRACTS.

Applications of the nitrile method. III. The cyanide and formaldehyde values of sugar and their analytical value. F. LIPPICH (Z. anal. Chem., 1929, 76, 401—408; cf. B., 1929, 414).—The common sugars have characteristic cyanide and formaldehyde values; using 5 g. of the sugar in 100 c.c. of solution and a contact time of 10 min., the following figures were obtained for the cyanide values: dextrose 9.14, laevulose 16.77, invert sugar 13.41, lactose 3.54, and maltose 5.10; the corresponding formaldehyde values were 2.91, 6.71, 5.15, 1.94, and 2.22, respectively. For different quantities of sugar the values are not linearly proportional, but lie on a smooth curve concave to the axis of concentration. By determining the cyanide value of a solution containing any one of the above sugars its concentration can be read sufficiently accurately from a graph previously constructed from the values obtained with solutions of known concentration. If a definite quantity of a syrup (e.g., 5 g.) is dissolved in 100 c.c. of water and the cyanide value determined as previously described for milk (*loc. cit.*), the percentage of sugar present may be ascertained to within $\pm 1\%$ by reference to the graph. A. R. POWELL.

Water content of potato starch. SPROCKHOFF (Z. Spiritusind., 1929, 52, 110).—When potato starch with a moisture content of 20% is exposed to ordinary air, it does not appreciably gain or lose moisture. This is due to the constitution of the starch grains, which are built up of infinitely small particles loosely packed together and leaving spaces in which moisture is retained by adsorption and capillarity. The vapour pressure of this water is lower than that of water in an open vessel, and varies according to the water content of the starch. Starch with 20% of moisture has a vapour pressure of approx. 75% of that of normal water at the same temperature. The actual humidity of the air is a fraction of its maximum capacity, and the average yearly value at ordinary temperature is slightly above 75%. Accordingly, the air and starch with a moisture content of 20% are in equilibrium, and the transference of moisture from one to the other is negligible. C. RANKEN.

Destruction of the oxidising enzymes of gum arabic. L. ROSENTHALER (Pharm. Ztg., 1929, 74, 317).—The gum solution is treated with acid or alkali, and neutralised after 24 hrs. S. I. LEVY.

See also A., April, 427, **Degradation of l-arabinose** (DEULOFEU and SELVA). 428, **Acetylation of carbohydrates** (TSUZUKI). **Influence of hydrogen sulphite solutions on sugars at higher temperatures** (HÄGG-LUND). 459, **Volumetric determination of dextrose** (AMICK). 462, **Determination of reducing sugars** (HAWKINS and VAN SLYKE). 476, **Determination of starch in vegetable tissue** (BISH).

Carbon dioxide under pressure and its application. KRASE and GOODMAN.—See VII. **Honey.** FABIAN and QUINET, also FABIAN.—See XIX.

PATENTS.

Purification of liquids [sugar solutions]. A. B. RAY, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,699,449, 15.1.29. Appl., 3.5.22).—Solutions containing dissolved salts and organic colouring matters, e.g., sugar solutions, are treated, separately or together, with activated carbon and a precipitated tricalcium or other alkaline-earth phosphate. R. BRIGHTMAN.

Cooling devices for mashing vessels for crystallisable material, more particularly for the sugar industry. A. ROLZ (B.P. 291,035, 24.5.28. Ger., 24.5.27).

Waste waters from sugar factories (B.P. 284,267 and 307,587).—See XXIII.

XVIII.—FERMENTATION INDUSTRIES.

Analysis of the bitter substances of hops. W. WINDISCH, P. KOLBACH, and M. WINTER (Woch. Brau., 1929, 46, 101—106, 111—117, 124—131).—Lupulone is prepared from commercial lupulin by a modification of the methods of Walker (J. Inst. Brew., 1924, 30, 570) and Espe (Diss., Berlin, 1925). Since the yield was only 15% from a soft resin fraction the iodine value of which indicated nearly 45% of lupulone, it is concluded that owing to its liability to change, published figures for the lupulone content of hops are valueless. An investigation of the lead precipitation method for determining humulone shows that the lead compound is more readily redissolved by excess of lead acetate when much hard resin is present. To avoid such excess small portions of the resin solution should be treated with varying quantities of the reagent and tested for lead after filtration. The precipitate, conveniently collected and weighed in sintered glass crucibles, consists of unaltered lead humulate, as shown by the proportion of lead and the iodine value of the recovered humulone. The latter was found to be 147.1—149.7 against 151.4 for pure humulone, and both values tended to be lower in presence of much (e.g., 20%) hard resin. Old or badly kept hops thus require special treatment, and the following methods are worked out. For fresh samples, containing less than 12% of the total resin in the form of hard resin, direct precipitation of the methyl-alcoholic solution of the total resin is suitable. In the case of older samples the soft resin should first be extracted by light petroleum or hexane, and, after evaporation, the residue dissolved in methyl alcohol and the lead humulate precipitated. Very altered samples fail to be precipitated except with much excess of lead acetate and give erroneous results. This may be corrected by washing the light petroleum extract with a phosphate buffer solution of p_H 6.4, the interfering substance, which is shown to be derived from humulone, being thus removed. The use of hexane in place of light petroleum gives satisfactory results and is to be preferred. The iodine value of the total resins is of little value, but that of the hexane-soluble portion, from which that of the soft resin minus humulone may safely be calculated by subtracting the value due to humulone, is a good guide to the changes which the resins have suffered. The original con-

stituents appear to be only humulone and lupulone, and their alteration products to have low iodine values, if any. It will, however, only occasionally be required to supplement the results of the humulone and hard resin determinations, since these suffice in the usual case of new hops. Electrometric titrations of solutions of humulone, lupulone, and resins show that their proportions cannot be determined by titration in stages over various p_H ranges. Since they are not in molecular solution in water, their buffering ranges vary with the concentration. Humulone and lupulone are equally strong acids in methyl alcohol, but humulone is markedly stronger in water. F. E. DAY.

Analysis of hops. W. WÖLLMER (Woch. Brau., 1929, 46, 121—124).—Details of the modifications of the usual methods used for convenience and speed at the Sternburg Brewery, Leipzig, are given. Cold-extraction for 3 hrs. with shaking gives very slightly lower results for total resin, but, if anything, slightly higher results for humulone and soft resin as compared with Soxhlet extraction. Treatment of 15 c.c. of the methyl-alcoholic solution of the resins with 50 c.c. of light petroleum and 25—50 c.c. of water gives results for soft resin agreeing with those obtained by extracting the dried total resin with light petroleum. Where comparative analyses are to be made, the substitution of hexane for light petroleum is suggested. F. E. DAY.

Frozen yeast. STAIGER and GLAUBITZ (Z. Spiritusind., 1929, 52, 116).—Yeast which was frozen at -10° to -25° for 24, 48, 72, and 96 hrs. and subsequently thawed by being stored for 24 hrs. either at room temperature ($17-20^\circ$) or at 5° showed little change in its properties. The stability and nitrogen content were unaltered, but there was a slight increase in the time taken for the fermentation of dough by the yeast which had been thawed at 5° . The water content was lower than that of the normal yeast, more especially where the yeast after being frozen for the longer periods was thawed at room temperature, in which case the difference approximated to 4%. Although in biological appearance the frozen and normal yeasts were identical, the frozen yeast yielded the very slight increase of less than 1% in the number of dead cells. C. RANKEN.

Metals in connexion with wort and beer. C. G. MATTHEWS (J. Inst. Brew., 1929, 35, 181—188).—The use of an iron yeast press may introduce iron into the barm ale and give rise to an astringency. This tends to produce later an iron haze and an undesirable colour when the barm ale is added to the brew in the fermentation vessel. Zinc or galvanised containers dissolve in appreciable amount and produce an unpleasant metallic effect on the palate. Copper can be safely used for mashing vessels, but may be corroded by fermenting worts with a consequent reduction in the amount of yeast reproduction. The corrosion is more intense when the metal is scoured than when it is simply mopped over and allowed to form a natural film of oxide or sulphide. Copper, especially when of high purity, may become pitted at points where particles of foreign substances are situated, and which determine small galvanic cells.

Copper alloys, such as gunmetal, are less subject to this action. Aluminium of 99% purity is superior and is hardly attacked by fermenting wort, but is corroded by prolonged contact with water. Aluminium vessels should, therefore, be kept dry if they have to remain empty for any length of time. Tin, which is seldom pure, is affected by drastic methods of cleansing and is likely to produce hazes. Stainless steel, monel metal, and, to a smaller extent, pure nickel appear suitable for all types of brewing vessels, but are relatively dear compared with copper and aluminium. Although there is a tendency for a protective coating to form, pure lead piping is attacked by beer. Lead alloys containing 15% Sn are much more resistant. C. RANKEN.

The nitrogen question [in brewing]. J. H. OLIVER (J. Inst. Brew., 1929, 35, 191—200).—The amount of soluble nitrogenous compounds in a wort made by mashing malt at approx. 67° increases as the concentration of the mash becomes greater and as the diastatic activity of the malt becomes higher. The nitrogen content of the wort is also augmented by the acidification of the mash, but the results under conditions of thick mashing, such as brewery mashes, are not so marked as with the 10% laboratory mash (thin mashing), since the increased concentration of the salts and proteins of the thicker mashes exerts a greater buffering effect. The maltose content of the wort tends to be similarly influenced by the concentration of the malt mashes. C. RANKEN.

Substance responsible for the bitter taste formed in the amertume of wines. E. VOISENET (Compt. rend., 1929, 188, 941—943).—A substance, b.p. 198° , with an intensely bitter taste has been isolated from Burgundy wine (3 c.c. from 4 litres of wine); it is possibly a derivative of acraldehyde. G. A. C. GOUGH.

Limiting factors in lactic fermentation. L. O. ROGERS and E. O. WHITTIER (J. Bact., 1928, 16, 211—229).—Lactic fermentation continues for a time after the bacterial cells cease to multiply. The optimal p_H for bacterial population is 5.8—6.0; mechanical stirring with air or, preferably, nitrogen further increases the population. The concentration of undissociated lactic acid is the principal factor in limiting growth and metabolism. A substance which is diffusible through a collodion membrane limits the growth of *Streptococcus lacticus*. CHEMICAL ABSTRACTS.

See also A., April, 405, Kinetics of hydrolysis of trehalose, α -methylglucoside, and tetramethyl- α -methylglucoside (MOELWYN-HUGHES). 470, Dextrose-oxidase (MÜLLER). 471, Determination of catalase in barley-malt (CHARMANDARIAN). Sporulation of yeast (STANTIAL). 472, Giant yeast cells (HOLT). Extraction and purification of co-enzyme from yeast (MYRBÄCK). Formation of citric and oxalic acids by *Aspergillus niger* (KOSTYTSHEV and TSCHESNOKOV). 473, *Bacterium gluconicum* in tea fungus (HERMANN). Fixation of atmospheric nitrogen by *Azotobacter* (MEYERHOF and BURK).

Fusel oil reaction of alcohol. DIETRICH and JEGLINSKI.—See III. Stall manures. RUSCHMANN. Hot fermentation of manure and weeds. FRU-

WIRTH.—See XVI. **Viscous fermentation of frozen beet.** COLIN and SIMONET.—See XVII.

PATENTS.

Yeast and its manufacture. G. O. W. HEIJKENS-KJÖLD, ASSR. to AKTIEBOLAGET BÄSTA (U.S.P. 1,703,272, 26.2.29. Appl., 13.6.27. Swed., 14.6.26).—Hot sulphite liquor obtained from digestion of wood is treated with limestone and lime to neutralise the acid and render the liquor slightly alkaline, the whole being continuously aerated. After removal of the precipitates, the liquor is added gradually to a yeast-propagating wort, aeration being continued to remove alcohol formed, and the mixture maintained slightly alkaline throughout.

F. R. ENNS.

Food from lower vegetables (U.S.P. 1,702,303).—See XIX. **Waste waters from distilleries etc.** (B.P. 284,267 and 307,587).—See XXIII.

XIX.—FOODS.

Comparison of some properties of normal and frosted wheat. A. H. JOHNSON and W. O. WHITCOMB (Mont. Agric. Exp. Sta. Bull., 1927, No. 204, 1—66).—No marked difference was observed in the crude protein content; the crude gluten is decreased only if the moisture content on freezing was greater than 44—46%. The amino-nitrogen content of frosted wheat is high. During auto-digestion the protein of frosted-wheat flour is broken down, and reducing sugars are formed, more rapidly than with normal flour. The reducing sugar content of frosted wheat is above the normal value, particularly if the wheat was immature when frosted.

CHEMICAL ABSTRACTS.

Relations between acidity and freezing point of milk. A. J. PARKER and L. S. SPACKMAN (Analyst, 1929, 54, 217—221).—The f.p. and acidity of a number of genuine and watered milks were determined on the fresh milks and after varying lapses of time. The normal acidity of fresh milk is suggested as 0.14% of lactic acid, and a temperature correction factor of 0.003° for each 0.01% excess acidity is shown to hold for acidities of 0.17—0.6%. The cryoscopic method only gives accurate results for added water in watered milks when applied to the fresh milks. Pasteurisation raises the f.p. 0.01°.

D. G. HEWER.

Cryoscopic method for detection of added water in milk. R. L. ANDREW (Analyst, 1929, 54, 210—216).—Analysis of 270 genuine milks in New Zealand showed a wide range in fat (2.35—5.9%) and of solids-not-fat (8.06—9.43%), but the maximum variation of f.p. was only from -0.545° to -0.565° , and in the case of 264 of the samples was between -0.550° and -0.560° . Beckmann's f.p. apparatus was used. This is filled with crushed ice and salt, and a strong freezing mixture is placed in another vessel. The zero point of the thermometer is found in water, and then a similar volume of milk is cooled in the strong freezing mixture, when supercooling usually occurs. Immediately the mercury begins to rise the tube is removed, placed in the apparatus, and stirring continued until the mercury reaches the highest point, which is taken as the f.p. If there is more than 1° of supercooling, the reading is

0.01° too low for every extra degree of supercooling. If no supercooling occurs the tube is removed at the stationary point of the mercury, and the lowest point of the subsequently falling mercury taken as the f.p. If the same conditions are adhered to no corrections need be applied. By this method added water which is not sufficient to bring the solids-not-fat below the legal limit may be readily detected. Naturally poor and adulterated milk may be distinguished. If the f.p. rises to -0.530° watering may be suspected, and if to -0.520° it is certain with approximately 5% of added water. The f.p. of normal milk is taken as not above -0.550° .

D. G. HEWER.

Direct determination of chloride in milk by conductivity titration. H. SCHORSTEIN, G. JANDER, and O. PFUNDT (Z. angew. Chem., 1929, 42, 335—336).—To 10 c.c. of milk 50 c.c. of water and 1 c.c. of 0.1N-nitric acid are added and the mixture is titrated conductometrically with silver nitrate. The result is in agreement with that obtained by ordinary titration of excess silver nitrate with thiocyanate, but increase of nitric acid gives lower results. If the nitric acid is replaced by acetic acid the variation is more complicated and the result unsatisfactory, but when the above quantity of nitric acid is used the method is reliable.

C. IRWIN.

Guaiaecum reaction of milk. G. SCHROETER (Milch. Forsch., 1928, 6, 533—537; Bied. Zentr., 1929, 58, 72—74).—The guaiaecum test is modified to ensure more reliable results and greater convenience in operation. The guaiaecum reagent and the "activated" acetone should be stored separately. In a suitably marked tube is placed the 10 c.c. sample of milk, followed by 0.5 c.c. of guaiaecum solution and 1 c.c. of the activated acetone, and the mixture is well shaken. A positive reaction is obtained with fresh or sour milk, but not with heated milk. The reaction with a mixture of fresh and heated milk is much weaker than with fresh milk and water. Cooked milk may contain an anti-catalyst. The rapidity of appearance and depth of colour produced is a measure of the peroxidase present. The species and method of feeding of the cow affect the intensity of the colour produced. Milk from diseased cows gives sometimes a positive and sometimes a negative reaction. Standard tints for comparison are made from artificial milk (tetralin-casein) coloured with Prussian-blue or acid-violet.

A. G. POLLARD.

Use of the nitrile method. II. Action of hydrocyanic acid on milk and its analytical value. F. LIPPICH (Z. anal. Chem., 1929, 76, 321—335; cf. A., 1929, 460).—The number of c.c. of 0.1N-potassium cyanide which react with 100 c.c. of milk is approximately a constant under standard conditions; for normal milk this "cyanide value" is 9.3—9.6, according to the sp. gr. of the milk. The value is increased by increasing the excess of cyanide added in the test, the time of contact, and the temperature, and it varies slightly with the casein : lactose ratio. The determination of the cyanide value of a milk is made as follows: 100 c.c. of milk are centrifuged to remove fat, raised to 19° in a water-bath, and poured into 20 c.c. of 0.25N-potassium cyanide solution at 19°; after 10 min. 40 g. of tartaric acid in 80 c.c. of water are added, the

mixture is heated to boiling, and a current of air passed through for 2–2.5 hrs. to expel free hydrocyanic acid, which is collected in 50 c.c. of 10% potassium hydroxide solution and determined as previously described (*loc. cit.*). This determination in combination with the sp. gr. is sufficiently accurate to detect 2% or more of added water, the decrease in cyanide value being roughly proportional to the dilution of the milk. By treating the milk with a known volume of standard formaldehyde solution before making the cyanide test and then conducting this test as above, a "formaldehyde value" may be obtained by subtracting the true cyanide value from the cyanide value for the formaldehyde-milk mixture and then subtracting the result from the c.c. of 0.1*N*-formaldehyde solution added. For normal milk the formaldehyde value is about 4.2.

A. R. POWELL.

Washing powders for dairy use. A. W. PHILLIPS, M. J. MACK, and J. H. FRANSEN (Mass. Agric. Exp. Sta. Tech. Bull., 1928, No. 13, 179–187).—Washing powder preferably contains anhydrous sodium carbonate (60%) and sodium phosphate dodecahydrate (40%), the total alkali content being 58%. The properties, for dairy use, of sodium carbonate, phosphate, and hydroxide (the commonest ingredients of washing powders) are discussed.

CHEMICAL ABSTRACTS.

The Schmid-Bondzynski-Ratzlaff method for determining fat in cheese. B. C. VAN BALEN WALTER (Chem. Weekblad, 1929, 26, 164).—Details of manipulation and results of control tests are given. The method is declared to be more accurate than that of Smetham.

S. I. LEVY.

Cause of the fermentation of honey. F. W. FABIAN and R. I. QUINET (Mich. Agric. Exp. Sta. Tech. Bull., 1928, No. 92, 1–40).—The critical moisture point for fermentation is 21%. The moisture content of extracted honey increased by absorption at 20° from 5 to 12%. Comb honey stored for 7 yrs. over calcium chloride lost 7.5%; when stored in a moist place it gained 3.5–5.7% of water. Honey may be pasteurised at 62.5° in 30 min.

CHEMICAL ABSTRACTS.

Cause of fermented honey and its control. F. W. FABIAN (Fruit Prod. J., 1928, 8, No. 2, 18–19, 22).—In a moist atmosphere, honey may absorb water, becoming sufficiently dilute to permit the growth of yeasts. Unripe or improperly cured honey contains an excessive amount of water and tends to become fermented. Comb honey loses and absorbs water very slowly.

CHEMICAL ABSTRACTS.

Extraction of apple juices in the manufacture of jelly. C. R. FELLERS (Mass. Agric. Exp. Sta. Res. Bull., 1928, No. 15, 218–251).—Optimal conditions are described, and the characters of the product are correlated with the sugar content and percentage of pectin.

CHEMICAL ABSTRACTS.

Influence of sugar inversion on gel formation. K. FIEDLER (Konserven-Ind., 1928, 15, 405–407, 427–428; Chem. Zentr., 1928, ii, 1831).—The conversion of sucrose into invert sugar reduces and may prevent gelation in jellies, marmalades, etc. Pectin remains as

an active gelatinising agent, without, however, exercising its effect on account of the inversion.

A. A. ELDRIDGE.

Detection, determination, and oxidation of sulphur dioxide [in foods]. D. HENVILLE (Analyst, 1929, 54, 228–231. Cf. B., 1927, 502; 1928, 346).—The apparatus used consists of two bulbs, the lower one preventing liquid being drawn back into the flask and acting as an absorption bulb, and the upper one containing the bulk of the liquid by which the sulphur dioxide is absorbed. Sulphuric acid (10 c.c.) is placed in the lower bulb, then two drops of bromphenol-blue and 0.1*N*-sodium hydroxide until the liquid is just blue; the vent is then closed. The apparatus is fitted into the top of a reflux condenser, and the flask at the bottom contains 150 c.c. of air-free water and is connected to a carbon dioxide cylinder. When air is expelled the sample is introduced, followed by 50 c.c. of 16 vol.-% solution of hydrochloric acid, and the flask is heated. The indicator in the lower bulb will change to yellow before the b.p. is reached if much sulphur dioxide is present, and traces of sulphur dioxide show a colour change within 5 min. from the commencement of boiling. The sulphur dioxide is entirely absorbed in the two bulbs and the test may be made quantitative. An extra bulb may be used to ensure retention of sulphur dioxide.

D. G. HEWER.

See also A., April, 413, **Determination of iodine in vegetables** (McCLENDON and REMINGTON).

Sunflower seed as feeding-stuff. WEISER and ZAITSCHEK.—See XVI.

PATENTS.

Yeast-saving composition in bread-making. C. B. HILL and M. H. GIVENS. Assrs. to NORTHWESTERN YEAST Co. (U.S.P. 1,702,259, 19.2.29. Appl., 10.1.27).—A mixture of 2 pts. of ammonium sulphate, 1 pt. of calcium sulphate, and 1 pt. of tricalcium phosphate is added either to the dough batch or to the yeast prior to making the dough.

F. R. ENNOS.

Cream separator. A. J. SQUIRE and J. J. LINDVART (B.P. 307,635, 20.3.28).—An air fan of disc-plate shape is arranged within the cream chamber and connected with the separator bowl spindle so as to aerate and deodorise the cream.

W. J. BOYD.

Treatment of tea in course of manufacture. J. P. CHALIKA (B.P. 307,591, 6.2.28).—Drying and/or conditioning prior to withering is effected by placing the leaf in a chamber maintained under reduced pressure and at 71–99° so as to cause the moisture to evaporate rapidly with minimum loss of aroma. A suitable water-jacketed chamber is described which also serves in the fermentation of the rolled leaf etc.

W. J. BOYD.

Manufacture of food etc. from lower vegetables. A. KAHN, Assr. to Soc. FRANÇ. DES PROD. ALIMENTAIRES AZOTÉS (U.S.P. 1,702,303, 19.2.29. Appl., 16.12.25).—Distillery yeast free from alcohol is kept at 40–55° for a few hours, and after addition of 5–20% of sodium chloride the whole is autolysed for 1–5 days above 40°, but below the minimum temperature at which the enzymes are destroyed. The autolysate is separated from

the cellulose residue, the former on concentration *in vacuo* affording a palatable food product whilst the latter is an efficient emulsifying agent. F. R. ENNOS.

Preventing the discoloration of canned foods. G. S. BOHART, Assr. to NAT. CANNERS ASSOC. (U.S.P. 1,699,274, 15.1.29. Appl., 13.3.26).—Zinc oxide or phosphate or other insoluble oxides or salts of metals which do not yield coloured sulphides are incorporated in a baking lacquer, which is applied to the inner surfaces of the container. R. BRIGHTMAN.

Cereal food preparation. L'AUGMENTINE S.A. (B.P. 289,060, 19.4.28. Ger., 21.4.27.)

Apparatus for ultra-violet ray treatment of flour. S. BONSOR (B.P. 308,390, 7.1.28).

Alimentary [chocolate] substance. C. MAGAT (B.P. 308,552, 28.9.28.)

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Acetylsalicylic acid. D. B. DOTT (Pharm. J., 1929, 122, 355).—Addition of the theoretical quantity of sodium bicarbonate to an aqueous solution of aspirin caused decomposition, after 4 hrs., equivalent to the formation of 4.27% of salicylic acid, and after 24 hrs. to 14.14% of salicylic acid. E. H. SHARPLES.

Ammonium salt of acetylsalicylic acid. M. N. DVORNIKOFF (J. Amer. Pharm. Assoc., 1929, 18, 213).—Following Woldman's procedure (B., 1929, 263) only aspirin contaminated with ammonium salts was obtained. Aspirin dissolves in ammonia solution, and is readily hydrolysed by the excess of ammonia giving ammonium acetate and salicylate. The consumption of ammonia approaches the ratio of 2 mols. of ammonia to 1 mol. of aspirin. When all the ammonia is neutralised more aspirin dissolves in the warm liquid and separates unchanged on cooling. E. H. SHARPLES.

Action of peroxidase on glycerophosphates. J. RAE (Pharm. J., 1929, 122, 354—355).—A 5% solution of sodium glycerophosphate containing 0.05% of phosphate after contact with a 10% potato extract for 2 months showed a phosphate content of 2.4%. With 10% mucilage of acacia the phosphate content increased to 5.0% in 14 days, and with a sterilised mucilage of acacia the change was retarded, but a content of 1.9% of phosphate was noted after 2 months. It is possible that deposits in Syrup. Glycerophos. Co. may be partly due to the action of peroxidase in the cudbear. The effect of sodium hydroxide, sulphuric acid, dextrose, and sucrose on sodium glycerophosphate is recorded. E. H. SHARPLES.

Determination of camphor in camphorated oil. L. DÁVID (Pharm. Ztg., 1929, 74, 437—438).—The solution of camphor in sesamé oil is treated with aqueous calcium hydroxide suspension to saponify the oil, the camphor distilled out of the resulting mixture with steam, extracted from the distillate, and weighed. S. I. LEVY.

Ergot and ergot extract. L. VAN ITALLIE [with (Frl.) HARMSMA] (Schweiz. Apoth.-Ztg., 1928, 66, 423—425; Chem. Zentr., 1928, ii, 1916—1917).—Various methods for the evaluation of ergot have been

examined; the absorption curves of ergotinine, ergot-oxin, ergotamine, and ergotaminine in the ultra-violet have been studied. Optical methods are suitable for the evaluation. Extraction of ergot according to the Dutch pharmacopœia with 0.25% tartaric acid is incomplete. The alkaloid content of well-preserved ergot falls less rapidly than that of extracts. A. A. ELDRIDGE.

Determination of total alkaloid content of *Lobelia inflata* and its preparations. L. DÁVID (Pharm. Ztg., 1929, 74, 419—420).—The filtered solution of the alkaloids is made alkaline and extracted with ether; the ethereal solution is clarified and an aliquot proportion evaporated in a tared flask. S. I. LEVY.

Determination of theobromine in diuretin and its derivatives. H. BOTE and A. LINDNER (Pharm. Ztg., 1929, 74, 418—419).—An excess of iodine in potassium iodide solution is added to the material dissolved in sodium hydroxide solution, the mixture acidified, the precipitated theobromine periodide removed by filtration, and the excess of iodine in the filtrate determined by titration. S. I. LEVY.

Detection of extracts and powders of medicinal plants in pharmaceutical preparations. F. DI STEFANO (Annali Chim. Appl., 1929, 19, 107—109).—A pharmaceutical preparation made from a solution of the active principles of a drug will not contain cell residues or cellulose, which will, however, be present if the powdered plant product itself has been used. To determine the cellulose, about 0.5 g. of the preparation is mixed in a 200—300 c.c. Kjeldahl flask with 50 c.c. of pure glycerol (*d* 1.23) containing 20 g. of sulphuric acid per litre, the liquid being then gently boiled under reflux for 30 min. with occasional shaking. When the flask has cooled to 90—100°, 100 c.c. of boiling distilled water are added and the solution is heated to boiling and filtered while hot through an alundum crucible. The residue is washed with about 400 c.c. of boiling water, then with alcohol at 80—90° until the washings are colourless, and finally with alcohol-ether. After being dried at 110° for 1 hr. and weighed, the crucible is heated in a Bunsen flame and weighed again, the difference between the two weights giving ash-free cellulose. The cellulose contents of a number of drug powders are given. If only detection of the cellulose is required, the precipitate obtained as above is collected on a glass crucible with porous base and dissolved in Schweitzer's reagent, acidification with dilute sulphuric acid yielding a flocculent precipitate of hydrocellulose. T. H. POPE.

Examination of some drugs according to D.A.B. VI. R. DIETZEL and F. SCHLEMMER (Apoth.-Ztg., 1928, 48, 907—909; Chem. Zentr., 1928, ii, 1918—1919).—The official methods for the following determinations have been examined, and certain modifications are suggested: potassium hydroxide, white (mercury) precipitate, sodium diethylbarbiturate and phenylethylbarbiturate, magnesium peroxide, ferrous sulphate, arsenious acid, "chloramine." Sodium thiosulphate is standardised by means of potassium bromate, and the effect of atmospheric oxidation in iodometric titrations is discussed. A. A. ELDRIDGE.

Determination of alkaloids in admixture with vegetable drugs. G. E. EWE (J. Amer. Pharm. Assoc., 1929, 18, 241—243).—The disturbing influence of the presence of oily and resinous material other than alkaloids which may be included with the alkaloidal residue separated during analysis when fatty acids or soaps are present has been investigated. Modifications of the usual ammonia-chloroform extraction methods applicable to the determination of small proportions of alkaloids in complex admixture with vegetable drugs are described. E. H. SHARPLES.

Testing antiseptic dyes. G. F. REDDISH (J. Amer. Pharm. Assoc., 1929, 18, 237—240).—A method developed by Himebaugh for the control and testing of antiseptic dyes is described. The test simulates actual conditions of use of such materials in that it measures the penetrating power and antiseptic action, and it has given very satisfactory results over a period of two years. E. H. SHARPLES.

Certain constituents of the leaf and their relation to the burning qualities of tobacco. D. E. HALEY, E. S. NASSET, and O. OLSON (Plant Physiol., 1928, 3, 185—197).—The organic acids are combined almost entirely with alkali and alkaline-earth metals. The burning quality is more closely related to the alkalinity of the ash than to the amount of ether-soluble organic acids. The alkalinity of the ash depends on the season and on the potassium salt employed; the sulphate is preferred to the chloride. The "burn" and composition of tobacco depend on base exchange in soil with the removal of "active substances," e.g., calcium chloride, and the ability of the plant to secure sufficient potassium. E. H. SHARPLES.

CHEMICAL ABSTRACTS.

Production of cigarette tobacco by flue-curing. F. J. F. SHAW and K. RAM (Agric. Res. Inst., Pusa, 1928, Bull. No. 187, 19 pp.).—The cultivation, flue-curing, and economics of production of Adcock tobacco in India are fully described. It can be grown successfully. E. H. SHARPLES.

See also A., April, 439, Chloro- and bromo-derivatives of 2:4-dihydroxydiphenylmethane (KLARMANN and VON WOWERN). 441, Synthesis of ephedrine (MANSKE and JOHNSON). 449, Indian turpentine from *Pinus longifolia*, Roxb. (GIBSON and SIMONSEN). 450, Contact transformations of nopinene, terpinene, and terpinolene (ZELINSKI and LEVINA). Individuality of humulene (CHAPMAN). Sylvestrenes and carenes (KONDAKOV). 453, Alkyl-barbituric acids (LYONS and DOX). 455, Porphyrins (KÜSTER and others). Protoporphyrin (HAMSIR). 457, Opium alkaloids. Synthesis of *dl*-apomorphine dimethyl ether (SPÁTH and HROMATKA; AVENARIUS and others). *Strychnos* alkaloids (LEUCHS). Derivatives of mono- and di-aminohydroxyphenylarsinic acids (FISHER and RAIZISS). 463, Syntheses of guanidine derivatives (KUMAGAI and others). 474, Micro-determination of adrenaline (CHIKANO). 475, Behaviour of insulin on irradiation with X-, radium, and ultra-violet rays (DEN HOED and others). 477, *l*-Asparagine in *Ulex europæus* (BREDEL). Alkaloids of *Corydalis ambigua* (CHOU and CHOU). Alkaloids of *Bocconia frutescens* (MILLER). Action of thorium-X

on active principle in certain medicinal plants (ÅVERSENQ and others). 478, Determination of organic arsenic acids (URBSCHAT).

PATENTS.

Process for arresting the glycolysis of living cells and its application to manufacture of technical or commercial products. SCHERING-KAHLBAUM A.-G. (B.P. 284,643, 5.1.28. Ger., 2.2.27).—Glycolysing cells (yeast cells, anaerobic bacteria, tumour cells) are treated with an organic fluoro-compound. The glycolytic action of yeast cells is reduced by $M/10^5$ -sodium *p*-fluorobenzoate to the extent of 62%, whilst respiration is increased by 11%. C. HOLLINS.

Composition for therapeutic uses. K. CONNELL (U.S.P. 1,704,727, 12.3.29. Appl., 17.1.25).—A briquette, which generates chlorine on treatment with acid, is partially coated with an inert material, so that the whole is not decomposed immediately. F. G. CLARKE.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Bleaching action of desensitisers. LÜPPO-CRAMER (Z. wiss. Phot., 1929, 26, 344—351).—Curves are given showing the effect of concentration of desensitisers, alone and in presence of potassium bromide, on the photochemical bleaching action of Pinacryptol Green and phenosafranine. These reveal that the sensitivity of a photographic plate is largely influenced by the particular concentration of the desensitiser used. The desensitising action of phenosafranine with potassium bromide on an old Agfa portrait film is illustrated, and experiments with Isoquinoline Red, Orthochrome T, and Ethyl Red, each in conjunction with potassium bromide, are briefly described. H. T. S. BRITTON.

See also A., April, 408, Lattice energies and photochemical decomposition of silver halides (SHEPPARD and VANSELOW). Intermediate regressions (LÜPPO-CRAMER). Limiting short wave-length for the Herschel effect (MAUZ).

PATENT.

Preparation of photosensitive photographic emulsions. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 307,716, 7.11.27).—Processes are described for the preparation of photographic emulsions similar in type to the ordinary print-out, gaslight, and bromide emulsions, containing mercuric oxalate or its insoluble complex ammine compounds as the light-sensitive substances. Such emulsions may be sensitised by the addition of salts of the heavy metals, mercury, silver, thallium, gold, and platinum, or by dyes such as eosin, erythrosin, etc.

J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Cellulose nitrates. J. CRAIK (5th Coll. Symp. Mon., 1928, 273—286).—The results of experiments on nitrating, denitrating, and "nitrating-up," and on the solubilities of the products, are consistent with the view that there are three cellulose nitrates.

CHEMICAL ABSTRACTS.

Falling-ball method for measurement of the apparent viscosity of cellulose nitrate solutions. J. K. SPEICHER and G. H. PFEIFFER (5th Coll. Symp. Mon., 1928, 267—272).—Mutually consistent results

were obtained. For low-, but not for high-, viscosity types the same relation obtains between the results with the falling-ball and capillary-tube methods as is observed for castor oil.

CHEMICAL ABSTRACTS.

PATENT.

Percussion caps and the like. W. DICKSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 307,560, 19.9.28).—The exposed surface of the priming composition of percussion caps is protected from moisture and from damage during insertion into the cartridge by being coated with a film of non-explosive esters or ethers or mixed esters and ethers of cellulose. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Activated sludge process. A. KUFFERATH (Z. angew. Chem., 1929, 42, 65—67).—Sewage containing phenols etc. from tar works can be dealt with in the activated-sludge process by the addition of selected micro-organisms. In Germany the aeration surface is only 6% of the bottom surface as compared with 16% in England. Ferroconcrete is used for the bottom and walls and supports; for the aeration vats and air-tubes iron coated with zinc is used. Special porous filter plates are used. A p_H of 7.2 appears to give the most favourable results, and a small addition of ferrous sulphate or chloride on the first day is advantageous.

R. BRIGHTMAN.

Effect of pure soaps on the bactericidal properties of phenolic germicides. B. HAMPIL (J. Bact., 1928, 16, 287—300).—Sodium oleate, sodium myristate, potassium palmitate, and potassium stearate inhibit the bactericidal properties of phenol, *m*-cresol, *sec*-butylphenol, butylresorcinol, and hexylresorcinol; hence it is impossible to produce a germicidal soap by the addition of small quantities of a phenolic compound. The germicidal activity of phenol is affected by soaps in the dispersed phase more than by soaps in the gel phase, apparently by removal of phenol from solution.

CHEMICAL ABSTRACTS.

Operation of the calcium hydroxide process [of water treatment]. M. GROECK (Gas- u. Wasserfach, 1929, 72, 319—324).—Bücher's method of treating water to prevent corrosion in pipes and the report on its operation at the Remscheider installation are discussed and criticised. It is pointed out that the theoretical basis advanced is inadequate, since the concentration of hydroxyl ions is too low to inhibit corrosion. The lime process is not new, and the figures given in the report are not conclusive.

S. I. LEVY.

Investigation of soda water for lead. H. W. PETHERICK (Bull. Hyg., 1928, 3, 402).—Samples (in Brisbane) contained lead originating from the solder in carbonators. Material free from lead was then employed, although in some cases chemical treatment of the water was necessary.

CHEMICAL ABSTRACTS.

Tannery waters. MEZEY.—See XV.

PATENTS.

Plant for dealing with sewage. PULSOMETER ENGINEERING Co., LTD., and J. BJÖRNSTAD (B.P. 307,561, 20.12.27).—The sewage is passed through a suitable screen and any matter deposited thereon is regularly removed and periodically disintegrated into pieces of

sufficiently small size to pass through the screen prior to discharge along with the sewage.

C. JEPSON.

Treatment of sewage. E. VON SPRINGBORN (B.P. 307,582, 19.1.28).—Sewage is passed through a series of tanks containing combustible material, *e.g.*, bituminous coal, which is reduced in size in successive tanks from about a 4 in. to $\frac{1}{4}$ in. cube, and finally through a straining filter of coke. When the filtering material is loaded with sludge it is removed and burned at a dull red heat with a limited air supply, and the steam and gases thus generated are condensed and collected with recovery of ammonia, methane, light oils, and grease.

C. JEPSON.

Biological purification of waste waters from yeast and sugar factories, distilleries, and other factories. A./S. DANSK GAERINGS-IND., Assees. of LESIENICKA FABR. DROZDY PRASOWANYCH I SPIRYTUSU SPOLKA AKCYJNA (LESIENITZER SPIRITUS U. PRESSHEFE-FABR. A.-G.) (B.P. 284,267, 25.1.28. Austr., 26.1.27).—The wastes from such factories are approx. 20 times the strength of normal sewage and contain mainly betaine substances, albumin, volatile vegetable acids, humin substances, and sulphur compounds. It is proposed to decompose the organic material by means of pure cultures of bacteria specifically suitable for the purpose and preferably in the zoögloea state, each group to be dealt with separately unless the organisms responsible are capable of working harmoniously together. The success of the process largely depends on the maintenance of the bacteria etc. in pure culture, and hence the waste should be sterile; as that from yeast factories and spirit distilleries consists largely of waste water from the stills, this condition is readily fulfilled, otherwise it would be necessary to sterilise, *e.g.*, with chlorine. In the sterile waste the decomposition of sulphates into sulphides does not occur, and so a frequent cause of nuisance is avoided.

C. JEPSON.

Biological purification of waste water. H. WADE. From A./S. DANSK GAERINGS-IND. (B.P. 307,587, 25.1.28).—The biological purification of waste water may be divided up into stages corresponding to the groups of organic impurities contained therein. In each stage the water is inoculated with a specific organism preferably in pure culture and in the form of zoögloea. When the reaction is complete the organisms are removed, and the degradation is carried a stage further by another organism in a similar way, the process being repeated until the desired stage of purification is attained. The process may be applied on sand or other filters, in tanks, or by a variation of the activated sludge process using an inorganic precipitate inoculated with the desired organism.

C. JEPSON.

Treatment of polluted waste water. J. T. TRAVERS, ASSR. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,703,373, 26.2.29. Appl., 25.3.27).—Alkaline liquids containing polluting organic matter are treated with 1%, by vol., of a solution of sulphuric acid (1%) containing 9% of clay in suspension.

C. JEPSON.

Softening of water. S. A. SANFORD (U.S.P. 1,705,589, 19.3.29. Appl., 30.11.26).—A mixture containing barium fluoride, but no other precipitant, is added.

F. G. CLARKE.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 7, 1929.

L—GENERAL; PLANT; MACHINERY.

Lord Kelvin's law in chemical manufacture. M. H. DOW (Ind. Eng. Chem., 1929, 21, 368).—The general application of the principle that elaborations in plant to obtain working economies are only justifiable up to the point where the capital charges become equal to the economy obtained is discussed and illustrated.

C. IRWIN.

Reactions [and corrosion phenomena] at high pressures. E. BERL (Chim. et Ind., 1929, 21, 452—465).—The variation of the composition limits necessary for the explosion of mixtures of combustible gases and air with increase of pressure from 0 to 800 atm. is depicted graphically. The narrowing of the limits which occurs in some cases at pressures up to 25—40 atm. is not continued at higher pressures, and is attributed to the heat supplied by the ignition spark. For hydrocarbon-air mixtures the limits at higher pressure widen greatly. In the combustion of water-gas with insufficient air, which at low pressures results in the preferential combustion of hydrogen, with increase of pressure an increase of formation of carbon dioxide occurs. With hydrocarbons a similar change occurs, and at high pressures it is suggested that the first stage of the combustion is a splitting into carbon and hydrogen, and that the oxidation of the former takes place by the water-gas reaction. Similar curves are given for mixtures of air with the vapours of ether, hexane, etc. Anomalies in the case of cyclohexane and hexane may be due to the elimination of hydrogen. The effect of various liquids on iron at high pressures was studied by the aid of a cast steel bomb charged with iron filings and the corroding liquid and mechanically shaken. The corrosion was measured by the additional pressure developed due to hydrogen. A trace of caustic soda reduces the corrosive effect of pure water, but greater quantities increase corrosion. As local concentrations around rivet heads might occur, protection by addition of sodium sulphate is preferable. Sodium sulphide, nitrate, and chloride are corrosive, and magnesium chloride is excessively so. This corrosion is reduced by sodium sulphate. Nitrates, chromates, etc. are protective if sufficiently dilute. In the absence of such oxidising agents the product of corrosion is a ferrous oxide-ferrosoferric oxide complex. The protective coating produced by dilute caustic soda or by sodium sulphate can also be produced by pre-treatment with these solutions at high temperatures. The severe action of magnesium chloride is explained by the partial dehydration of magnesium hydroxide under high temperatures and pressures. Such dehydrated hydroxide only reprecipitates ferrous chloride slowly. As sodium hydrogen sulphate is corrosive, sodium sulphate can only

act protectively by reducing the dissociation of magnesium chloride. This hypothesis is confirmed by determinations of p_H , but absolute p_H values are not a measure of power of corrosion in all cases.

C. IRWIN.

Tower packings and the back-pressure created by their arrangement. J. ARNOULD (Chim. et Ind., 1929, 21, 478—482).—The relation between back-pressure, flow of liquid, and flow of gas was determined for 3 types of packing, stoneware rings 25 mm. diam. \times 25 mm. height, flat annular stoneware discs 3 cm. diam. \times 5 mm. thick, and steel spirals, all packed without any regular arrangement. The free space and the surface per unit volume increased in the order mentioned above. It was found that the back-pressure was not a strictly linear function of either the height of the column or the gas or liquid flows. For different packings it varied approximately with the free space. The spirals appear to be the best wherever metals are admissible. The rings and discs have practically the same free space, and when dry give the same back-pressure, but with liquid flow the greater tendency of the rings to retain liquid increases the resistance. On account of their strength they are suitable for the lower parts of high towers. Star packing offers relatively little surface, and gives a higher back-pressure.

C. IRWIN.

Measurement of steam quantity in works' practice. J. L. HODGSON (J. Inst. Fuel, 1929, 2, 235—239).

Calorimeter. UCHIDA.—See II.

PATENTS.

Chemical heat storage. H. LER. CROOKER (B.P. 309,244, 21.1.28).—A solution which yields heat on crystallisation is prepared by heating a mixture of 16 oz. of sodium acetate containing about 40% of water of crystallisation with $\frac{1}{2}$ oz. of calcium chloride, to which sufficient water is added to bring the total up to about 46%.

L. A. COLES.

Drying apparatus. L. BUCK, Assr. to J. HUNTER MACHINE Co. (U.S.P. 1,703,290—1, 26.2.29. Appl., [A] 2.2.27, [B] 1.8.27).—(A) The apparatus comprises a chamber with a false roof some distance below the true roof, so as to form an air space through which the drying air passes before entering the chamber at the far end through a wide opening. The false roof is supplied with narrow apertures, through which the drying air passes in high-velocity jets which compensate for the increase in moisture content of the main volume of drying air, and thus keep the drying conditions uniform. (B) A long chamber, through which passes a conveyor to carry the articles to be dried, is provided with heating elements

disposed along the walls, with fans which circulate the drying air through the heating elements and the goods, and with an exhaust duct extending along a considerable part of its length. Part of the exhausted air is recirculated to the circulating fans. A. R. POWELL.

Evaporator. F. W. SIEVERT (U.S.P. 1,704,064, 5.3.29. Appl., 15.5.26).—The liquid passes through the perforated wall of a horizontal rotary drum, and falls upon horizontal tubes within, which are supplied with heating gases. Deposited solids are continuously removed from the inside of the drum and the surface of the tubes, and the mother-liquor is discharged.

F. G. CLARKE.

Porous body for use as a filter or diaphragm. L. MELLERSH-JACKSON. From SIEMENS & HALSKE A.-G. (B.P. 309,316, 17.4.28).—A compressed mixture of chromium oxide with one or more other chromium compounds, *e.g.*, chromic acid, chromium nitrate, and common salt or sawdust is heated until these chromium compounds are entirely or partly converted into the oxide. The resulting body may be strengthened by means of wire netting.

J. S. G. THOMAS.

Production of dense foam [for cellular building materials etc.]. G. M. THOMSON (B.P. 308,029, 31.1.28).—Foam produced by air passing at constant pressure through a uniformly perforated plate into a solution of a foaming agent rises into contact with vertical, stream-lined, rotary beaters. To prevent the atmosphere reaching the latter from above, and destroying the uniformity of the foam, the outlet is placed above the beaters.

F. G. CLARKE.

Separation and liquefaction of gases. W. S. BOWEN (U.S.P. 1,704,649, 5.3.29. Appl., 9.8.27).—An enlarged chamber, arranged between pressure and expansion conduits, which are in heat-exchange relation, contains a stator and a rotor, through which the gases pass in their passage from the pressure to the expansion conduit. The stator consists of a series of restricted passages, of such size that the critical temperature of one of the gases is reached.

F. G. CLARKE.

Apparatus for heating fluids. S. P. VAUGHN, ASSR. to SURFACE COMBUSTION CO., INC. (U.S.P. 1,700,961, 5.2.29. Appl., 3.9.24).—The apparatus comprises essentially a series of concentric worm tubes, the annular spaces between the worms being packed with porous material, in which the heating gases are burnt on the surface-combustion principle.

A. R. POWELL.

Apparatus for separating liquids. B. D. COMYN (U.S.P. 1,707,077, 26.3.29. Appl., 17.8.28. U.K., 10.8.27).—See B.P. 297,551; B., 1928, 879.

Apparatus for cooling and purifying gases. F. G. INGLIS (U.S.P. 1,707,163, 26.3.29. Appl., 25.4.27. U.K., 21.7.26).—See B.P. 278,118; B., 1927, 897.

Chamber furnaces [with rotatable platforms]. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 300,905, 16.11.28. Ger., 19.11.27).

[Automatic] heat-control systems for furnaces etc. UNITED GLASS BOTTLE MANUFACTURERS, LTD., and W. A. MOORSHEAD (B.P. 309,246, 23.1.28.)

Refrigerating machines of the absorption type. G. MAIURI and R. F. BOSSINI (B.P. 309,050, 29.9.27).

Refrigerating apparatus of the absorption type. N. V. KODOWA REFRIGERATOR CO., and W. A. SLAGER (B.P. 309,007, 2.1.28).

Distilling apparatus (B.P. 309,317).—See XVIII. (B.P. 308,956).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

A new adiabatic calorimeter. S. UCHIDA (J. Fuel Soc. Japan, 1929, 8, 33—37).—An adiabatic bomb calorimeter, consisting of a calorimeter surrounded by a water-jacket containing electrical heating elements and special stirrers, is described. In the determination of the calorific value of a fuel the temperature of the water in the jacket can be accurately controlled to follow that of the calorimeter. This type of apparatus allows determinations to be made independently of room temperature, no radiation corrections are necessary, and the time of operation is considerably shortened.

C. B. MARSON.

Composition of coal. Plant entities as oil-yielding constituents. R. HOLROYD and R. V. WHEELER (J.C.S., 1929, 633—641; cf. B., 1928, 880; 1929, 116).—Distillation of the vitrain and durain fractions of two samples of bituminous coal in a vacuum shows that at about 300° the plant entities present begin to decompose. The active decomposition point (305—310°) is well-marked with the vitrain, but less distinct with the durain. Above this temperature (310—320°) the evolution of gases increases rapidly. A larger volume of gas is obtained from the durain owing to carbon dioxide production from the plant entities. The amount of oils obtained over the range 300° to 320° is also greater with the durain. The durain oil consists of unsaturated hydrocarbons and neutral oxygenated compounds, whilst the vitrain oil is composed of saturated hydrocarbons and phenols. The amount of phenols in the former oil is much smaller than in the latter. Distillation of the megaspore exines isolated from a durain at 100—400°/vac. gives (a) gaseous products rich in oxides of carbon, together with olefines and paraffins, (b) liquid products consisting chiefly of water, unsaturated hydrocarbons, and neutral oxygenated compounds. The difference in character of the products from the vitrain and durain is thus due to the plant entities. Small amounts of nitrogenous and sulphur compounds appear in both sets of products at all distillation temperatures.

H. BURTON.

Decomposition of wood; composition of fossilised wood. S. A. WAKSMAN and K. R. STEVENS (J. Amer. Chem. Soc., 1929, 51, 1187—1196).—Evidence is adduced to show that in the rotting of wood certain constituents are broken down more rapidly than others. The material attacked undergoes complete degradation, so-called intermediate products being actually derived from the cell-substance of the organisms effecting decomposition. Accordingly analyses of wood anaerobically decomposed or fossilised show a large increase in lignin (up to 70—80% of the whole), protein, and ash, and a decrease in celluloses, hemicelluloses, and water- and ether-soluble matter, as

compared with those of healthy wood. Under aerobic conditions, however, the course of decomposition depends on the nature of the organisms present (cf. Rose and Lisse, B., 1917, 457). The conclusions of Marcusson (B., 1926, 809; 1927, 129) are criticised, and it is shown that the acid and alkaline treatments which he employed to remove "oxycelluloses" also dissolve a considerable part of the lignin. For comparison, the solubility of acid lignins from rye straw and from peat in 2% sodium hydroxide at 15°, 100°, and 120° has been determined. Part only of the dissolved matter is reprecipitated by acid.

H. E. F. NOTTON.

Chemical composition of peat. III. Chemical studies of two Florida peat profiles. S. A. WAKSMAN and K. R. STEVENS (Soil Sci., 1929, 27, 271—281. Cf. B., 1928, 880; 1929, 231).—A saw-grass peat (Everglade) and a sedimentary lake profile (Gyttja) were examined. The upper layer of the saw-grass peat was characterised by low contents of ether-, alcohol-, and water-soluble matter, medium hemicellulose and ash, and higher protein and lignin contents. This is typical of low-moor peats. The lower sedimentary layers had low hemicellulose and high ash contents. The lignin and protein constituents were of the same order as those of the upper layers when calculated on the ash-free dry matter. In the decomposition of both upper and lower layers nitrate formation was rapid and carbon dioxide production relatively slow. The ratio carbon dioxide-carbon:nitrate-nitrogen was narrow (1.3—3.3:1). Differences in the fertility of the different peat layers when cultivated cannot be attributed to differences in the activity of micro-organisms.

A. G. POLLARD.

Rapid determination of water in brown coals for briquette manufacture. A. FABER (Z. angew. Chem., 1929, 42, 406—407).—An outline is given of the methods for determining water in brown coals which gained awards in a competition organised by the German brown coal industry. The first prize was given for a rapid method based on the variation of the dielectric constant with the moisture content; other methods proposed rely on direct measurement of the water evolved on heating, on the dilution produced in added standard acetic acid, and on the rise of temperature obtained by the addition of concentrated sulphuric acid.

E. LEWKOWITSCH.

Dry distillation of some Japanese coals. C. IWASAKI and K. SASAKI (Tech. Rep. Tōhoku Imp. Univ., 1929, 8, [2], 95—117).—The rate of evolution of moisture at 105° and of gases and tar on sudden heating at 600° and at 900° and during stepped heating between 150° and 900° has been determined for several varieties of Japanese coals. The results and analyses of the gases are shown in a series of tables and diagrams.

A. R. POWELL.

Gasification of coal in producers. F. MORAWSKI (Gas-u. Wasserfach, 1929, 72, 149—154).—Coal may be continuously gasified by means of air, a mixture of air and steam, or a mixture of air and combustion gas containing carbon dioxide. Assuming pure carbon to be gasified and the gas to lose no sensible heat on leaving

the producer, gasification with air yields, per kg. of carbon, a gas having a heating value of 5688 kg.-cal., a temperature of 1318°, which will give a combustion temperature of 2252°. For gasification with air saturated with moisture at 60°, and with air and combustion gases containing 21% CO₂, the corresponding figures are 7488 kg.-cal., 400°, 2082°; and 6908 kg.-cal., 562°, and 1904°, respectively. A truer comparison is obtained by reckoning the figures on the cold gas at 0°; thus the gas from (a) air alone, (b) air-steam mixture, and (c) air-carbon dioxide has calorific values of 1050, 1570, and 1054 kg.-cal./m.³, with combustion temperatures of 1648°, 1949°, and 1644°, respectively. The best results are thus obtained using an air-steam mixture. The above calculations assume complete decomposition of the steam, which is justifiable, and the production of carbon monoxide and no dioxide from the carbon. The latter assumption is not quite sound, and curves are drawn from which the necessary corrections can be made. Mixtures of air and water vapour can be produced either by the direct introduction of steam into air or by saturating air with vapour by contact with hot water. The latter method is more practical and economical, and with English coals the gas having the highest cal. value and combustion temperature is obtained by using air saturated with moisture at 60°.

W. T. K. BRAUNHOLTZ.

Explosive gas mixtures. P. H. PRAUSNITZ (Oesterr. Chem.-Ztg., 1929, 32, 63—64).—Instead of using a wire gauze to prevent a flame from spreading in an explosive mixture, a permeable disc of glass or silica may be employed; this device is not so liable to rupture. Experiments have been carried out with mixtures of illuminating gas or hydrogen and air or oxygen, and the appearance of the combustion under varying conditions of concentration and pressure is described. If a mixture of hydrogen and oxygen is fired, the explosion is stopped by a porous silica surface, but the combustion on it is so intense that the solid rapidly becomes hot enough to start the explosion anew.

R. H. GRIFFITH.

Gas analysis. J. T. DONNELLY, C. H. FOOT, and J. REILLY (Sci. Proc. Roy. Dubl. Soc., 1929, 19, 165—172).—A number of modifications made in a Bone and Wheeler apparatus are described, which include the use of a six-way tap for introducing reagents by suction and improved mercury-sealed stopcocks; special reference is also made to methods of eliminating the necessity of handling mercury. An apparatus for applying to gas analysis the method of using copper oxide for determining hydrogen in presence of hydrocarbons is also described.

N. M. BLIGH.

Determination of benzene and toluene in gases. F. SCHULZ (Coll. Czechoslov. Chem. Comm., 1929, 1, 228—233).—The gas is shaken in a gas-pipette with a mixture of bromine and anhydrous aluminium bromide whereby benzene is converted into hexabromobenzene, m.p. 326°, and toluene into pentabromotoluene, m.p. 228°, which are collected and weighed. When gasoline hydrocarbons or ethylene are present the bromination products are washed with methyl alcohol saturated with hexabromobenzene and with pentabromotoluene.

A. I. VOGEL.

Determination of small quantities of hydrogen sulphide in gases. H. BACH (Gas- u. Wasserfach, 1929, 72, 154—155).—The gas (2 litres) is slowly passed through a 10-bulb tube containing 10% sodium hydroxide (25 c.c.). The solution is diluted and its sulphide content determined colorimetrically. For the comparison, a standard solution of arsenic trisulphide is used (1 c.c. = 0.1 mg. H_2S or 0.065 c.c. H_2S at N.T.P.), and an alkaline solution of lead acetate (25 g. of sodium potassium tartrate, 5 g. of sodium hydroxide, 1 g. of lead acetate in 100 c.c. water) is added to both solutions. The arsenic trisulphide may conveniently be kept in compressed tablets, weighing 1 g. each and containing 24 mg. of the sulphide and 976 mg. of anhydrous sodium carbonate, from which the standard solution is prepared by dissolving the tablet in 100 c.c. of water. W. T. K. BRAUNHOLTZ.

Industrial uses of gas. F. W. GOODENOUGH (J. Inst. Fuel, 1929, 2, 258—292).—A lecture.

Continuous distillation of tar or crude mineral oil by superheated steam. H. J. V. WINKLER (Chim. et Ind., 1929, 21, 466—472).—The Ab der Halden process effects the distillation of tar at 300° by superheated steam (using 40—50% of the weight of tar) and separates the products by fractional condensation. The tar is preheated to above 100° before it enters the still; it is claimed that the working is not affected by the presence of water in the tar. Four condensers are supplied, the temperature of which is regulated by dephlegmators. The steam injected is condensed in the last two with the light oil and crude naphtha. Comparatively sharp fractionation is obtained. A plant designed for the distillation of low-temperature tar on the same general principle has one high fractionating column into the top of which dilute caustic soda is injected. Sodium phenolate etc. is drawn off from one of the upper sections and treated as usual so that four oil fractions and the tar acids are the products. A plant for the treatment of crude petroleum of low asphalt content works in two stages, the heavy residue from the first still being treated in a second still. Flow sheets are given to show the simplification effected over usual methods. C. IRWIN.

Distinction between American and Russian petroleum. T. P. RAIKOWA (Z. anal. Chem., 1929, 77, 42—46).—2 c.c. of a 0.2% solution of iodine in chloroform, 2 c.c. of a 20% solution of sodium nitrite, and 2 c.c. of the petroleum are placed in a test tube and a 1:3 solution of sulphuric acid is added dropwise, the whole being agitated after each addition. Acid is added until the iodine colour disappears or, if this does not occur, until nitrous fumes are no longer evolved. If the petroleum is of American origin decolorisation occurs after the addition of 2—3 drops of acid. All fractions, including vaseline, of American oils react thus. With Russian oils no decolorisation occurs even after several hours. J. S. CARTER.

Cracking of light oils. G. EGLOFF (Petroleum, 1929, 25, 507—509).—Contrary to the usually accepted view, a high pressure is not necessary for the production of benzine in high yield by the cracking of light oils. Thus an oil of d 0.825—0.834, b.p. 176.7—388.8° is cracked

at 490° and 15.4 atm., yielding 86% of pressure distillate, d 0.750, b.p. about 29—249.3°, 9.5% of coke, and 4.5% of gas (and loss). The pressure distillate is treated continuously with 2.9 lb. of sulphuric acid (d 1.83) per brl., then with water, soda, and water, and is then distilled in a pipe still, giving 65% (on the original oil) of benzine, d 0.735, b.p. 25.5—218.3°, as well as fuel oil, d 0.8325, b.p. 220—348.3°.

W. S. NORRIS.

Anti-knock ratings of pure hydrocarbons. S. F. BIRCH and R. STANSFIELD (Nature, 1929, 123, 639).—A reply to Nash and Howes (B., 1929, 346), and a description of methods employed. A. A. ELDRIDGE.

[Anti-knock ratings of pure hydrocarbons]. A. W. NASH and D. W. HOWES (Nature, 1929, 123, 640).—A reply to Birch and Stansfield (preceding abstract).

A. A. ELDRIDGE.

Coal-dust firing for boilers and industrial furnaces. H. BERG and E. VOGT (J. Inst. Fuel, 1929, 2, 240—257).

Reactions at high pressures. BERL.—See I.
Explosives in coal mines. ANON.—See XXII.

PATENTS.

Coke ovens. L. WILPUTTE, Assee. of W. H. PAVITT (B.P. 308,120, 30.5.28. U.S., 3.4.28).—A coke oven is described with horizontally elongated coking chambers, having heating walls with vertical flues, and heating gas inlets opening at different levels; in each inlet the supply of fuel gas can be regulated whereby a better distribution of the heating effects is obtained and "hot bottoms" are avoided. C. B. MARSON.

Coke oven. A. PUTSCH, Assr. to KOPPERS CO. (U.S.P. 1,705,029, 12.3.29. Appl., 6.8.19. Renewed 1.6.28).—The heating walls between the coking chambers of an oven contain two parallel series of triangular flues, facing respectively the chambers on either side of each heating wall. Cross-regenerators below the chambers communicate with all the flues in the adjacent series in each adjacent heating wall. Each flue series is provided with gas supply and is adapted to be fired in alternation with the contiguous series in each heating wall. A. B. MANNING.

Coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS CO. (U.S.P. 1,705,841, 19.3.29. Appl., 15.6.21).—The oven has vertical combustion flues arranged in inside and outside groups. Separate inside and outside regenerators extend crosswise of the battery below the coking chambers and are individually connected with the inside and outside groups of flues respectively. Below the regenerators are tunnels formed by crosswise-running walls. Means are provided for supplying coke-oven gas, at each reversal period, to some of the inside and outside groups of flues. Reversing valve connexions for controlling the supply of air to the regenerators lead from the latter to the tunnels below. Means are provided for supplying an alternative fuel gas, e.g., producer gas, to some of the regenerators. A pair of waste-gas tunnels extend along each side of the battery; some of the regenerators are connected with one pair of tunnels and the remainder with the other pair. A. B. MANNING.

Apparatus for [heat] treatment of [carbonaceous] materials. H. M. ROBERTSON (U.S.P. 1,706,128, 19.3.29. Appl., 18.6.27).—Solid carbonaceous materials are distilled in a tunnel retort comprising a preheating, a heating, and a cooling chamber in series. At the outlet end of the heating chamber are combustion chambers, the hot gases from which pass through pipes extending through the heating chamber. Air pipes pass from the cooling chamber through the heating chamber and open into the preheating chamber, and means are provided for exhausting the vapours from the last-named.

A. B. MANNING.

Apparatus for destructive distillation of powdered fuel. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H. (B.P. 285,015, 8.2.28. Ger., 8.2.27).—The powdered fuel is fed to a distributor at the lower end of a vertical distillation vessel. The material is carried up through the vessel and thence through a dust separator. The distillation vessel is externally heated, being preferably surrounded by a heating jacket supplied with fuel gas and air, and packed with radiating bodies for flameless combustion. The fuel distributor has a funnel-shaped mouthpiece with a constricted portion at the bottom for the passage of the fuel, and means at the upper end, *e.g.*, a centrifugal device consisting of a set of rotating rings, for spreading the powdered fuel over the entire cross-section of the vessel. In a second type of distributor the powdered fuel is injected into the vessel by a stream of gas blown through a nozzle, the position of which relative to the constricted portion of the distributor is adjustable. The gas used for this purpose may be a portion of the distillation gas from which the tar has been removed, and which has been preheated by the waste gases from the heating jacket.

A. B. MANNING.

Distilling carbonaceous material. F. PUENING (U.S.P. 1,698,240, 8.1.29. Appl., 6.11.22).—Low-temperature carbonisation of bituminous coal etc. is effected in a furnace with movable walls, which are first brought to, *e.g.*, 760° by contact with hot gases, while the side walls are retracted. The side walls are then brought against the ends of the heating walls by suitable mechanism, and the coal is charged in and distilled at the desired final temperature, *e.g.*, 540°, by heat transfer from the walls. At the end of the carbonisation the side walls are retracted, breaking any adhesion due to a surface formation of coke on the coal, and the product is discharged at the base of the furnace by opening the bottom doors.

R. BRIGHTMAN.

Carbonisation of coal and the like. G. CELLAN-JONES (B.P. 307,811, 12.12.27).—The heat of waste gases of high-temperature coke ovens is utilised for carbonisation of coal at a lower temperature; the semi-coke produced forms 10–30% of the charge of the high-temperature oven.

C. B. MARSON.

Manufacture of absorptive carbon. CARBIDE & CARBON CHEMICALS CORP., Assees. of A. B. RAY (B.P. 291,043, 23.9.27. U.S., 25.5.27).—Non-coking bituminous or sub-bituminous coal, admixed with a "carbonising agent," *e.g.*, zinc chloride, phosphoric acid, is calcined at a high temperature and then activated by

controlled oxidation, preferably with steam. Coking or swelling coals may be used if they are subjected to a preliminary heat treatment under oxidising conditions, whereby their caking power is lost. Lignitic coals may also be used if subjected to a similar pre-treatment.

A. B. MANNING.

Apparatus for extraction of volatile constituents from carbonaceous materials. R. B. PARKER (B.P. 282,415, 14.12.27. U.S., 14.12.26).—A retort with a grate at the bottom and means for intermittently operating the fuel feeding by the ejector without opening the retort to the atmosphere is described. The admission of gas from boxes to the fuel bed can be controlled and the generated or used gases removed from the top of the retort.

C. B. MARSON.

Recovery of valuable organic products from solid carbonaceous materials. I. G. FARBERIND. A.-G. (B.P. 282,691 and 283,545, 21.12.27. Ger., [A] 21.12.26, [B] 13.1.27).—(A) The materials are mixed with a hydrocarbon oil boiling above 300°, and the mixture, without the further addition of hydrogen or water, is heated at 300–400°, the temperature being below that at which the oil cokes, and under a pressure of at least 30 atm. Catalysts may be used if desired. No appreciable amount of gas is formed, and the yield of liquid products from the coal etc. is several times as great as that obtained by low-temperature carbonisation. The process is continuous. The products may subsequently be subjected to low-temperature carbonisation, or may be cracked, or may be extracted with a suitable solvent to separate the liquid from the solid portion. (B) In a modification of the process an oil, boiling range 100–300°, is used, the materials being then heated under a pressure of at least 75 atm. If desired, the process may be carried out in stages.

A. B. MANNING.

Treatment of coal and other solid carbonaceous materials for production of liquid hydrocarbons or other organic substances. I. G. FARBERIND. A.-G. (B.P. 308,633, 21.12.27. Addn. to B.P. 282,691; preceding).—Carbonaceous materials which are to be hydrogenated or cracked are subjected to a preliminary treatment in which they are heated under pressure at a temperature above 100° but below the coking point of the materials. The process is carried out preferably in the presence of water and of a substance with an alkaline reaction, *e.g.*, sodium sulphide. Low-boiling organic solvents and/or gases free from hydrogen may be added during the treatment.

A. B. MANNING.

Manufacture of hydrocarbons and especially those of low b.p. J. Y. JOHNSON. From I. G. FARBERIND A.-G. (B.P. 307,946, 12.9.27).—Coal, tar, mineral oils, their distillation and extraction products, residues, etc. are hydrogenated under high temperature and pressure in the presence of a catalyst consisting of an oxide of a metal or metalloids of groups 3–7 of the periodic system deposited in small amounts on the metals aluminium, silver, manganese, chromium, or chromium-nickel alloy employed in a form presenting a dense coherent surface. The catalysts are applied to the etched metal supports in the form of acidified solutions of the salts of the catalytic elements.

H. S. GARLICK.

Dissolution of coal. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 277,659, 12.9.27. U.S., 14.9.26).—Very finely-divided bituminous coking coal is suspended in a liquid hydrocarbon and the mixture is heated at the softening point of the coal until the liquefiable constituents have dissolved in the oil, in which the residual carbonaceous matter then remains in colloidal form or as a substantially permanent suspension. [Stat. ref.]

A. B. MANNING.

Decomposition of coke-oven gas by cooling to low temperatures. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 289,817, 2.5.28. Ger., 2.5.27).—Substances favourable to formation of explosive compounds, *e.g.*, ammonia, water vapour, and acetylene hydrocarbons, are partially removed, the last-named sufficiently to prevent their separation in solid form during the cooling. The copper parts of the apparatus are protected by a coating of tin, lead, etc.

C. B. MARSON.

[Fuel]-gas purification. KOPPERS Co., Assees. of J. BECKER (B.P. 283,948, 16.12.27. U.S., 22.1.27).—Gas is freed from acidic impurities by alkali solution, and the heated spent lye is regenerated by agitating it with a portion of the purified gas, the latter being afterwards used as a fuel for heating coke ovens etc. (Cf. B.P. 169,996; B., 1921, 762 A.)

C. B. MARSON.

Conversion of natural or artificial inflammable gases into unsaturated hydrocarbons. Soc. D'ETUDES ET D'EXPLOIT. DES MATIÈRES ORGANIQUES, Assees. of SYND. D'ETUDES DES MAT. ORG. (B.P. 282,690, 21.12.27. Luxembourg, 21.12.26).—Combustible gases, *e.g.*, natural gas, coal gas, are mixed with air or oxygen and passed over a catalyst, *e.g.*, platinum black, oxides of iron, copper, nickel, etc., whereby the proportion of unsaturated hydrocarbons in the gas is increased. These are then converted into liquid hydrocarbons by passage over another catalyst such as nickel.

A. B. MANNING.

Manufacture of porous material [for receptacles containing explosive or combustible gases]. J. HAUSEN (B.P. 293,697, 9.7.28. Ger., 9.7.27).—Receptacles for storing, *e.g.*, acetylene dissolved in acetone are filled with inorganic material graded from 0.5 mm. to 0.1 mm. in diam.

L. A. COLES.

Bituminous product. M. R. CONE, Assr. to UNION TRUST Co. (U.S.P. 1,698,878, 15.1.29. Appl., 24.10.21).—Bituminous material is atomised into contact with a stream of air or water at relatively high temperature, but below its fusion point, and of increasing velocity. The particles are finally immersed in a heated liquid which is allowed to cool slowly, giving a mass readily miscible with water.

R. BRIGHTMAN.

Removal of phenols from tars or tar oils. J. KÁRPÁTI and M. G. HÜBSCH (B.P. 283,569, 28.11.27. Hung., 15.1.27).—The tar or tar oil is extracted under pressures of 1.5–6 atm. and at 100–150° with an aqueous solution of a solvent for phenols, such as methyl alcohol. On removing and cooling the aqueous layer the phenols separate therefrom and the residual aqueous solution can be used for the extraction of a further quantity of tar. The aqueous solution used,

which preferably contains about 20% of methyl alcohol, may also contain a neutral salt, *e.g.*, common salt.

A. B. MANNING.

Dehydrator for petroleum emulsions. J. H. C. DE BREY (U.S.P. 1,704,463, 5.3.29. Appl., 20.7.26).—A wide tank is surmounted by a cover having a central aperture communicating with a casing carrying an insulated electrode. In the casing are apertures closed with thin plates which open under internal pressure and are of sufficient size to allow the immediate escape of gases produced by an explosion within the apparatus.

H. S. GARLICK.

Cracking of oils. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 309,227, 3.10.27. Cf. B.P. 302,941; B., 1929, 232).—Tars, mineral oils, etc. are destructively distilled in the presence of organic compounds which contain a radical of a mineral acid and reduce the surface tension of water when added thereto, *e.g.*, benzene-sulphonic acid, phenyl borate, etc.

H. S. GARLICK.

Distillation [of oil]. W. K. LEWIS and N. E. LOOMIS, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,697,195, 1.1.29. Appl., 28.4.27).—Oil is fed continuously to a still heated by direct fire under low pressure, *e.g.*, 25 mm. The vapours escape vertically without constriction or temperature drop through a hot jacket to a condenser. Refluxing to the still is avoided and a drip reflector and collector pan discharge the condensate to a receiver. Residuum is withdrawn continuously from the still.

R. BRIGHTMAN.

Refining of oil. C. B. FORWARD (U.S.P. 1,698,811, 15.1.29. Appl., 19.8.19. Renewed 26.3.27).—Oil is forced under pressure through heating coils in counter-current to superheated steam at, *e.g.*, 540°, and discharged through an atomiser, together with steam, into a closed separator from which the liquid is run off to a residue tank for lubricating oil while the vapours are passed through heating coils heated to 390–480° by superheated steam. The vapour from these coils is freed from carbon before passing into a condenser in countercurrent to a spray of water. Gasoline passes on to further condensers and separators, and the condensed intermediate oil is separated, cooled, and discharged.

R. BRIGHTMAN.

Extraction of oils from carbonaceous material by means of solvents under pressure. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 309,229, 3.10.27. Cf. B.P. 302,941; B., 1929, 232).—Coal, tar, mineral oils, etc. are extracted with, *e.g.*, tetrahydronaphthalene in the presence of substances capable of acting when water is present as wetting and emulsifying agents, *e.g.*, sulphonic acids, fatty acids, saponins, etc.

L. A. COLES.

Manufacture of liquid hydrocarbons from olefines. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 309,199, 28.9.27).—Olefines, or gases containing them, are heated under pressure in contact with a metal that has been coated by heat-treatment with tin, zinc, aluminium, or chromium; the metal may be part of the reaction chamber.

H. S. GARLICK.

Oxidation of petroleum. Cracking and oxidation of [hydrocarbon] oils. C. ELLIS, Assr. to ELLIS-

FOSTER Co. (U.S.P. 1,697,265—6, 1.1.29. Appl., [A] 3.4.23, [B] 13.10.24).—(A) Intermediate oxidation products are obtained by submitting the products of the cracking of petroleum hydrocarbons in the vapour state to the action of oxygen in the presence of a catalyst at incipient red heat. (B) In oxidising kerosene at below red-heat, an amount of oxygen is used sufficient to oxidise only a minor portion of the hydrocarbons.

H. S. GARLICK.

Oxidising petroleum oils. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,697,262, 1.1.29. Appl., 20.8.24).—A mixture of petroleum vapour and air is passed over a catalyst mass maintained at reaction temperature, *e.g.*, 500°, by cooling. Thus vapour obtained by cracking kerosene at 500–600° is mixed with dry air in a Venturi tube, cooled, and passed through a U-tube heated in a lead-bath at 425–450°, containing pumice in one leg and pumice coated with vanadium oxide in the other, the temperature of the exit gases just above the catalyst being 320–360°. Substantial amounts of phthalic anhydride are formed. The oxidation may be effected under pressure. The amount of aromatic derivatives produced depends on the proportion of naphthenes in the original oil.

R. BRIGHTMAN.

Oxidation of kerosene. C. ELLIS, Assr. to ELLIS-FOSTER CORP. (U.S.P. 1,697,263, 1.1.29. Appl., 27.12.21).—Hydrocarbon vapours containing propane, butane, and pentane, and unsaturated hydrocarbons, *e.g.*, from kerosene oil cracked in a tube at 540–590°, are mixed with air and oxidised at 400–500° in presence of a metallic catalyst and steam to control the reaction temperature.

R. BRIGHTMAN.

Oxidation [of oil gas]. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,697,267, 1.1.29. Appl., 22.3.19).—Oil gas containing at least 20% of olefines, obtained, *e.g.*, by cracking heavy petroleum oil, after purification, if necessary, from sulphur compounds, is mixed with air in slight excess of that required for oxidation of the olefines to fatty acids or aldehydes, and passed over a catalyst heated to just below red heat.

R. BRIGHTMAN.

Intermediate partial oxidation product and its manufacture. J. H. JAMES, Assr. to C. F. BYRNES (U.S.P. 1,697,653, 1.1.29. Appl., 7.3.19. Renewed 4.8.26).—Hydrocarbon vapour mixed with air is heated to 230–500° and passed through a catalytic screen of, *e.g.*, blue oxides of molybdenum, affording a mixture of aliphatic aldehydes and acids in which the former are in excess. *E.g.*, gas oil, b.p. 250–295°, at 270° and 32 sec. contact gave a liquid product containing 45% of oxy-acids and 55% of other products (alcohols to acids) including unchanged hydrocarbons. Similarly, kerosene, b.p. 250–295°, in contact (33 sec.) with uranyl uranite and uranate catalyst at 310° gave a liquid containing 70% of aldehydes and 30% of aldehyde-acids.

R. BRIGHTMAN.

Production from montan wax of valuable products suitable for fixing solvents used in shoe creams etc. J. Y. JOHNSON. From I. G. FARNENIND. A.-G. (B.P. 308,996, 3.12.27).—Deresinified montan wax is treated with a 25–50% excess of oxidising agent over that required for complete bleaching. H. S. GARLICK.

Retort for treating carbonaceous matter. P. DVORKOVITZ (U.S.P. 1,706,825, 26.3.29. Appl., 9.6.28. U.K., 8.3.27).—See B.P. 296,793; B., 1928, 841.

Distillation apparatus [for carbonaceous material]. P. M. and E. M. SALERNI, Assrs. to E.M.S. INDUSTRIAL PROCESSES, LTD. (Re-issue 17,251, 2.4.29, of U.S.P. 1,541,071, 9.6.25).—See B., 1925, 535.

Treatment [distillation] of carbonaceous materials. A. M. A. STRUBEN (U.S.P. 1,706,468, 26.3.29. Appl., 15.1.26. U.K., 26.1.25).—See B.P. 250,699; B., 1926, 523.

Preparation of bituminous emulsions. H. PLAUSON, Assr. to MINERAL A.-G. BRIG (U.S.P. 1,706,590, 26.3.29. Appl., 12.1.27. Italy, 31.7.26).—See B.P. 276,543; B., 1927, 836.

Manufacture of petroleum sulphonic acids and salts thereof. P. I. SCHESTAKOV (U.S.P. 1,706,940, 26.3.29. Appl., 6.2.26. Ger., 17.2.25).—See B.P. 247,940; B., 1926, 864.

Bleaching of montan wax. W. PUNGS and T. HELLTHALER, Assrs. to I. G. FARNENIND. A.-G. (U.S.P. 1,698,450, 8.1.29. Appl., 26.11.27. Ger., 3.12.26).—See B.P. 299,133; B., 1928, 919.

[Removal of carbon deposits from interior surfaces of] coke-oven apparatus. KOPFERS Co., Assces. of J. A. B. LOVETT (B.P. 285,070, 16.12.27. U.S., 12.2.27).

Burner for liquid fuels. R. ZANIROLI (B.P. 278,374, 29.9.27. Italy, 29.9.26).

Gases containing nitrogen, hydrogen, and carbon dioxide (U.S.P. 1,698,722). Purification of gases (U.S.P. 1,698,718).—See VII. Bitumen emulsions (B.P. 308,389).—See IX. Briquettes (B.P. 282,104).—See X. Active carbon (U.S.P. 1,701,272).—See XI.

III.—ORGANIC INTERMEDIATES.

Thermodynamic consideration of the synthetic methyl alcohol process. K. K. KELLEY (Ind. Eng. Chem., 1929, 21, 353–354; cf. B., 1926, 214; A., 1929, 251).—Recent determinations by the author of the sp. heat of methyl alcohol at temperatures down to 16° Abs. and the adoption of a new value for the entropy of hydrogen have caused a modification of the free energy equation previously suggested for the formation of methyl alcohol from carbon monoxide and hydrogen. This equation now becomes $\Delta F^\circ = -20,740 + 45T \times \log T - 0.01586T^2 - 69.4T$. Values of the equilibrium constant K (in terms of fugacities) at temperature intervals of 100° from 300° to 900° Abs. are given where $K = (f_{\text{MeOH}})/(f_{\text{H}_2})^2(f_{\text{CO}})$. These values are, respectively 895×10^4 , $196,262 \times 10^{-3}$, 303×10^{-5} , 124×10^{-6} , 115×10^{-7} , 191×10^{-8} . This series does not agree with recent results of Lewis and Frolich (B., 1928, 359), Audibert and Raineau (B., 1928, 920), Brown and Galloway (B., 1928, 780), and Morgan, Taylor, and Hedley (B., 1928, 439), all of whom worked at high pressures. Correction from pressure to fugacity cannot be made with certainty under these conditions. Smith and Branting (A., 1929, 265) have obtained a value of $K = 5.57 \times 10^{-4}$ at 576.9° Abs. at 1 atm. total pressure, which, although

more in accord with the author's calculated value, does not agree sufficiently well to give a satisfactory experimental confirmation of the equation. Possible reasons for this disagreement are discussed. H. INGLESON.

Methyl alcohol from hydrogen and carbon monoxide. II. **Dimethyl ether.** R. L. BROWN and A. E. GALLOWAY (Ind. Eng. Chem., 1929, 21, 310—313; cf. B., 1928, 780).—The hourly production of methyl alcohol has been studied under the following conditions: catalyst normal zinc chromate, gas ratio hydrogen: carbon monoxide 2:1, average pressure 180 atm., temperatures 300—400°, space velocities 3000 and 7500, catalyst volume 250 c.c. The catalyst employed has been found to accelerate both the reactions $\text{CO} + 2\text{H}_2 = \text{MeOH}$ and $2\text{MeOH} = \text{Me}_2\text{O} + \text{H}_2\text{O}$. As, however, normal zinc chromate is primarily a methyl alcohol-forming catalyst the second reaction only takes place to a limited extent under conditions which favour high hourly yields of the alcohol. The presence of methyl ether among the products has been established by the isolation of the liquid, the determination of its density, the calculation of its mol. wt., and the examination of the properties of the gas. At a space velocity of 3000, the conversion of carbon monoxide into methyl alcohol increased from 7% at 300° to a maximum of about 30% at 375°, and decreased to 20% at 400°. At the same space velocity the conversion into methyl ether increased with the temperature from 1.0% at 307° to 5.2% at 397°. The effect of increasing the space velocity at a given temperature is to decrease the percentage of carbon monoxide converted into the ether.

H. INGLESON.

Preparation of salts of naphthylsulphuric acid with aromatic organic bases, and their application to the dyeing of silk and wool by diazotisation on the fibre. E. I. ORLOV (Ukraine Chem. J., 1928, 3, [Tech.], 181—182).—Salts of β -naphthylsulphuric acid with various aromatic bases are diazotised on wool and silk fibres, which are thereby dyed various colours according to the base used. R. TRUSZKOWSKI.

Possibility of industrial poisoning with ethylene dibromide. M. KOCHMANN (Münch. med. Woch., 1928, 75, 1334—1336; Chem. Zentr., 1928, ii, 2041).

2:4-Diaminodiphenylamine. KARPUKHIN.—See IV. Dehydration of alcohol. SMITH.—See XVIII. Diethyl ether. KING. Synthetic drugs. KAUFMANN and others.—See XX. Determination of diphenylamine and diphenylnitrosoamine in presence of their derivatives. RYAN and others.—See XXII.

PATENTS.

[Manufacture of] methyl ether. DELCO-LIGHT Co., Assees. of F. R. BICHOWSKY (B.P. 278,353, 23.9.27. U.S., 23.9.26).—Methyl ether may be synthesised by passing carbon monoxide and hydrogen successively over a hydrogenating and then a dehydrating catalyst, or by passing the gas mixture over a mixed hydrogenating-dehydrating catalyst. Suitable dehydration catalysts are partially hydrated alumina or titania, thoria, or silica gel. Hydrogenation is conducted at 500° and 125 atm. or more, and the dehydration at 300° and 25 atm. or more, respectively. When the mixed catalyst is

used the intake end of the furnace is heated at 500° and the other end at 300° only, and a corresponding drop in pressure is maintained by a suitable conformation of the furnace, or by a special distribution of the catalyst, which offers considerable resistance to the passage of the gas. When methyl alcohol itself is used as the starting material, and the hydrogenation catalyst dispensed with, an inert carrier such as carbon dioxide is used. E. HOLMES.

Production of acetic acid. H. D. GOLDING, F. D. LEICESTER, H. S. HIRST, S. W. ROWELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 308,937, 18.7.28).—Acetaldehyde dissolved in acetic acid in the liquid phase, or acetaldehyde vapour passed into acetic acid, may readily be oxidised by means of air or oxygen at 30—40° in the presence of cobalt acetate as catalyst. Manganese acetate is a good catalyst once the reaction is started, but it requires a small amount of cobalt acetate as a "starter." E. HOLMES.

Continuous manufacture of anhydrous acetic acid from its aqueous solutions. SOC. ANON. DES DISTILLERIES DES DEUX SEVRES (B.P. 300,246, 30.1.28. Fr., 10.11.27. Addn. to B.P. 296,974; B., 1929, 349).—A heat-exchanger is introduced into the plant previously described. C. HOLLINS.

Production of ethyl alcohol. H. G. SMITH, C. J. BRIDGER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 308,468, 19.3.28).—Water and ethylene at 40 atm. are heated to 300° (pressure 150—200 atm.) in presence of inorganic salts having affinity for ethylene (e.g., mercuric chloride, cuprous chloride, silver nitrate). About 10% conversion into ethyl alcohol results. C. HOLLINS.

Production of ethyl alcohol. R. E. SLADE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 308,859, 30.1.28).—Ethylene (5—10 vols.) is treated with steam at 400—500° and 25—200 atm. in the presence of a dehydrating catalyst such as thoria or phosphoric acid deposited on charcoal. A high space velocity (10,000—50,000 litres of gas at N.T.P. per hour per litre of catalyst) is preferable, and heat interchange between the outgoing and incoming gases assists the reaction. E. HOLMES.

Manufacture of butyl alcohol and other organic compounds from ethyl alcohol. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 282,448, 19.12.27. Ger., 18.12.26).—Ethyl alcohol vapour is passed over a catalyst (oxides or hydroxides of magnesium, calcium, barium, manganese; sodium ethoxide) at 400—500°. Barium oxide gives 20—30% conversion into butyl alcohol. C. HOLLINS.

Production of ketonic alcohols ["diacetone alcohol"]. NOBEL INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 308,285, 21.10.27).—Acetone is converted into "diacetone alcohol" by treatment at 6—25° with a suspension of potassium hydroxide in benzene or other liquid hydrocarbon in presence of an inert extender (fullers' earth, slate, talc, alumina, gypsum, etc.). C. HOLLINS.

Manufacture of ethylene glycol monoalkyl ethers. C. O. YOUNG, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,696,874, 25.12.28. Appl., 7.2.24).—Ethylene

oxide and absolute alcohol are heated at 150° for 12 hrs. or at 200° for 3—4 hrs. C. HOLLINS.

Manufacture of esters. W. I. BANNISTER, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,695,449, 18.12.28. Appl., 18.2.27).—In the azeotropic method of esterification, improved yields are obtained in presence of a dehydrating agent (aluminium sulphate) which acts as a "transfer agent," the water being eventually nearly all entrained as ternary mixture. C. HOLLINS.

Preparing metallo-organic compounds [lead ethyl]. C. A. KRAUS and C. C. CALLIS, Assrs. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,697,245, 1.1.29. Appl., 5.4.23).—Lead alloyed with about 10% of sodium is disintegrated in an inert non-oxidising atmosphere, *e.g.*, in the reaction vessel itself, and treated with ethyl chloride at 35—70° for 2—6 hrs.; 0.25—0.3% of anhydrous aluminium chloride, ferric chloride, or zinc chloride may be added as catalyst. R. BRIGHTMAN.

Manufacture of tetra-alkyl lead [lead tetraethyl]. W. S. CALCOTT and H. W. DAUDT, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,692,926, 27.11.28. Appl., 15.10.27).—Sodium-lead alloy is fed gradually through a hopper into an autoclave containing ethyl chloride at 80—100°, the temperature of the hopper being maintained at about 30—40° to prevent premature reaction. C. HOLLINS.

Purification of crude carbon tetrachloride. W. B. VAN ARDOEL and H. P. VANNAH, Assrs. to BROWN CO. (U.S.P. 1,697,483, 1.1.29. Appl., 6.3.25).—Crude carbon tetrachloride (*e.g.*, 77% carbon tetrachloride, 20% carbon disulphide, and 3% sulphur chloride), obtained by distillation from the reaction product of 2 pts. of sulphur monochloride, containing some dichloride, and 1 pt. of carbon disulphide, is agitated with a solution containing about 5% of sodium hydroxide and 5% of sodium sulphite. The neutral carbon tetrachloride is washed and distilled with steam and the distillate dried and fractionated, the first fractions containing carbon disulphide being returned for further treatment or reaction. R. BRIGHTMAN.

Esters of hydrogenated aromatic alcohols. R. H. VAN SCHAACK, JUN., Assr. to VAN SCHAACK BROS. CHEM. WORKS (U.S.P. 1,697,295, 1.1.29. Appl., 21.11.27).—A cyclohexanol is heated with an organic acid containing more than seven carbon atoms. *cycloHexyl stearate*, m.p. 25°, b.p. 232°/8 mm., is specially claimed. C. HOLLINS.

Production of calcium or other alkaline-earth benzoates and benzoic acid. D. A. W. FAIRWEATHER, E. G. BECKETT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 309,202, 29.9.27).—Mixtures of calcium phthalate and calcium hydroxide, with or without the addition of diluent solids, *e.g.*, calcium carbonate, are heated at the usual pressure under such conditions that the heat of reaction is removed from the mixture and the maximum temperature does not exceed about 440—450°, *e.g.*, by passage by means of a spiral conveyor through a heated tube. Calcium benzoate is recovered from the product by extraction with boiling water, and is converted into benzoic acid by the usual method. L. A. COLES.

Production of substituted benzoyl halides. P. F.

BANGHAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 308,231, 18.11.27).—A substituted benzotrichloride is heated at 110—175° with the corresponding benzoic acid in presence of zinc chloride and in absence of a diluent. C. HOLLINS.

4'-Halogeno-2-benzoylbenzoic acid intermediates. W. MÜLLER and E. APPENZELLER, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,699,671, 22.1.29. Appl., 30.12.27. Ger., 12.3.27).—Phthalic anhydride, chlorobenzene, and finely-divided aluminium chloride are stirred at 15° and the resulting oily mass is heated at 80—100° in thin layers, hydrogen chloride being removed. The product is a foamy, brittle mass, readily converted by acid into 4-chloro-*o*-benzoylbenzoic acid. C. HOLLINS.

Alkyl- and aralkyl-resorcinolcarboxylic acids [disinfectants]. H. STENZL (U.S.P. 1,697,332, 1.1.29. Appl., 1.6.27. Ger., 4.6.26).—A 4- β -phenylethylresorcinol is carboxylated by heating with sodium bicarbonate solution at 140° or by heating the sodium salt with carbon dioxide at 100—120° to give 4- β -phenylethylresorcinol-6-carboxylic acid, m.p. 208°. 4- β -p-Bromophenylethylresorcinol-2- and -6-carboxylic acids, m.p. 181° and 204°, respectively, 4-heptyl- (m.p. 109—110° and 163—165°) and 4-benzylresorcinol-2- and -6-carboxylic acids are similarly obtained. C. HOLLINS.

Catalytic oxidation of naphthalene. A. O. JAEGER, Assrs. to SELDEN CO. (U.S.P. 1,692,126, 20.11.28. Appl., 12.12.27).—Silicate zeolites containing or carrying the catalytic elements are used, preferably with a diluent (kieselguhr); any alkalinity should be neutralised. C. HOLLINS.

Production of condensation products from crude cresol and acetone. H. JORDAN, Assr. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,696,769, 25.12.28. Appl., 25.10.27. Ger., 26.10.26).—See B.P. 279,856; B., 1929, 316.

Production of formates [cyclohexyl formate]. R. WEITZEL and F. KREMP, Assrs. to I. G. FARBERIND. A.-G. (U.S.P. 1,698,573, 8.1.29. Appl., 27.4.28. Ger., 28.5.27).—See B.P. 294,396; B., 1928, 703.

Manufacture of alkyl isoalkyl cyclohexanols [menthol]. W. SCHOELLER and H. JORDAN, Assrs. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,696,782, 25.12.28. Appl., 25.10.27. Ger., 26.10.26).—See B.P. 279,855; B., 1929, 237.

Preparation of aromatic mercaptans. E. HOFFA and P. JÖRG, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,706,489, 26.3.29. Appl., 21.9.27. Ger., 18.10.26).—See B.P. 279,136; B., 1929, 164.

6-Aminoacenaphthene-5-carboxylic acid and its manufacture. W. ECKERT, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,697,111, 1.1.29. Appl., 5.7.27. Ger., 24.7.26).—See B.P. 274,902; B., 1928, 293.

Phenols from tar or tar oils (B.P. 283,569). Liquid hydrocarbons from olefines (B.P. 309,199). Oxidation of hydrocarbons (U.S.P. 1,697,262—3, 1,697,267, and 1,697,653).—See II. Pickling of metals (B.P. 308,407).—See X. Rendering organic bodies soluble (B.P. 289,002).—See XII. Preserving ether (U.S.P. 1,697,320).—See XX.

IV.—DYESTUFFS.

New nitro-dyes. P. P. KARPUKHIN (Ukraine Chem. J., 1928, 3, [Tech.], 233—235).—2:4-Dinitrochlorobenzene-6-sulphonic acid, prepared by the successive sulphonation and nitration of *p*-chloronitrobenzene, yields with *m*-phenylenediamine a condensation product which dyes animal fibres a golden-brown. Condensation with 2:4-diaminodiphenylamine yields a reddish-brown dye. R. TRUSZKOWSKI.

2:4-Diaminodiphenylamine as an ursol dye. P. P. KARPUKHIN (Ukraine Chem. J., 1928, 3, [Tech.], 229—231).—The preparation of 2:4-diaminodiphenylamine and its application to the dyeing of furs are described. R. TRUSZKOWSKI.

PATENTS.

Primary disazo dyes derived from diamino-carbazoles and arylimino-compounds. H. CLINGSTEIN and H. W. GRIMMEL, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,697,107, 1.1.29. Appl., 28.11.27. Ger., 24.3.26).—3:7-Diaminocarbazole is tetrazotised and coupled in substance or on the fibre with hydroxynaphthacarbazole (from 1:7-aminonaphthol) to give deep black shades (cf. B.P. 290,733; B., 1928, 518).

C. HOLLINS.

Production of vat dyes. G. KALISCHER and R. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,697,361, 1.1.29. Appl., 1.10.25. Ger., 12.11.24).—See F.P. 604,450; B., 1927, 212.

Manufacture of vat dyes of the anthraquinone series. W. MIEG, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,706,933, 26.3.29. Appl., 22.3.27. Ger., 24.3.26).—See B.P. 291,878; B., 1928, 598.

Vat indigoid dyes from dihalogenoalkylisatins. C. KRAUSS, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,698,233, 8.1.29. Appl., 25.10.26. Ger., 25.11.25).—See B.P. 271,906; B., 1928, 849.

Preparation of [yellow] azo dyes [for acetate silk]. E. FISCHER and C. E. MÜLLER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,706,484, 26.3.29. Appl., 21.4.27. Ger., 3.5.26).—See B.P. 270,352; B., 1928, 8.

Manufacture of *o*-hydroxyazo-dyes. L. LASKA and F. WEBER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,706,927, 26.3.29. Appl., 26.9.27. Ger., 5.10.26).—See B.P. 302,770; B., 1929, 165.

Production of metallic compounds of [azo] dyes. H. KRZIKALLA and W. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,693,448, 27.11.28. Appl., 1.4.27. Ger., 15.6.26).—See B.P. 272,908; B., 1928, 849.

Polyazo dyes derived from tetrazotised halogenated 4:4'-diaminodiphenylmethanes. R. KRECH and E. KEINER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,697,122, 1.1.29. Appl., 4.12.25. Ger., 16.12.24).—See B.P. 244,782; B., 1926, 816.

Manufacture of indigoid dyes. B. MAYER and J. WÜRGLER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,707,918, 2.4.29. Appl., 6.3.26. Switz., 18.3.25).—See B.P. 249,489; B., 1927, 360.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Reddening of unbleached sulphite celluloses. B. RASSOW and G. BRANDAU (Papier-Fabr., 1929, 27,

181—187, 202—210, 217—225).—The red coloration which is shown by unbleached but not by bleached sulphite cellulose is due to the formation of an intermediate product from a substance which is present in the cellulose in a colourless form, and can be oxidised first to a red and then to a white material. It occurs in both easily and difficultly bleachable cellulose, and is generally less intense the greater is the degree of decomposition of the material. It is independent of the moisture content and of the quantity of iron or other metallic compounds present, and is not caused by the free fat or rosin, since the red-coloured substance is not removed by prolonged benzol-alcohol extraction, neither is it due to the cellulose itself since bleached cellulose does not show the effect. In the view of the authors it is attributed to the fact that, during cooking, the lignin, besides being sulphonated, undergoes a chemical transformation, and the so-formed (? *iso*-)lignosulphonic acid forms an absorption compound with the carbohydrate, which is changed by oxidation into a red substance. This additive compound is concentrated in the cellulose during the first stages of cooking and later goes into solution as a result of hydrolysis. The ash content of the material rises as this concentration increases and falls with increasing hydrolysis. The extent of the hydrolysis depends on the p_H of the cooking liquor, the temperature, and the duration of cooking, and the reaction is accelerated by increasing the sulphurous acid content of the liquor. Both the amount of the additive product formed and the extent of reddening are proportional to the lignin content of the cellulose, but the latter effect is not due to the lignosulphonic acid alone, since neither the aqueous extract of the cellulose nor the lignosulphonic acid precipitated from it is coloured. Parallel to the reddening runs a luminous violet fluorescence which is changed to dark violet by oxidation. Both phenomena are sensitive to alkaline treatment and to oxidation; the fluorescent groups disappear on oxidation like the reddening. Whilst the latter is due to the formation of an additive compound, the fluorescence is due to lignosulphonic acid as such. The aqueous extract is as fluorescent as is the cellulose before the extraction. The fluorescence may be taken as a measure of the reddening to be expected, but not as a measure of the relative degrees of decomposition of two samples of cellulose, since the fluorescence of the cellulose is completely destroyed if the latter is treated with warm water, and is diminished in intensity by exposure of the material, so that otherwise identical materials exposed for different times would give different values. The tendency to reddening of cellulose is dependent on its degree of decomposition, the composition of the cooking liquor, the treatment with water, and on the duration of the storage, and can be removed by oxidation or by treatment of the cellulose with dilute mineral acids, hot water, or pyridine. B. P. RIDGE.

Qualitative test for degraded artificial silk. O. S. RHODES (J. Text. Inst., 1929, 20, 55—56 T).—Test samples, from which starch and other foreign materials have been removed, are treated for 1 min. with a boiling Nessler solution of reduced sodium hydroxide concentration, rinsed in warm 1% potassium

iodide solution, and washed in cold water. Overbleached artificial silk, or material containing excess of oxycellulose, shows up much darker than the normal material—whether viscose or cuprammonium silk. The test also serves to distinguish between viscose and cuprammonium silks. Cuprammonium silk remains unstained, viscose becomes light grey, and undesulphurised viscose turns orange and then orange-brown. It is further useful in cases of barring on dyed viscose, since its effects suggest that differences in dyeing properties between cop and cop, or even throughout the same cop, are due to differences in oxycellulose content, and also for the detection of oxycellulose on cotton, when it is as sensitive as the silver thiosulphate test and gives less coloration in the places where oxycellulose is absent.

B. P. RIDGE.

Determination of α -cellulose. E. SCHMIDT (Papier-Fabr., 1929, 27, 249—251).—Variations in the temperature of the mercerising sodium hydroxide solution (17.5%) between 5° and 30° have a negligible effect on the percentage of α -cellulose found; on the other hand, the temperature of the 8% solution used in washing the extracted cellulose has a considerable effect. The lower the temperature of this solution, the lower is the percentage of α -cellulose found, and washing, *e.g.*, with a solution at 5° may give results of the order 8% lower than those obtained with liquid at 30° when conditions are otherwise identical. If the temperature of the washing solution is controlled at, say, 20°, but that of the mercerising liquid in contact with the cellulose is lower, loss of α -cellulose will occur, since the temperature of the mixture during the washing will be less than 20°. Hence the temperatures of all solutions should be controlled. The time taken in washing is also of importance even when conditions of temperature are standardised; the longer the time the greater is the loss of α -cellulose. Prolonging this time from 10 to 60 min. results in a loss of about 2%.

B. P. RIDGE.

Decomposition wood. WAKSMAN and STEVENS.—See II. **Starch.** WREDE.—See XVIII. **Cellulose for smokeless powder.** OLSEN.—See XXII.

PATENTS.

Preparation of artificial staple fibre for spinning.

I. G. FARBENIND. A.-G., Assees of M. F. THOMA (B.P. 282,776, 23.12.27. U.S., 27.12.26).—Artificial staple fibre is soaked in a moderately warm solution containing ingredients for softening, *e.g.*, soap or glycerin, for improving the lustre, *e.g.*, turpentine oil, for increasing the hygroscopicity, *e.g.*, magnesium chloride, turpentine oil, for improving the feel, *e.g.*, oil soap containing a trace of sulphuric acid, and for leaving the fibre in a crinkled condition, *e.g.*, soda.

F. R. ENNOS.

Acetylation of cellulose. Production of cellulose esters. RUTH-ALDO Co., INC., Assees. of H. L. BARTHELEMY (B.P. 282,791 and 282,793, 28.12.27. Fr., 28.12.26).—Cellulose is esterified (A) in a vessel which rotates about an axis which is not symmetrical with respect to the surrounding surfaces in planes perpendicular to itself, in order to ensure complete mixing, (B) by means of acetic anhydride in the presence of a catalyst consisting of sulphuric acid and sufficient

oxidising agent, *e.g.*, manganic sulphate, chromic acid, to yield not more than 0.3% of active oxygen calculated on the weight of cellulose treated.

F. R. ENNOS.

Manufacture of cellulose acetate. J. W. BULMER (U.S.P. 1,692,622, 20.11.28. Appl., 15.2.28. U.K., 2.1.28).—See B.P. 298,819; B., 1929, 126.

Singeing of textile goods. W. OSTHOFF (B.P. 309,320, 23.4.28).

[Trough for hardening-liquid in] **manufacture of artificial silk.** J. P. BEMBERG A.-G. (B.P. 284,618, 5.1.28. Ger., 1.2.27).

Fastening devices for spinning centrifuges used in production of artificial silk. J. P. BEMBERG A.-G. (B.P. 302,646, 24.8.28. Ger., 19.12.27).

Caustic alkali (U.S.P. 1,702,589 and B.P. 309,237). **Wood-pulp liquors** (U.S.P. 1,702,586).—See VII. **Active carbon** (U.S.P. 1,701,272).—See XI. **Coating of fabric, paper, etc.** (B.P. 308,700).—See XII. **Wool-washing water** (B.P. 308,634).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of pure silk hose. H. D. MUDFORD (J. Soc. Dyers & Col., 1929, 45, 107—109).—Natural silk may be dyed with practically all classes of dyes. Basic colours are mainly used for the production of shades of exceptional brightness; acid dyes also give very bright colours and possess excellent fastness to light. The direct cotton colours serve to produce shades of good all-round fastness; those direct dyes the fastness to washing of which can be improved by after-treatment with formaldehyde, or by diazotisation and development, are of particular value. Certain mordant colours used with a chromium chloride mordant and, especially, vat colours and sulphur colours are employed when outstanding fastness is essential.

L. G. LAWRIE.

Effects of after-treatments on the degree of aggregation, location, shade, and fastness properties of insoluble azo colours on the fibre. P. BEAN, JUN., and F. M. ROWE (J. Soc. Dyers & Col., 1929, 45, 67—77; cf. Rowe, B., 1926, 741).—The state of aggregation and location of the azoic colour particles within the fibre are influenced largely by the nature and duration of the hot after-treatments to which the dyed material is subjected. Dry heat does not produce aggregation, although only a trace of moisture appears to be necessary. The changes taking place within the fibre were followed by means of a microscopical examination. A dyed but untreated fibre appeared uniformly coloured, whilst after a mild treatment the minute particles of pigment became visible. Progressively severe treatments first increase the size of the particles, which later migrate to a free surface. More severe treatments produce swelling of the fibre, and so facilitate migration, but the direction of migration depends on the nature of the fibre. In solid fibres the particles always migrate outwards, but with hollow fibres they migrate towards the lumen as well as outwards. Growth and migration of the particles continue until in some instances they leave the fibre. It was found that the same phenomena take place to a limited extent after many years, under normal

atmospheric conditions of temperature and moisture. The aggregation and migration of the particles cause a change in shade of the dyed fibres, and also alter their fastness to light, rubbing, and bleeding. The fastness to rubbing and bleeding depends on the aggregation and size of the particles at the surface of the fibres, whilst the fastness to light increases with increase in the size of the particles remaining in the fibre. It is concluded that, in order to give maximum fastness to light of the dyed material together with minimum decrease in fastness to rubbing, the after-treatment of the azoic colours with hot soap solution should be such as will remove loose surface pigment and produce the first stage of aggregation within the cell wall; after-treatment should limit subsequent growth of the pigment particles and, as far as possible, their migration.

L. G. LAWRIE.

Dyestuffs fastness and perspiration. B. A. McSWINEY (J. Text. Inst., 1929, 20, 65—66 p).—Perspiration was collected from various individuals and its p_H and ammonia content were determined. It consisted of a turbid, colourless fluid with varying amounts of epithelium debris, and had p_H 5.1—6.77. The ammonia-nitrogen content was found to vary with the individual, 3.86 and 6.99 mg./100 c.c. being found in two cases. On keeping, the urea content of the liquid diminishes, with a corresponding increase in ammonia and rise of p_H . The production of ammonia is accelerated by rise of temperature, and may also be activated by the presence of bacteria.

B. P. RIDGE.

Comparison of some chemical and physical properties of cotton and viscose silk. A. J. HALL (J. Soc. Dyers & Col., 1929, 54, 98—104).—The action of caustic soda is greater on viscose silk than on cotton, the viscose, however, showing an increased affinity for certain direct dyes only, whilst the cotton shows an affinity for all direct dyes. The natural moisture content of viscose is unaffected by mercerisation, whilst that of cotton is increased. The deleterious action of caustic soda on the handle, lustre, and tensile strength of viscose silk decreases with rise of temperature. The suitability of direct dyes for dyeing irregular grades of viscose silk evenly may be determined by dyeing untreated and alkali-treated viscose silk together, when suitable dyes give practically the same depth of colour on both materials.

L. G. LAWRIE.

Action of light in the dyeing and finishing of cotton and viscose silk material. F. SCHOLEFIELD (J. Soc. Dyers & Col., 1929, 45, 82).—A lecture (cf. Scholefield and Patel, B., 1928, 811).

Test for degraded artificial silk. RHODES.—See V. **Coloured cellulose acetate solutions.** NEWBY.—See XIII.

PATENTS.

Bleaching of natural and artificial fibres. E. C. R. MARKS. From CHEM. FABR. STOCKHAUSEN & CO. (B.P. 307,251, 24.5.28).—The textile materials are pre-treated with solutions of the sulphonated oils or fats described in B.P. 293,480 and 293,719 (B., 1929, 273), or the sulphonated products are added to the bleaching liquors, since they are very stable to chlorine and peroxides; bleaching is thereby accelerated. A. J. HALL.

Manufacture of piece-dyed tissues. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 306,876, 25.10.27).—In the dyeing of yarns and fabrics containing effect threads consisting of esterified cellulose, staining of the latter by dyes is largely diminished by adding to the dyebath condensation products of aromatic phenols or derivatives thereof with sulphuric acid and formaldehyde, or sulphurised aromatic phenols, or amorphous aromatic sulphonic acids, or similar synthetic products having the power to precipitate glue from its acid solutions (cf. B.P. 211,108, 230,872, and 251,294; B., 1925, 605; 1927, 675). A. J. HALL.

Treatment of animal or vegetable textile materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,913, 24.8.27).—The wetting out of textile materials is much assisted by addition to the aqueous liquors of an aliphatic or aromatic monohydric alcohol (the former containing more than two carbon atoms in the molecule), or of a mixture of such alcohol with methyl or ethyl alcohol in the absence of soaps and similar wetting-out agents or phenols. Such monohydric alcohols are equally applicable in acid, neutral, or alkaline liquors, and include benzyl alcohol and a wholly or partially hydrogenated aromatic monohydric alcohol.

A. J. HALL.

Dyeing of cellulose derivatives [esters and ethers]. H. EICHWEDE, E. FISCHER, and C. E. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,692,492—3, 20.11.28. Appl., 23.12.25. Ger., 9.1.25).—See B.P. 245,790; B., 1927, 438.

Treating [delustring] artificial silk. H. A. GARDNER (B.P. 290,263, 2.1.28. U.S., 13.5.27).—See U.S.P. 1,692,372; B., 1929, 169.

Water-soluble products (B.P. 307,776).—See XII. **Fireproofing products** (B.P. 273,268).—See XVI.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Preparation of potassium nitrate. A. L. MEHRING, W. H. ROSS, and A. R. MERZ (Ind. Eng. Chem., 1929, 21, 379—382).—The possibility of preparing potassium nitrate by treating potassium chloride with nitric acid was investigated. Treatment with the equivalent of 20% acid and evaporation to dryness, and treatment at reduced pressure or with addition of water so that no nitrosyl chloride was formed, followed by crystallisation, both proved unsatisfactory. A better result was obtained by digestion with 40—50% acid under a reflux condenser. The evolved nitrosyl chloride was absorbed in concentrated sulphuric acid, which was afterwards treated with steam. When a 12% mixture of nitrogen peroxide with air is passed over potassium chloride solution a solution is recovered containing 20% of potassium nitrate in 45% nitric acid. This solution was then digested with solid potassium chloride. The gases evolved were substantially chlorine and nitrosyl chloride, and upwards of 90% of the potash was crystallised out as nitrate on cooling. C. IRWIN.

Determination and control of acidity in the crystallisation of ammonium sulphate. J. A. CRANSTON and (Miss) J. STOCKDALE (J. Roy. Tech.

Coll. Glasgow, 1929, 2, [1], 36—39).—In saturated ammonium sulphate solutions small percentages of sulphuric acid cannot be readily determined by titration using the usual indicators, as the acid does not appear to be dissociated; conductivity measurements are useless for the same reason. A rapid method for determining the free acidity of such solutions based on the amount of sulphur dioxide liberated by treating the solution with saturated sodium sulphite solution is described. The liberated gas is swept by means of a current of air into a conductivity vessel, and the change of conductivity is used to operate a relay controlling the valve through which sulphuric acid is admitted to the crystallisation apparatus. A. R. POWELL.

Preparation and properties of the ammonium phosphates. W. H. ROSS, A. R. MERZ, and K. D. JACOB (Ind. Eng. Chem., 1929, 21, 286—289).—The ammonium meta- and pyro-phosphates are described. The three ammonium orthophosphates are of increasing stability in the acid direction. The two alkaline salts lose ammonia more rapidly in solution than in the solid state. The diammonium salt is the most soluble and the triammonium salt the least, its solubility in ammonia of d 0.90 being very slight. The mono-salt is of about the same solubility as ammonium sulphate at 65°. Its preparation in an analogous manner is rendered difficult by the presence of iron and alumina in commercial phosphoric acid. The crystals grow more rapidly than those of the sulphate and tend to clog. A discontinuous method is accordingly used, and the whole mass is dried in a rotary kiln at below 120°. The diammonium salt offers advantages as a fertiliser, and processes are being developed in which it is prepared by precipitating the triammonium phosphate in a closed vessel and gently heating the latter with recovery of the ammonia. Another method involves the decomposition of phosphate rock by acid ammonium sulphate and addition of ammonia. C. IRWIN.

Absorption of oxygen by alkaline tannates. E. P. FAGER and A. H. REYNOLDS (Ind. Eng. Chem., 1929, 21, 357—359).—Determinations have been made of the amounts of oxygen absorbed by alkaline solutions of commercial tannates with the view of comparing their relative suitability as rust-retarding agents for addition to boiler-feed waters. The absorptive power of some of these solutions is almost as great as that of alkaline pyrogallol. Free tannins and those to which an equivalent of alkali has been added show no absorptive power. Increase in alkalinity is accompanied by an increase in absorptive power until a certain limit is reached. Preliminary experiments made under a pressure of 25 lb./in.² at 130° appear to show that under these conditions the absorptive power of the solutions is increased. H. INGLESON.

Pure carbon monoxide for experimental purposes. J. G. THOMPSON (Ind. Eng. Chem., 1929, 21, 389—390).—An apparatus is described in which carbon monoxide of 99.9% purity can be prepared. No impurities which could be identified were found in the gas. A regulated stream of formic acid is dropped into 85% phosphoric acid heated at 170°. The water vapour and unchanged formic acid are condensed from the gas

stream, which is then washed with dilute sodium hydroxide solution. After compressing into steel cylinders traces of iron carbonyl and carbon dioxide were found in the gas. The combined output of two generators (10-litre Pyrex flasks) is 200 cub. ft. in 7 hrs. with an efficient utilisation of formic acid of 92%. The phosphoric acid baths can be used indefinitely.

H. INGLESON.

Toxicity of sulphur. R. C. WILLIAMS and H. C. YOUNG (Ind. Eng. Chem., 1929, 21, 359—362).—Commercial sulphur on being triturated with water gives a clear filtrate which contains sulphurous, sulphuric, pentathionic, and probably tetrathionic acids; sulphide and trithionate ions are not present. Tests made with these acids on spores of *Sclerotinia cinerea* show that by far the most toxic are the polythionic acids. Although the acidity of the sulphur filtrates is due mainly to sulphuric acid, yet a solution of this acid of equivalent concentration is not toxic. If commercial sulphur is freed from its polythionic acids by suitable treatment with ammonia or nitric acid, it loses its toxicity, but regains it on exposure to air and water. Artificially oxidised sulphurs are also very effective provided the oxidising agent does not destroy the polythionic acids. Since the polythionate ion is not toxic in neutral or alkaline solution, the toxicity of lime-sulphur sprays is probably dependent on the development of acidity, to liberate the polythionic acid, by weathering of the sulphur. F. R. ENNOS.

PATENTS

Manufacture of sulphuric acid and oleum by the contact process. METALLGES. A.-G. (B.P. 293,450, 13.6.28. Ger., 7.7.27).—Gas mixtures containing sulphur dioxide, after preliminary purification by treatment with sulphuric acid of increasing concentration, e.g., by successive treatment with acid of d 1.38, 1.53, and 1.61, respectively, are passed through electrical precipitation apparatus and thence into the contact chambers. The sulphur trioxide formed in these chambers is returned to the electrical precipitation apparatus, where it purifies and dries the gases passing through and, by combination with the moisture in the gases, is itself converted into sulphuric acid or oleum. L. A. COLES.

Manufacture of nitric and sulphuric acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,232, 16.1.28).—A mixture of ammonia and sulphur dioxide, such as is obtained by the decomposition of ammonium sulphite solutions produced by scrubbing industrial gases, is passed together with oxygen over a catalyst, e.g., platinum gauze or iron powder containing tellurium and bismuth, at 600—700° to oxidise the ammonia to oxides of nitrogen. The gas mixture is then passed through a tower in countercurrent to a stream of nitrosyl-sulphuric acid whereby the sulphur dioxide is oxidised to sulphur trioxide and oxides of nitrogen are liberated by the heat of the gases. The nitrous gases pass to a cooling and oxidising chamber, where the nitrogen peroxide formed is absorbed in sulphuric acid. The liquid is heated to expel the nitrous gases, which are absorbed in dilute nitric acid and finally in alkali solutions. If the original gas mixture contains hydrogen sulphide

and cyanogen compounds, these are oxidised prior to the above treatment by passing the gas mixture at 250–350° together with air over finely-divided iron, nickel, or copper salts deposited on fireclay.

A. R. POWELL.

Recuperation of hydrochloric acid gas from aqueous hydrochloric acid. Recovery of hydrochloric acid. INTERNAT. SUGAR & ALCOHOL Co., LTD. FROM SOC. INDUS. DE LA CELLULOSE (B.P. 307,985–6, 21.12.27).—(A) Equal weights of about 30% aqueous acid and calcium chloride (30–40 pts. of water to 100 pts. of calcium chloride) are gradually heated in a retort to 165°; after the dry hydrogen chloride has been led off the heating is discontinued and the mass subjected to reduced pressure in the same vessel to concentrate the calcium chloride for use again. A continuous evolution of gas is maintained by using two or more retorts having their gaseous acid outlets connected independently to a common conduit, and the acid distillation period is longer than the reduced pressure distillation. (B) Liquid containing hydrochloric acid is heated with sulphuric acid in stages of respectively increased temperatures, *e.g.*, 108°, 135°, and 150°, in a series of vessels or in a partitioned vessel. The sulphuric acid is initially of 70% strength, and the residual acid is concentrated for use again by reducing the pressure on the hot acid.

W. G. CAREY.

Manufacture of arsenic acid. F. ULLMANN and G. TREWENDT, ASSRS. to J. MICHAEL & Co. (U.S.P. 1,699,823, 22.1.29. Appl., 26.2.24, Ger., 6.3.23).—In the oxidation of arsenious acid with chlorate in boiling water, a little hydrochloric acid is used as catalyst.

C. HOLLINS.

Promotion of chemical reactions [between gases]. S. RUBEN (U.S.P. 1,702,950, 19.2.29. Appl., 15.2.26).—Ammonia is synthesised by electrolysis lithium hydroxide (or other alkali hydroxide) in a container, the walls of which form the anode and a hollow central iron tube, oxidised by heating at 1000° in steam, and coated externally with dielectric material, the cathode. Nitrogen under pressure is delivered through the hollow cathode, and, under a potential greater than the decomposition voltage of the solution and than the ionisation or spark potential of the resultant gaseous mixture of hydrogen and nitrogen, combination occurs intermittently with an arc discharge of frequency about 2000 per sec. The ammonia escapes by a gas outlet pipe and, if desired, may be catalytically oxidised to nitric acid by the oxygen liberated at the anode. The container surface may be coated with mercury to obtain increased catalytic effect.

R. BRIGHTMAN.

Oxidation of ammonia. C. W. DAVIS, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,706,055, 19.3.29. Appl., 18.2.28).—Ammonia is catalytically oxidised to nitric oxide by passing it and a gas containing oxygen through a platinum alloy containing 2–50% Rh.

H. ROYAL-DAWSON.

Production of caustic alkali etc. L. BRADLEY and E. P. MCKEEFE, ASSRS. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,702,589, 19.2.29. Appl., 5.11.21. Renewed 5.5.27).—Residual liquors from the digestion of wood with sodium sulphite are evaporated, *e.g.*, to $d\ 1\cdot2-1\cdot3$,

dried in a rotary furnace, and, preferably after preliminary calcination to remove volatile organic matter, the residue is converted at least partly into sodium carbide in an electric furnace. The calcined residue is treated with steam (air and carbon dioxide being excluded) to give acetylene and dry or aqueous sodium hydroxide.

R. BRIGHTMAN.

Purification of caustic alkali solutions. A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 309,237, 11.1.28).—Waste alkali lyes containing organic material obtained, *e.g.*, in the manufacture of viscose silk, are treated with oxygen or gas mixtures containing it at above 100° and at 1 atm. or above. Alkali carbonates formed during the treatment are converted subsequently into the hydroxides by treatment with lime, or the two processes may be combined by adding strontium or barium oxide or hydroxide to the solution prior to oxidation.

L. A. COLES.

Treatment of residual liquors [from production of wood pulp]. L. BRADLEY and E. P. MCKEEFE, ASSRS. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,702,586, 19.2.29. Appl., 2.3.25. Can., 9.2.22. Renewed 11.5.27).—Residual liquors from the digestion of wood with sodium sulphite, or sodium sulphite and hydroxide, are evaporated to dryness, and the residue is calcined to eliminate at least some sulphur compounds and afford a product containing relatively large amounts of sodium carbonate. The calcined product is leached from insoluble carbon etc. and converted into sodium sulphite by treatment with sulphur dioxide, part of which may be supplied from gases liberated in the calcination. Alternatively, part of the sodium carbonate may be causticised.

R. BRIGHTMAN.

Manufacture of potassium manganate. SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 296,074, 20.3.28. Fr., 26.8.27. Addn. to B.P. 292,991; B., 1929, 244).—In the prior process caustic lye of about 50% concentration, instead of 70%, may be used, provided that the temperature is above 200°, preferably 210–220°.

W. G. CAREY.

Manufacture of dicalcium phosphate and ammonium sulphate. J. F. C. HAGENS, L. ROSENSTEIN, and W. HIRSCHKIND, ASSRS. to BARRETT Co. (U.S.P. 1,699,393, 15.1.29. Appl., 12.5.27).—Gypsum is treated with phosphoric acid (80%) and ammonia until 2 mols. of ammonia have reacted for each mol. of gypsum and phosphoric acid. Alternatively, the phosphoric acid may be first converted into diammonium hydrogen phosphate and then treated with gypsum and water.

R. BRIGHTMAN.

Manufacture of alkali xanthates. W. HIRSCHKIND, ASSR. to GREAT WESTERN ELECTRO CHEM. Co. (U.S.P. 1,704,249, 5.3.29. Appl., 7.7.26).—Carbon disulphide is introduced into a mixture of alcohol and caustic alkali at below 35°.

F. G. CLARKE.

Production of alkaline-earth titanates [for use as pigments etc.]. E. C. R. MARKS. FROM TITANIUM, LTD. (B.P. 308,516, 29.5.28).—Titanium hydroxide from which sulphates have been removed by treatment with normal acetates, *e.g.*, zinc, sodium, or ammonium acetate, followed by washing, is heated at 750–880° with an alkaline-earth (barium) carbonate in the presence

of an accelerator, *e.g.*, hydrochloric acid or barium chloride. L. A. COLES.

Manufacture of nitrates. J. I. BRONN, G. FISCHER, and CONCORDIA-BERGBAU A.-G. (B.P. 308,028, 31.1.28).—Nitric acid vapours or oxides of nitrogen mixed with 10–15 times their weight of steam are introduced near the bottom of a reaction tower filled with solid sodium or potassium chloride, or with packing through which the chloride solution percolates; the resulting nitrate solution, passing through the false bottom of the reaction chamber, is siphoned off and concentrated, and the free acid neutralised. W. G. CAREY.

Manufacture of anhydrous metallic chlorides. W. F. DOWNS (U.S.P. 1,699,229, 15.1.29. Appl., 12.1.25).—Alumina (85 pts.) is mixed with sufficient molasses solution (or other suitable organic solution) to yield 15 pts. of residual carbon on evaporation and charring, and the residual mass thus obtained is charged into a revolving reaction chamber at, *e.g.*, 700–800° in a current of hot chlorine. R. BRIGHTMAN.

Manufacture of aluminium chloride. C. G. MINER (U.S.P. 1,698,238, 8.1.29. Appl., 19.5.27).—Aluminous material is mixed with 5–10% excess of coke and treated with hydrogen chloride above 1500°, preferably at 1700–2000°. The alumina and hydrogen chloride may be recovered from oil sludge produced in cracking processes by running the sludge to coke residue and treating with just sufficient steam at 200–500° to form dry hydrogen chloride. R. BRIGHTMAN.

Manufacture of cyanides. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 307,778, 13.12.27).—Carbon monoxide is passed over a cyanate at 700° or above until the issuing gas is free from carbon dioxide. The cyanate used may be pure or mixed with an indifferent diluent or filler, *e.g.*, magnesia, graphite. W. G. CAREY.

Iron oxides and their manufacture. C. L. DELACHAUX (U.S.P. 1,702,250, 19.2.29. Appl., 16.2.26. Fr., 29.7.25).—Forge and mill scale is heated in a rotating or agitated furnace at about 900° in presence of gases containing 9% O to give a product, containing less than 31.03% FeO, which is used for aluminothermal processes. R. BRIGHTMAN.

Preparation of titanium oxide. R. H. MONK (U.S.P. 1,695,341, 18.12.28. Appl., 7.7.26).—Titanium concentrates are heated with 1.5 pts. of sodium hydrogen sulphate and 2.2 pts. of 66% sulphuric acid at 120° for 3 hrs. and then on a tray at 260° to give a solid cake. This is lixiviated, and the iron in solution reduced. On warming at 75–95°, titanium oxide free from iron is precipitated. C. HOLLINS.

Exploitation of gases containing nitrogen, hydrogen, and carbon dioxide. W. N. HENDERSON, Assr. to SOLVAY PROCESS CO. (U.S.P. 1,698,722, 15.1.29. Appl., 21.3.25).—Producer gas is mixed with nitrogen, and after catalysis with steam to convert the carbon monoxide into hydrogen and carbon dioxide, the nitrogen:hydrogen ratio is adjusted to approx. 3:1, and the gases are compressed, *e.g.*, to 100 atm., and passed through a water scrubber to remove carbon dioxide. The stripped gas, after purification, is con-

verted into ammonia by catalysis, the ammonia being subsequently liquefied and unconverted gases returned for further catalysis. Water from the scrubber is discharged against a Pelton wheel or other device to utilise energy, and the pressure is partly released, the gas mixture (80–85% CO₂, with some nitrogen and hydrogen) being fed to a tower and treated with concentrated sodium carbonate solution. Sodium bicarbonate is collected and discharged, and the residual gas containing, *e.g.*, 15% CO₂ and 83% (H + N), is returned to the system before the compressor. R. BRIGHTMAN.

Purification of gases. J. G. DELY, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,698,718, 15.1.29. Appl., 17.4.26).—Hydrogen and nitrogen mixtures containing oxides of carbon as impurities are compressed to, *e.g.*, 100 atm. and scrubbed with water, water from the scrubber being charged against a Pelton wheel or other means for conserving energy. The scrubbed gas is treated successively in one or in successive towers with cuprammonium solution at the ordinary and at a refrigerated temperature. This solution is regenerated by heating to expel oxides of carbon, and is used again. R. BRIGHTMAN.

Binder for laminated mica products. T. OKURI (U.S.P. 1,707,277, 2.4.29. Appl., 9.11.26. Japan, 5.8.26).—See B.P. 272,384; B., 1927, 607.

Separation of [didymium etc. from] cerium. F. SOMMER, Assr. to DEUTS. GASGLÜCHLICHT-AUER GES. M.B.H. (U.S.P. 1,707,450, 2.4.29. Appl., 21.4.25. Ger., 18.1.24).—See F.P. 594,783; B., 1926, 237.

Heat storage (B.P. 309,244).—See I. Cement and sulphuric acid (B.P. 309,298).—See IX. Copper leaching solutions (U.S.P. 1,703,435).—See X.

VIII.—GLASS; CERAMICS.

Effect of soda, barium, and zinc on the elasticity and thermal expansion coefficients of glass. L. D. FETTEROLF and C. W. PARMELEE (J. Amer. Ceram. Soc., 1929, 12, 193–216).—The effect of progressively replacing the soda by barium oxide and zinc oxide in a 60% SiO₂–40% Na₂O glass has been studied. Five glasses were made up for each series, containing 6–30% BaO and ZnO, respectively. Enough batch was melted to cast 8 test-bars which were ground to size and annealed. Difficulty was experienced in fining the small amounts of batch. The elasticity was measured at room temperature by the beam deflexion method. The expansion was measured up to the softening point (480–600°) by the interferometer method. The results show that replacement of sodium by barium or zinc increases the softening temperature and elasticity. The effect of the zinc is the more pronounced in each case. The generally accepted figure for the elasticity factor for zinc oxide is 15 (Winkelmann and Schott), but this appears to be much too low and the value 80 is now proposed. The equal effectiveness of sodium and barium oxides in contributing to elasticity is also disputed. The values 35 and 55, respectively, are claimed to be more in harmony with the results than the common factor of 100. The proposed values may be too low, but are proportionately so. When replacing soda, both barium and zinc oxides reduce the expansion,

and the latter to the much greater extent. The expansion factors of English and Turner (B., 1921, 510 A) for sodium and barium oxides are sufficiently correct for most purposes, whilst the Winkelmann and Schott factor of 1.8 for zinc oxide is certainly to be preferred to that of 0.21 of English and Turner. J. A. SUGDEN.

Fracture systems of glass. F. W. PRESTON (J. Soc. Glass Tech., 1929, 13, 3—15).

China clay in rubber. DRAKELEY and POLLETT.—See XIV.

PATENT.

Ceramic ware (B.P. 282,104).—See X.

IX.—BUILDING MATERIALS.

Fuel economy in the rotary kiln burning Portland cement clinker. R. D. PIKE (Ind. Eng. Chem., 1929, 21, 307—310; cf. B., 1929, 324).—Equations give the thermal efficiency of the calcining zone in terms of dimensions, temperatures, and heat-transfer coefficients, and the length for maximum efficiency. On these as basis a series of curves are constructed showing the influence on efficiency of dimensions of kiln, rate of output, excess air present, etc. Amongst the conclusions are that for kilns of a given length the fuel consumption per unit output is less with smaller diameters, and that the loss through overloading is less with longer kilns. It is considered that, using a pre-heater and no excess air, a thermal efficiency of 50—60% is possible. C. IRWIN.

Effect of fuel and air mixture in burning cement clinker. H. H. BLAISE (Rock Products, 1929, 32, 38—39).—When burning raw batches of identical composition it was noted that some kilns produced clinker very hard and black on the exterior, and although well burned throughout, the centre was brown, the whole grinding to cement having a yellow tinge. Other kilns produced clinker black throughout grinding to a blue-grey colour. From examination it was suspected that the former resulted from reducing conditions in the kiln with subsequent external oxidation. In burning trials using large excess of air without regard to economical heat balance, the same type of clinker was produced and found to be due to stratification of the gas streams in the burner pipe which caused the fuel gas to be projected to the fusion zone, the clinker therefore being formed in a reducing atmosphere. Later, as it progressed down the kiln, a coat of oxidised clinker resulted. In further trials complete oxidation of clinker was obtained by thorough mixing of fuel and air supply in the burner, using theoretical proportions for complete combustion. C. A. KING.

Electrical investigation of the setting and hardening of rapid-hardening cements. Y. SHIMIZU (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 1213—1225).—In the setting of aluminous cements two discontinuities are observed in the electrical conductivity, corresponding to the hydration of calcium aluminate and calcium silicate or aluminosilicate, respectively, whilst with Portland cements only the second discontinuity occurs. It is concluded that Portland cement clinker does not contain calcium aluminate. The

resistivity during setting and hardening is greater for aluminous cements than for Portland cements, and increases more rapidly with time. The more rapidly a cement sets, the less is the effect of temperature on the setting time. C. J. SMITHELLS.

Cause of failure of brickwork. E. DONATH and H. LEOPOLD (Chem.-Ztg., 1929, 53, 277—279).—Failure of mortar and plaster has been traced in the case investigated to the use of (a) water containing relatively large amounts of chlorides and nitrates; (b) lime derived from a limestone containing iron and aluminium oxides and silica; (c) too high a proportion of sand. J. S. CARTER.

Photographic action from wood. STRACHAN.—See XXI.

PATENTS.

Joint manufacture of sulphuric acid and cement. K. GORDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,298, 31.3.28).—The quantity of air admitted to the cement kiln is slightly in excess of that required to burn the fuel, and the air required for the oxidation of the sulphur dioxide is added after the gases have passed through a Glover tower. L. A. COLES.

Manufacture of a wall product [plaster]. C. E. HITE, Assr. to UNIVERSAL GYPSUM & LIME Co. (U.S.P. 1,702,730, 19.2.29. Appl., 21.4.24).—A plastic mass obtained by boiling starch with sulphuric acid, neutralising with lime, and adding finely-ground burnt gypsum, is used for facing a cellular cast product in the form of sheets or slabs. A. R. POWELL.

Cellular heat- and sound-insulating material. G. M. THOMSON (B.P. 309,256, 31.1.28).—Cellular gypsum etc. in the form of spheres ($\frac{1}{8}$ — $\frac{1}{2}$ in. diam.) which are dried and dipped in waterproofing material, e.g., casein glue, is claimed. L. A. COLES.

Artificial marble, stone, etc. BINDPHAST PRODUCTS, LTD., and E. WOOD (B.P. 309,242, 16.1.28).—Mixtures comprising calcined magnesite, magnesium chloride, zinc oxide, iron oxide, manganese oxide, chromium oxide, or aniline dyes or pigments, together with filling material are claimed. L. A. COLES.

Wood impregnation. A. M. HOWARD, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,703,239, 26.2.29. Appl., 30.11.26).—The penetration power of oily wood-preserving agents is increased by agitation with about 5% of dilute mineral acid, e.g., 30% sulphuric acid, and separating the acid solution from the oily material. R. BRIGHTMAN.

Treating wood. F. LAUTER, Assr. to LIGNEL CORP. (U.S.P. 1,697,248, 1.1.29. Appl., 28.10.27).—Wood is impregnated with an aldehyde and subjected simultaneously to heat and pressure, e.g., up to 2000 lb./in.² and 115°, after superficial treatment with phenolic substances if desired. R. BRIGHTMAN.

Manufacture of emulsions of bitumen or pitch for road-making etc. T. M. HICKMAN (B.P. 308,389, 7.1.28).—The emulsifying solution is prepared by adding liquid or solid rosin, distilled Yorkshire grease, sulphonated fatty oils, or mixtures of these, containing 5% of acetanilide, to a solution of casein in dilute alkali hydroxide, alkali carbonate, or ammonia solution;

calcium hydroxide, salicylic acid, and sodium sulphate may also be added. L. A. COLES.

Floor covering. HERCULES POWDER Co., and J. H. RILE (B.P. 308,524, 11.6.28. U.S., 3.4.28).—A mixture of soluble nitrocellulose, 30–70% of an alkyl (ethyl) abietate alone or mixed with other plasticisers, *e.g.*, tolyl phosphate, and fillers, pigments, etc. is claimed. L. A. COLES.

Manufacture of cement by the wet process. T. RIGBY (U.S.P. 1,706,747—1,706,750, 26.3.29. Appl., [A] 8.12.22, [B] 8.7.26, [C] 5.11.26, [D] 9.3.27. U.K., [A] 19.12.21, [B] 24.7.25, [C] 13.11.25, [D] 28.7.24).—See B.P. 190,056, 261,814, 270,356, 243,410; B., 1923, 778 A; 1927, 76, 464; 1926, 129.

[Apparatus for] manufacture of road-making materials. J. WHITE (B.P. 309,264, 6.2.28).

Foams (B.P. 308,029).—See I. **Fireproofing products** (B.P. 273,268).—See XVI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

A.I.B. sinter plant [for iron ores] at Messrs. Guest, Keen, & Nettlefolds, Ltd., Cardiff works. W. E. SIMONS (Iron and Steel Inst., May, 1929. Advance copy. 14 pp.).—The sintering plant consists essentially of steel circular pans 8 ft. 6 in. diam. and 13 in. deep, holding about 1.5 tons of finished sinter. The pans, carried on a truck, are filled from a hopper and ignited by means of oil burners carried on a travelling wagon. The charge of fine ore (up to $\frac{1}{4}$ in.) and coke (7%) is mixed with water to a content of about 15% to assist in maintaining porosity, and when filled into the pan is dusted over with anthracite to promote combustion. All materials should be free from lime owing to the low m.p. of lime-silica compounds which causes a dense clinker. After completion in 1–1½ hrs., the sintering pan is overturned and any fines are returned to the process; the returned material amounts to 15–20%.

C. A. KING.

First report on blast-furnace plant and practice. COMMITTEE No. 2, OF IRON AND STEEL INST. (Iron and Steel Inst., May, 1929. Advance copy. 27 pp.).—In a general survey of conditions of blast-furnace practice, strong coke of open texture is favoured, though as the ash content increases beyond 10% increased brittleness becomes an adverse factor. The practical point of view on the reactivity of coke to carbon dioxide is that any virtue a coke may have by reason of its combustion reactivity at the tuyère level is counterbalanced by the loss of carbon by such reactivity in the upper levels and is also affected by carbon deposition. The considerable losses of ore in the form of flue dust are best reduced by sintering the dust and returning it to the furnace—now standard practice in Germany and America. Uniformly graded coke assists regularity of operation. Whilst even charging at the furnace top is of importance, the tendency in modern practice is to keep the ore and coke out of direct contact in the upper zone as much as possible in order to restrict direct reduction by solid carbon to a minimum. To this end the size of coke is increased. Design of furnace lines has developed in

the direction of larger hearth diameters, smaller bosh, and narrower shafts, the hearth diameter controlling the other dimensions within fairly narrow limits. The interior construction of hot stoves is still generally of firebrick, though recently a heat- and oxidation-resisting casting has been used in the bottom section, and in one case short spiral-shaped bricks have replaced the usual checker flues. The advantages of dry blast are admitted, but the high cost has militated against general adoption; silica gel is now being tried at a Scottish works. Casting in sand beds is rapidly becoming obsolete, and where the metal is not taken direct to mixers machine-casting is becoming standard practice. The enrichment of the blast with oxygen awaits further work both on the successful utilisation of oxygen in the furnace and the possibility of producing large quantities of relatively pure oxygen at low cost. C. A. KING.

Twenty months' results of dry-blast operation. E. H. LEWIS (Iron and Steel Inst., May, 1929. Advance copy. 4 pp.).—Analysis of monthly figures from the Wishaw furnaces taken over twenty months shows that Evans and Bailey's equation is substantially correct except that for furnaces which are not water-cooled the radiation constant of 500 should be modified to about 350, with slight variations between winter and summer. The uniformity of results with irons containing 1.26–3.74% Si points to the accuracy of the silicon coefficient of 0.12. Using a blast temperature of 788° the extra carbon required per ton of iron for each 1% Si is 184 lb., and it has actually been found to be much easier to make lower-silicon iron with dry blast, the optimum dryness being 1–1½ grains/cub. ft. Working advantages of dry blast include the absence of "slips" and the possibility of using a larger proportion of raw coal. C. A. KING.

Conditions determining the action of refining agents in the open-hearth furnace. E. KILLING (Stahl u. Eisen, 1929, 49, 527–531).—The relative values of four different iron ores as decarburising agents in the open-hearth steel process have been determined. The results obtained confirm the theoretical conclusion that ores containing a proportion of ferrous oxide are more efficient than those containing only ferric oxide. A high silica content increases the cost of working in spite of the lower cost of silicious ore, owing to the necessity of using larger amounts of lime for slagging and to the greater loss of iron in the slag.

A. R. POWELL.

New method for production of sound steel. (SIR) C. PARSONS and H. M. DUNCAN (Iron and Steel Inst., May, 1929. Advance copy. 12 pp.).—The method is based on the principle that the freezing of the ingot should proceed from the bottom upwards, the isothermals being substantially parallel to the bottom of the ingot. The mould consists of a strong sheet-steel casing slightly conical in form, lined internally with shaped firebrick. The longitudinal dimensions are much greater than the vertical, allowing a heavy bottom chill to be caused by a cast-iron chill block 6 ft. deep. An arched cover of refractory material is provided through which are holes permitting the heating of the surface of the metal by means of oil or gas burners and the

escape of the combustion gases. A circular ingot (diam. 70 in., height 45 in.) weighing $20\frac{1}{2}$ tons cast in such a mould showed no trace of V-segregate or axial weakness, and the main segregate was noticeable only in the discard at the top. A rapid rate of pouring is advisable to minimise the oxidation and non-metallic inclusions likely to occur in the use of so large a chilling base.

C. A. KING.

Transformation of austenite into martensite in a 0.8% carbon steel. D. LEWIS (Iron and Steel Inst., May, 1929. Advance copy. 11 pp.).—The steel may be quenched in a molten salt bath at 232° to comparatively stable austenite, which is transformed into martensite on cooling, the final hardness (Brinell 650) being unaffected by the rate of cooling. The austenite is stable at 232° for at least 5 min.; hence, forming operations at this temperature with subsequent hardening on air-cooling should be possible. It decomposes rapidly below 232° , the product being a hard martensite. From 232° to 316° the decomposition is slow, and the product has a martensitic appearance, but the needles are large and the hardness is low; in the former case crystallisation probably takes place from a labile state, and in the latter from a metastable state. By means of a two-stage hardening of this type quenching can be conducted under controlled conditions.

M. E. NOTTAGE.

Stepped lowering of the A1 transformation in steels. T. MURAKAMI (Tech. Rep. Tôhoku Imp. Univ., 1929, 8, [2], 119—127).—As the rate of cooling increases, the A1 transformation in eutectoidal steels takes place in two steps, the temperature of the higher step being lower than that of the normal A1 point. With more rapid cooling, the first step is entirely suppressed, and at the second step the austenite is converted into martensite. The stepped transformation is explained on the basis of the velocity of crystallisation curves for the respective phases, ferrite, cementite, and martensite, deduced from Tammann's theory.

A. R. POWELL.

Microstructure of rapidly cooled steel. J. M. ROBERTSON (Iron and Steel Inst., May, 1929. Advance copy. 29 pp.).—To test the truth of the conception that martensite, troostite, sorbite, and pearlite are alternative products of, and do not represent consecutive stages in, the decomposition of austenite, the temperature of decomposition determining which product is formed, small steel specimens were cooled in molten metal at various temperatures between 600° and 220° . Increase in the rate of cooling slightly lowers, interrupts, and suppresses the temperature of the Ar1 point, which is initiated by the formation of cementite, and progressively lowers the temperature of the Ar2 point, which is initiated by the allotropic change, and this difference in behaviour determines the relation between the two. When the transition takes place at the normal temperature the product of the decomposition of austenite consists of α -iron and cementite; as the temperature of the transition is lowered, more carbon is retained in solution in the α -iron and less separates as cementite. The crystallographic form of the product depends on whether the decomposition is initiated by the allotropic change or by the formation of cementite. Two

series of structures are produced by increasing the rate of cooling. All structures formed at the Ar2 point may be tempered, the resulting structure depending on the original structure, the time, and temperature of tempering. The crystallographic structure formed by cooling at different rates cannot be obtained by tempering other structures, but the same ultimate constitution may be reached by either method. By reheating the α -solid solution to different temperatures all variations in constitution between solid solution and ferrite-cementite aggregate may be obtained.

M. E. NOTTAGE.

Constitutional diagrams for cast irons and quenched steels. A. L. NORBURY (Iron and Steel Inst., May, 1929. Advance copy. 26 pp.).—Constitutional diagrams have been constructed for iron-carbon alloys containing 0, 2, and 4% Si respectively, showing both the normal and supercooled systems for the austenite-graphite and the austenite-cementite eutectics. When graphite nuclei are present in the molten metal, grey irons have been assumed to crystallise on cooling according to the normal graphite-austenite system; they contain coarse flakes of graphite which tends to be associated with pearlite. When graphite nuclei are absent supercooling occurs and the graphite separates in a very finely-divided form associated usually with ferrite. This difference in structure caused by the difference in the size of the graphite particles is explained on the assumption that the γ -solid solution surrounding the graphite flakes retains carbon in solid solution according to the lower solid solubility of graphite, while the remainder of the γ -phase retains carbon in solid solution according to the higher solid solubility of cementite. Hence the coarser and fewer are the graphite flakes the greater is the amount of carbon retained in solid solution. In white irons it is assumed that the nuclei cause the precipitation of a small amount of the graphite eutectic, which then causes the precipitation of the white iron eutectic at the highest temperature possible and its rapid coalescence. A constitutional diagram for quenched steels based on the work of earlier investigators is constructed.

A. R. POWELL.

Tempering changes in steels. R. HAY and R. HIGGINS (J. Roy. Tech. Coll. Glasgow, 1929, 2, [1], 73—80).—The changes in hardness during tempering of martensitic steels can be expressed by an equation of the form $y_t = y_0 e^{-\alpha t}$, where y_t is the hardness at t° , y_0 the initial hardness in the martensitic state, and α is a constant. A classification of iron alloys, based on the thermodynamic equation $\Delta T = RT^2(x_1 - x_2)/L$ is suggested, where x_1 is the concentration of the solute in the high-temperature phase, and x_2 that in the low-temperature phase, R , T , and L having the usual meaning. The various alloying elements may be thus divided into two groups: those which lower the $\delta \rightarrow \gamma$ transformation point and raise the $\gamma \rightarrow \alpha$ point, and those which have the converse effect. To the first group belong silicon, chromium, vanadium, molybdenum, and tungsten, and to the second group carbon, nickel, copper, and aluminium. In the iron-carbon system this theory indicates that the eutectoid point is merely the intersection of the line representing the change in the solubility

of iron carbide in γ -iron with the line representing the depression of the A3 point by carbon. A. R. POWELL.

Influence of heat-treatment on properties of tungsten steel. F. PÖLZGUTER and W. ZIELER (Stahl u. Eisen, 1929, 49, 521—526).—For most tungsten steels there is a critical range, depending on the composition, within which annealing causes considerable reduction in the hardness and magnetic properties due to the rapidity with which the carbide WC is precipitated within this temperature range. Thus, with a steel containing 5.7% W and 0.75% C, maximum hardness (670) and coercivity is obtained by annealing at 750° or at 1200°, and minimum hardness (450) and coercivity at 950—1000°. The addition of a small proportion of chromium tends to restrain the precipitation of tungsten carbide, and therefore reduces the deleterious effect of annealing within the critical range. A. R. POWELL.

Influence of pickling operations on the properties of steel. H. SURRON (Iron and Steel Inst., May, 1929. Advance copy. 24 pp.).—From experiments on mild, medium- and high-carbon steels, pickling in sulphuric or other acid solution embrittles the metal, the maximum effect being shown when the particular steel is in its hardest condition. Immersion in boiling water for 30 min., or allowing the pickled steel to remain at atmospheric temperature for several days, substantially removes the brittleness. Medium- and high-carbon steels tempered within certain ranges of temperature, e.g., oil-hardened from 850° and tempered at 100—300°, were subject to cracking when pickled, if they had been deformed after the heat-treatment. Mild steel was unaffected. Addition of certain organic substances, e.g., glue, starch, to the pickling bath reduced the rate of acid attack, but did not prevent brittleness; pyridine and quinoline diminished embrittlement considerably. Electrolytic pickling in neutral or alkaline solutions could be effected without appreciable embrittlement of the steel, though in this respect anodic pickling was less injurious than cathodic. The results confirm the deleterious effect of hydrogen generated at the surface of steels during the process of pickling. C. A. KING.

Oxidation of iron and steel at high temperatures. L. B. PFEL (Iron and Steel Inst., May, 1929. Advance copy. 47 pp.).—High-temperature oxidation of iron and steel produces a smooth compact scale consisting normally of three distinct layers. With few exceptions non-ferrous impurities present in the iron are concentrated in the innermost layer of scale, and foreign substances placed on the surface of the iron are not forced outwardly, but remain and become enveloped by the scale. This disproves the commonly accepted view that the exterior of the scale is formed first and is outwardly displaced by fresh formations of scale underneath. Experiments indicated that the middle portion was the first to form, and a hypothesis is demonstrated by which iron and oxygen counter-diffuse through the scale deposit. C. A. KING.

Effect of cold-working on corrosion of iron and steel. H. ENDŌ (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 1265—1278).—Steels of various carbon contents were subjected to different kinds of mechanical stress, and the loss in weight in 1% sulphuric acid solution was deter-

mined. In tension, torsion, and compression the loss in weight was proportional to the amount of deformation, the effect being most marked for steel containing 0.9% C. C. J. SMITHELLS.

Corrosion of steel by acid solutions. H. ENDŌ (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 1245—1263).—The effect of additions of up to 30% of cobalt, nickel, chromium, manganese, molybdenum, vanadium, tungsten, and copper, and of smaller amounts of carbon, phosphorus, silicon, sulphur, and titanium to steels, on their corrosion in nitric, hydrochloric, and sulphuric acids was determined. Corrosion by all acids increases with increase in carbon and sulphur, but is reduced by silicon. Corrosion by nitric acid decreases with increase in cobalt, chromium, molybdenum, titanium, and phosphorus. Addition of up to 1% of copper, tungsten, and manganese reduces corrosion by nitric acid, but larger amounts cause increased corrosion. Corrosion is increased in hydrochloric and sulphuric acids by increase in chromium, manganese, and phosphorus, and also by small additions of cobalt, nickel, molybdenum, and vanadium, but is decreased by the latter group when present in excess of 3%.

C. J. SMITHELLS.

Erosion of guns. R. H. GREAVES, H. H. ABRAM, and S. H. REES (Iron and Steel Inst., May, 1929. Advance copy. 55 pp.).—Evidence is adduced to show that the erosion of large guns is almost entirely due to the melting and sweeping away of metal from the inner surface by the rapid stream of highly heated propellant gas. The quantity of metal removed depends on the nature of the explosive, the calibre and ballistics of the gun, and the m.p., sp. heat, latent heat of fusion, and thermal conductivity of the metal. Surface cracking due to heating and cooling through the critical range is not an important factor in determining the amount of erosion. Armeo iron owing to its higher m.p. has a smaller erodibility than any steel, but is not suitable for gun construction, so that the only practical method of reducing erosion appears to be the use of a cooler propellant. The frictional wear by the projectile and the effect of various propellants on erosion are discussed, and many photomicrographs of sections of the inside skin of worn guns are reproduced.

A. R. POWELL.

Corrosion—apparent relation of protective film to microstructure. C. VAN BRUNT (Ind. Eng. Chem., 1929, 21, 352).—It was found that a pump handling a sodium xanthate solution of cellulose was protected from corrosion by a film of ferrous sulphide with the exception of the parts of the cast iron side-plates near the peripheries of the gears. In this case the relative motion no doubt rubbed off the film and corrosion occurred. Photomicrographs show that the corrosion occurred along the grain boundaries, and it is suggested that the segregations on the boundaries form lines of weakness in the film. C. IRWIN.

Electrochemical corrosion of painted or lacquered steel. U. R. EVANS (Amer. Electrochem. Soc., May, 1929. Advance copy. 22 pp.).—Sheet steel was rubbed with fine emery paper, degreased with benzene, dried, cut into strips, and coated with varnish or paint.

When dry, a drop of sodium chloride solution (usually 0.5*N*) was placed on the coated surface. After being kept for a specified time (usually 24 hrs.), the specimen was washed and then rubbed in various ways to test the adherence of the coating in the region which had been covered by the drop and its immediate neighbourhood. Various varnishes and paints were tested in this way, using both coatings which were apparently complete and others which had been scratched in the region covered by the drop. The cause of peeling is electrochemical action, exposed points of metal under the solution dissolving anodically, while sodium hydroxide is formed at points around the edge of the drop which act cathodically. The loosening or alteration in properties of the coating is due to the alkali so formed, for salts which do not give caustic alkali have comparatively little action. In cases when this "alkaline peeling" occurs from unscratched specimens it may be concluded that there is some slight porosity or imperfection in the coating. A very slight exposure of the underlying metal is sufficient to initiate peeling over a much greater area since the alkali tends to creep between the metal and the coating. With the various varnish media examined the tendency to peeling varies enormously, and the order of resistance to peeling cannot be predicted from the order of resistance to rusting. Resinous substances, however, such as copal, definitely reduce the tendency to peel. The introduction of a pigment into the medium may retard or prevent peeling by obstructing diffusion processes in the film, by strengthening the film against distension or abrasion, or, in the case of pigments having an oxidising action, by rendering the underlying metal passive. It is suggested that the main function of red lead in paints for protecting iron is to keep the metal in a passive condition. Various pigments were examined, as well as aluminium and "gold" paints. Paints containing flaky particles are relatively impermeable owing to the long, sinuous diffusion paths between the particles, but if the particles are metallic electrochemical attack of the underlying metal may become considerable.

H. J. T. ELLINGHAM.

Alkaline electrolytic iron. S. J. LLOYD (Amer. Electrochem. Soc., May, 1929. Advance copy. 7 pp.).—Estelle's process (cf. U.S.P. 1,275,161; B., 1918, 659 A) for electrodepositing iron from a suspension of ferric oxide in hot concentrated caustic soda solution has been examined on a laboratory scale. Experiments were made using artificial ferric hydroxide and various iron ores under various conditions. By electrolysis with sheet-iron electrodes of a well agitated suspension of limonite or pyrites residues, ground to 200-mesh, in 35% caustic soda solution at 90° with 4 amp./dm.², a good deposit of iron was obtained on the revolving cathode. The current efficiency was high and the estimated power consumption is 3000–3300 kw.-hrs. per ton of iron. The regeneration of the electrolyte is discussed.

H. J. T. ELLINGHAM.

Properties of special brasses containing nickel and manganese. A. LE THOMAS (Metallbörse, 1928, 18, 1996–1997; Chem. Zentr., 1928, ii, 2055).—Physical and mechanical properties, and the (low)

corrodibility of brasses containing (a) Cu 53.20, Zn 38.82, Ni 4.46, Mn 2.95, Fe 0.56%, and (b) Cu 52.8, Zn 37.44, Ni 4.74, Mn 3.13, Fe 1.05, Sn 0.9% were studied.

A. A. ELDRIDGE.

Effect of annealing on the solidus temperature of alloys. J. H. ANDREW and H. M'NEIL (J. Roy. Tech. Coll. Glasgow, 1929, 2, [1], 64–72).—From theoretical considerations based on thermodynamics and the phase rule it is shown that a perfectly homogenous solid solution should melt at a constant temperature and not over a temperature range. This has been tested by annealing a tin-copper alloy with 7.89% Sn for a prolonged period at temperatures just below and just above the usually accepted solidus point and redetermining the m.p. After 30 hrs. at 900° the melting range is reduced from 140° to about 25°, and the completion of melting lies at a temperature of 20–25° above that at which freezing begins. The question of grain refinement in an alloy which undergoes a transformation on heating is discussed, and it is suggested that the refinement is due to diffusion occurring during heating and not during cooling, and that grain growth takes place only when heating is continued after diffusion has ceased.

A. R. POWELL.

Corrosion of aluminium. J. CALVET (Compt. rend., 1929, 188, 1111–1114).—In spite of the absence of a period of passivity (A., 1928, 302), aluminium (99.94%) produced by the Hoopes process was less rapidly attacked by *N*-hydrochloric, -sulphuric, or -nitric acid, or 5% phosphoric acid than "pure" (99.75%) or ordinary (99.18%) aluminium. Nitric acid had the greatest effect. Solutions of citric and tartaric acids (5%) showed no appreciable effect after 61 days, whilst after 51 days 5% acetic acid had only a slight action on all three metals. The Hoopes aluminium was almost unaffected by exposure to 3% brine for 56 days at 24°, whilst the other samples were badly corroded.

J. GRANT.

Rapid determination of the tin content of tinplate and tinplate residues. J. HÆGE (Chem.-Ztg., 1929, 53, 287).—The sample is warmed with 1:1 hydrochloric acid until all the tin is dissolved, the residual iron is removed and washed, and the solution treated with zinc or aluminium to precipitate tin sponge, which is redissolved in hydrochloric acid in a flask fitted with a Bunsen valve. The stannous chloride solution so formed is cooled and titrated with iodine.

A. R. POWELL.

Spectrographic analyses of Irish ring-money and of a metallic alloy found in commercial calcium carbide. A. G. G. LEONARD and P. F. WHELAN (Sci. Proc. Roy. Dubl. Soc., 1928, 19, 55–62).—Spectrographic study of specimens of "ring money" discovered in Ireland and dating about 500 B.C. indicated that the core of the ring was tin in a very pure form with traces of copper and lead, the tin being of greater purity than "pure" tin electrodes used for comparison. The gold sheath showed several silver lines and a few of copper. A small metallic specimen (*d* 9–10) associated occasionally with calcium carbide contained 11.3% Si, 22% Ti, 66.2% Fe, besides traces of their oxides.

C. A. KING.

Relation between stress and strain in the impact test. R. YAMADA (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 1179—1211).—Stress-strain diagrams for Charpy impact test-pieces were obtained by the method of Körber and Storp, and the results compared with static tests. The maximum stress is greater in the impact test, and both this and the absorbed energy increase with the velocity of deformation. The effect of the dimensions of the test-piece are examined. At low temperatures steels appear to be more brittle in impact than in static tests.

C. J. SMITHELLS.

Corrosion phenomena. BERL.—See I. **Rust preventers.** JAKEMAN.—See XII. **Photographic action from metals.** STRACHAN.—See XXI.

PATENTS.

Blast furnace. Apparatus for protection of blast-furnace jackets. J. P. DOVEL (U.S.P. 1,703,517 and 1,703,520, 26.2.29. Appl., [A] 30.11.25, [B] 17.9.23).—(A) The stack of a blast furnace comprises a main shell with suitable lining and a contracted upper shell lined with metal-wear plates. (B) The lining of the main shell comprises agglomerated refractory material in which are embedded water-cooled projections.

A. R. POWELL.

Heat-resisting structure etc. N. V. HYBINETTE (U.S.P. 1,704,087, 5.3.29. Appl., 5.5.22).—Chromium-nickel-iron alloys may be produced by smelting together 10—15% of chromium, 30—35% of nickel, and 50—60% of iron in an open-hearth reverberatory furnace, the carbon content being regulated to about 0.50—0.75%.

M. E. NOTTAGE.

Heat-enduring metal. N. V. HYBINETTE (U.S.P. 1,698,098, 8.1.29. Appl., 12.7.4).—Iron alloys containing 28—40% (30%) Cr and 18—40% (35%) Ni are heat-resistant in presence of alkali at high temperatures. Addition of 1—10% of cobalt or 0.5—5% of copper softens the alloy without lessening the endurance, but 0.1% of copper reduces the strength of the alloy. 1% of silicon, 0.5% of manganese, and 0.5% of aluminium may be present.

R. BRIGHTMAN.

Alloy. F. A. FAHRENWALD (U.S.P. 1,704,733, 12.3.29. Appl., 19.12.24).—A heat-resisting alloy casting contains 3—20% Al and 97—80% Cr.

H. ROYAL-DAWSON.

Manufacture of briquettes and production of ceramic ware. A. UHLMANN, Assee. of A. UHLMANN KOMMANDITGES. (B.P. 282,104, 12.12.27. Ger., 11.12.26).—Pulverulent matter may be briquetted, and ceramic ware produced, without the use of binding substances by subjecting the material, which may previously be moistened with acidulated water, to the action of a magnetic field of great density and of high frequency, by which means the particles are fritted together.

M. E. NOTTAGE.

Separation of tin from oxidic stanniferous and plumbiferous materials. HÜTTENWERKE TROTHA A.-G., and W. WETTER (B.P. 290,628, 3.5.28. Ger., 18.5.27).—The material is stirred continuously with fused sodium hydroxide and a small quantity of carbon at about 300—450° until all the stannic oxide has been attacked, but not the lead oxide. The melt is treated first with sufficient water to extract only the free alkali,

the solution being concentrated for re-use, and the residue is extracted with sufficient water to yield a tin solution of the desired concentration.

L. A. COLES.

Concentration of ores by froth flotation. R. E. SAYER, Assr. to METALS RECOVERY Co. (U.S.P. 1,697,614, 1.1.29. Appl., 11.5.28).—Organic bivalent selenium compounds, e.g., selenobenzamide, ethyl selenide, seleno-carbanilide, diphenylselenocarbamide, phenylseleno-urethane, selenophenol, selenocresol, are used as mineral-collecting agents in froth flotation. Utah copper ore pulp treated with 0.4 lb. of selenobenzamide, 0.25 lb. of pine oil, and 4 lb. of sodium hydroxide per ton of ore gives 79.8% recovery of copper, the concentrates containing 14.0% and the tailings 0.21% Cu.

R. BRIGHTMAN.

Purification of [copper] leaching solutions and recovery of acids or dissolving agents. A. E. WHEELER (U.S.P. 1,703,435, 26.2.29. Appl., 17.11.23).—The nearly neutral liquors obtained by leaching copper ores with sulphuric acid are purified by agitation with further quantities of coarse ore until the ferric oxide and alumina are precipitated. The residue is separated and the precipitate removed from the undissolved ore by classification.

A. R. POWELL.

Brazing alloys. R. T. GILLETTE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,706,154, 19.3.29. Appl., 22.6.27).—An alloy of copper containing about 8% Sn and an appreciable amount of silver is claimed; it has m.p. about 925°.

H. ROYAL-DAWSON.

Anti-fatigue alloy. Non-corrodible structure. Plastic, light aluminium alloy. V. N. HYBINETTE, Assr. to HYBINETTE PATENTS CORP. (U.S.P. 1,704,251—3, 5.3.29. Appl., 11.7.27).—(A) An alloy containing at least 95% Al, 2% Ni, < 1% of metals of the chromium group, and < 0.5% each of copper, magnesium, and silicon is quenched at 482° and aged at 92—204°. (B) An aluminium alloy containing < 2% Ni, < 1% of metals of the chromium group, and 0.2—0.5% each of copper and magnesium is similarly quenched and aged. (C) Aluminium is alloyed with < 2.5% of heavy metals, < 0.5% Cu, and 0.5% Mg. It is then quenched, partly aged, and subjected to alternate periods of ageing and working.

F. G. CLARKE.

[Non-corrodible] aluminium alloys. VICKERS-ARMSTRONGS, LTD., W. B. GOUDIELOCK, and W. MACHIN (B.P. 308,491, 13.4.28).—An aluminium alloy containing 0.05—4% (1.5%) Mg, 0.05—6% (3—4%) Sb, and 0.05—4% (1.5%) Ni is claimed.

C. A. KING.

Alloy and its manufacture. J. V. O. PALM, Assr. to CLEVELAND GRAPHITE BRONZE Co. (U.S.P. 1,704,208, 5.3.29. Appl., 10.12.25).—The alloy contains 82% Pb, 9% Sn, 4% Sb, 3% Zn, 1.5% Cu, and 0.5% Al.

F. G. CLARKE.

Pickling of metals. IMPERIAL CHEM. INDUSTRIES, LTD., and E. CHAPMAN (B.P. 398,407, 27.1.28).—Inhibitors for addition to acid pickling baths to prevent attack of the metal comprise sulphouated or sulphonated and alkylated mineral oil fractions described in B.P. 274,611 and 279,990 (B., 1927, 841; 1928, 50), sulphonated formaldehyde-naphthalene or aldehyde-phenol condensation products, polynuclear alkylated aromatic or hydroaromatic sulphonic acids, and products obtained

from ligninsulphonic acid by moderated oxidation or by treatment with hot alkalis or with aqueous ammonia under pressure. L. A. COLES.

Cleaning and/or annealing of metal. N. V. HYBINETTE (U.S.P. 1,704,086, 5.3.29. Appl., 29.7.21).—Nickel, nickel alloys, and metals containing chromium may be freed from coating of oxide by subjecting them to the action of a molten slag in which the oxide is soluble and which contains boric acid and other ingredients which render it elastic when hot, but brittle when cold. M. E. NOTTAGE.

Composite articles for safe or vault construction. J. G. DONALDSON, Assr. to GUARDIAN METALS CO. (U.S.P. 1,703,416—7, 26.2.29. Appl., [A] 17.6.21, [B] 3.7.26).—(A) A unit for the construction of vaults comprises a refractory core encased in metal. The core consists of one or more substances which are poor conductors of heat when cold, but become good conductors when hot. (B) The core is obtained by fusing a mixture of zirconia, zircon, and graphite, with or without iron or other metal which forms a hard carbide. A. R. POWELL.

Metal-plating non-metallic substances. H. C. FISHER, Assr. to RICHARDSON CO. (U.S.P. 1,704,125, 5.3.29. Appl., 12.4.24).—The substance is coated with a bituminous surface rendered tacky by means of a solvent, and is then brought into contact with a film of finely-divided metal supported on water by means of surface tension; the metal picked up by the tacky surface is then rendered coherent by electroplating. M. E. NOTTAGE.

Application of [rubber etc.] coatings to metal surfaces. R. M. WITCOMBE (B.P. 282,737, 7.12.27. Austral., 30.12.26).—A solution consisting of 3 oz. of rubber and 1 oz. of sulphur in 3 qts. of a solvent (e.g., benzol) is applied to the surface, the solvent allowed to evaporate, and the coated surface subjected to heated air at 107—132°. M. E. NOTTAGE.

Manufacture of metal sheets [by electrodeposition]. H. C. HARRISON (U.S.P. 1,704,247, 5.3.29. Appl., 4.12.23. Renewed, 8.9.25).—The metal is deposited on a horizontal, cylindrical surface, which is rotated by the metal itself as it is stripped therefrom. Between the anode and the cathode is a fixed, porous partition which is shaped to fit the latter. F. G. CLARKE.

Production of thin nickel flakes. E. BREUNING, Assr. to ELECTRIC STORAGE BATTERY CO. (U.S.P. 1,707,217, 2.4.29. Appl., 28.6.27. Ger., 10.7.26).—See B.P. 274,064; B., 1928, 412.

Production of alloys of lead with alkaline-earth metals. G. J. KROLL (U.S.P. 1,707,059, 26.3.29. Appl., 20.12.26. Ger., 29.12.25).—See G.P. 443,641; B., 1928, 20.

Apparatus for use in electrolytic deposition of metal [chromium] on metal tubes. J. STONE & CO., LTD., F. J. PIKE, and C. J. LYTH (B.P. 309,056, 30.11.27).

Magnetic materials (U.S.P. 1,703,287).—See XI.

XL—ELECTROTECHNICS.

Application of Talbot's law to photo-electric cells with a non-linear illumination-current charac-

teristic. G. H. CARRUTHERS and T. H. HARRISON (Phil. Mag., 1929, [vii], 7, 792—811). Talbot's law, fatigue, and non-linearity in photo-electric cells. W. S. STILES (*ibid.*, 812—820).

Setting of cements. SHIMIZU.—See IX. Electrolytic iron. LLOYD.—See X. Java citronella oil. O'DONOGHUE and others.—See XX.

PATENTS.

Heating liquids electrically. G. BAUM, Assr. to NIAGARA ELECTRO CHEM. CO., INC. (U.S.P. 1,685,210 and 1,685,266, 25.9.28. Appl., [A] 19.10.26, [B] 15.10.25. Austr., [A, B] 28.10.24).—Distillation, concentration, etc. of liquids may be effected electrically by placing in the liquid a porous, three-sided, open frame, the connecting cross-piece being above the level of the liquid. Liquid is absorbed and passes into the porous bridge through which an electric current is passed. The amount of liquid in the porous bridge decreases and the current strength and heating effect increase with the distance above the liquid level in the vessel, so that the distillation or vaporisation temperature is ultimately attained. R. BRIGHTMAN.

Electric furnace product [active carbon]. J. J. NAUGLE (U.S.P. 1,701,272, 5.2.29. Appl., 21.4.23).—The residues obtained by evaporation of the spent liquors derived from the alkali treatment of wood pulp are carbonised, and the product is heated in an electric furnace until the inorganic matter present sinters. The mass is broken up and carbon containing less than 3% of ash is obtained by elutriation or air-levigation. A. R. POWELL.

Manufacture of photo-electric cells. GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 309,224, 7.1.28. Addn. to B.P. 306,996; B., 1929, 362).—The red sensitivity of photo-electric cells, constructed in accordance with the prior patent, is made permanent by filling the cell with hydrogen in place of argon, the hydrogen being preferably introduced through a palladium tube after the cell is removed from the exhaust pump. The metal electrode on which the thin film is deposited consists of a metal other than silver or platinum, e.g., copper, preferably superficially oxidised. J. S. G. THOMAS.

Oxide cathode [for vacuum tubes]. K. M. VAN GESSEL (U.S.P. 1,699,639, 22.1.29. Appl., 24.12.25. Holl., 12.2.24).—A metal (tungsten) filament is oxidised superficially by heating at 700° in air, then inserted into the vacuum tube opposite an anode which is coated with a reducing alkaline-earth compound, e.g., barium azide. After evacuating the tube the electrodes are heated to 1200°, whereby the azide decomposes and the liberated barium volatilises on to the oxidised filament, thus reducing the oxide surface and leaving a tightly adherent coating of barium oxide. A. R. POWELL.

Production of magnetic materials [iron-nickel alloy]. J. W. ANDREWS, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,703,287, 26.2.29. Appl., 16.3.27. Renewed 7.7.28).—An alloy of 80% Ni and 20% Fe is melted in the presence of oxygen, "boiled" until thoroughly oxidised, cast, and rolled hot until it is cooled below the recrystallisation temperature. The

cold alloy is then crushed to a fine powder for use in magnet cores. A. R. POWELL.

Insulation and securing of coil windings of lacquered wire. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 308,508, 9.5.28).—The coil windings are impregnated with lacquers containing cellulose derivatives, plasticisers, and solvents which attack only slowly the original lacquer coating, colouring material, resins, etc. being added if desired.

L. A. COLES.

Furnace electrode. P. L. J. MIGUET, Assr. to Soc. ELECTROMÉTALL. DE MONTRICHER (U.S.P. 1,707,406, 2.4.29. Appl., 9.2.27. Fr., 21.12.26).—See B.P. 282,672; B., 1928, 716.

Porous body for diaphragm (B.P. 309,316).—See I. Sulphuric acid and oleum (B.P. 293,450). Reactions between gases (U.S.P. 1,702,950).—See VII. Briquettes (B.P. 282,104). Metal sheets (U.S.P. 1,704,247).—See X.

XII.—FATS; OILS; WAXES.

Classification of fats. II. W. HALDEN (Chem. Umschau, 1929, 36, 109—115; cf. B., 1928, 678).—Vegetable oils and fats are grouped according to the families to which the parent plants belong botanically; each family is assigned to one or other of the two broad classes: (a) producers of drying oils, or (b) producers of non-drying oils and fats, according to the chemical characters of the oils from the majority of its members. Castor oil occupies an anomalous position as an "intermediate group." E. LEWKOWITSCH.

Cacao butter. I. Determination of unsaturated fatty acids of expressed cacao butter. H. P. KAUFMANN (Z. angew. Chem., 1929, 42, 402—406).—A large number of cacao butters from various sources have been examined. The presence of 34—38% of oleic and 2—5% of linoleic acids (reckoned as glycerides) is demonstrated by the iodine and thiocyanogen values, which are 35—37 and 32—35, respectively. The use of the difference between these characteristics as an analytical criterion considerably reduces the possible limit of undetectable adulteration. E. LEWKOWITSCH.

Distinction between expressed and extracted cacao butter. AUFRECHT (Chem.-Ztg., 1929, 53, 318).—The addition of fuming hydrochloric acid to a solution of cacao butter in chloroform produces a green coloration if extracted fat (except doubly-refined) is present, which changes to brownish-yellow on warming with the addition of nitric acid (d 1.42); expressed cacao butter gives no coloration. Characteristic colour reactions are also obtained with concentrated sulphuric acid. E. LEWKOWITSCH.

Thiocyanogen value of fats. H. P. KAUFMANN [with A. BROCKE] (Arch. Pharm., 1929, 267, 229—232).—The earlier survey (cf. B., 1926, 758 etc.) has now been extended to fish oils. White medicinal cod-liver oil of iodine value 168 (determined bromometrically) has a thiocyanogen value of 105.2 (200% excess of thiocyanogen used in the determination, and 24 hrs. allowed for interaction). Other samples of cod-liver oil of various grades ranging from crude oil to medicinal quality showed iodine values of 149—167, and thiocyanogen values of 96.5—104. Corresponding

iodine and thiocyanogen values for some other oils are, respectively:—whale oil (filtered) 117.0, 82.5; sperm oil (filtered) 92.0, 88.0; seal oil 125.0, 107.0; sardine oil 178.0, 103.0; herring oil 127.0, 79.0. There is thus considerable variation in the difference between the iodine value and the thiocyanogen value. The relationship between these values and the nature of the unsaturated acids present is discussed.

W. A. SILVESTER.

Hardened-fat manufacture. Oil impurities as catalytic poisons. T. ANDREWS (Chem. Trade J., 1929, 84, 277—278, 302—303, 351—352, 369—370).—The effect of various oil impurities on the hydrogenation of a high-grade arachis oil in the presence of a nickel-diatomite catalyst was investigated. A standard homogeneous catalyst with good keeping qualities was obtained by wetting the freshly reduced catalyst with a minimum of pure oil, which is then hydrogenated to zero iodine value. Addition to the standard oil of 2% of the unsaponifiable matter, or of the stearine separated by freezing from a low-grade, crude cod-liver oil, caused a marked decrease in the rate of hydrogen absorption; a definite decrease in activity of the catalyst also followed the addition of 1% of blood or of 0.3% of lecithin (i.e., 0.02% of organic phosphorus), whilst hydrogen absorption was completely inhibited by 0.5% of allylthiocarbimide (cf. hardening of rape oils from imported seed). The effect of added isinglass (organic nitrogen) was small and seemed to be physical rather than chemical in nature. E. LEWKOWITSCH.

Lead soaps. M. RAGG (Farben-Ztg., 1929, 34, 1605—1609, 1661—1664).—Basic lead pigments form anisotropic lead soaps with fatty acids, and appear as spherulites or needles by polarised light. These soaps are difficultly soluble in cold oils and organic solvents. At moderate temperatures litharge dissolves in linseed oil by satisfying secondary valencies to produce glycerol-containing complexes that differ from true soaps in composition and form. As an intermediate stage in the dissolution of red lead in hot linseed oil lead peroxide is formed. This is reduced to the monoxide in the subsequent processes, and the intermediate peroxide formation is not apparent in stored paint or paint films. Anisotropic lead soap-glycerol complexes can be detected both analytically and microscopically in old paint films containing red lead, basic lead sulphate, etc. For the most part these lead soaps exist in the film in the isotropic sol condition, separating as anisotropic gels on the addition of a suitable solvent. The assumption of a mechanical strengthening of the film by the formation of lead soaps is therefore not corroborated. S. S. WOOLF.

Detection of rosin in soaps. N. SCHAPIRO (Z. anal. Chem., 1929, 77, 41).—The method depends on the fact that in acid media ferricyanides are reduced to ferrocyanides by rosin. An alcoholic solution of the soap is acidified with dilute hydrochloric acid, and a few drops of a dilute solution of potassium ferricyanide are added. The whole is heated to boiling and, after cooling, a drop of a dilute solution of ferric chloride is added. A blue coloration indicates the presence of rosin. J. S. CARTER.

Atmospheric oxidation of esters of β -elæostearic acid with monohydric alcohols. A. B. MILLER and K. L. ROHRBACH (Ind. Eng. Chem., 1929, 21, 338—342).—The propyl, isopropyl, butyl, and benzyl esters of β -elæostearic acid were prepared, and the physical and chemical changes occurring during their oxidation were observed by the method previously described (cf. B., 1928, 163). Oxidation data are tabulated and graphed. The course of oxidation is, in general, similar to that of the polyhydric alcohol esters, but the monohydric alcohol esters do not dry. Further differences are attributable to the shape and size of the alcohol group. Hydrolysis is one of these variables, and the present experiments are considered to furnish further support for the hypothesis that an important function of the alcohol group is the stability it imparts to the ester, thus influencing gel formation.

S. S. WOOLF.

Lanoline rust preventers. C. JAKEMAN (Dept. Sci. Ind. Res., Eng. Res. Spec. Rep. No. 12, 1929, 22 pp.).—Acid-free lanoline is superior to petroleum grease for the protection of metals liable to corrosion. A number of solvents for lanoline which would be satisfactory for the purpose are excluded on account of danger of inflammability or to health, e.g., benzene, trichloroethylene (Westrosol). Petrol, paraffin, turpentine, linseed, or castor oil, and tetrahydronaphthalene, when used as solvents, did not give satisfactory coatings. Solvent naphtha and "white spirit" proved most suitable, though the latter dries somewhat slowly. For application by brush or for dipping, solutions of lanoline in these solvents may be used or a mixture with 10–15% of solvent may be applied by means of rag. It is generally sufficient to clean the metal with a dry cloth, though for fine work, e.g., gauges or razor blades, extra cleaning of the work with petrol or naphtha may be advisable. If desired, the protective grease may be coloured with suitable oil-soluble aniline dyes.

C. A. KING.

Report of tests at the [olive oil] experiment station at Ghaba. M. ROUSSEAU (Bull. Mat. Grasses, 1929, 46—52).—Olive pulp treated in finishing presses alone furnished oils identical with those obtained by the use of two sets of presses, both as regards yield and quality: in the former case, however, with existing types of press it is necessary to refill the machine after a preliminary compression, and a modified press with a longer stroke is recommended for efficient working. Preliminary experiments indicate that little or no advantage is gained by chopping the olives prior to crushing. The Galimberti plant for cleaning and washing olives was found to be very satisfactory for the removal of leaves, adherent soil, etc.

E. LEWKOWITSCH.

Palm-kernel oil and palm oil. G. DE BELSUNCE (Bull. Mat. Grasses, 1929, 3—34).—A summary is given of work on the constitution of palm-kernel and palm oils, with especial reference to the investigations of Collin and Hilditch and of Boemer on the constituent fatty acids and glycerides and of Starkle on rancidification. It is suggested that Lewis' method (A., 1927, 39) for the separation of fatty acids might usefully be employed in order to assist in establishing the presence

or absence of lauric, myristic, stearic, and linoleic acids in palm oil, the constitution of which is still uncertain. The non-fatty constituents of these oils are considered: a study of the oxidation of carotin suffices to explain abnormalities observed in the bleaching and examination of palm oil, but the characteristic odour is not accounted for, the presence of iron being doubtful.

E. LEWKOWITSCH.

Drying oils. X. Fumes from oil kettles. D. S. CHAMBERLIN, E. R. THEIS, P. F. SCHLINGMAN, and J. S. LONG (Ind. Eng. Chem., 1929, 21, 333—338).—Linseed and fish oils were destructively distilled out of contact with air, the gases given off were analysed, and the characteristics of condensates collecting in successive parts of the absorbing trains were recorded. The results indicate that in varnish manufacture the oil layer in contact with the hot metal undergoes cracking (mainly at the double linkages), producing undesirable decomposition products. The fumes given off when linseed oil is heated consist largely of fine droplets of such products. These may be absorbed and retained by wetting them preferably with products of similar nature, and it is suggested that the fine droplets be caused to impinge on surfaces wet with linseed oil containing fatty acids, aldehydes, etc. Cooking losses are diminished by the introduction of a small percentage of driers during cooking, the course of the reactions proceeding being largely modified. The elimination of fumes by burning them is discussed, and it is shown that at temperatures above 450° the fumes coming from oil kettles may be completely burned by the oxygen normally admitted with them. The extent to which industrial processes are limited by inherent fundamental characteristics of the oils used is suggested. Minimum values for the critical temperatures of various drying oils were directly observed and their surface tensions were measured by means of a suitably-modified du Noüy tensimeter. The Ramsay-Shields modification of Eötvös' equation is applied to the results obtained.

S. S. WOOLF.

Effect of age on the apparent gain in weight of drying-oil films. P. E. MARLING (Ind. Eng. Chem., 1929, 21, 346—347).—The apparent gain in weight of various drying-oil films on glass plates was observed over a period of 6½ years, and the results are tabulated and graphed. The progressive alterations in the physical condition of the films are discussed. All were soft and tacky after 6½ years, oxidised linseed oil being the least, and tung, soya-bean, and fish oils being the most affected.

S. S. WOOLF.

Amounts of the antirachitic vitamin in different samples of cod-liver oil, milk, and butter. K. H. COWARD (Quart. J. Pharm., 1928, 1, 534—538).—Marked variations are reported.

F. C. HARPOLD.

Determination of fat in leather. ENNA.—See XV.

PATENTS.

Manufacture of water-soluble products from commercial fatty acids obtained from raw wool fat. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 307,776, 13.12.27).—Products soluble in alkaline or acidic aqueous solutions and suitable for use in dyeing

and in leather manufacture are obtained from commercial fatty acids prepared from raw wool fat by sulphonation with sulphuric acid monohydrate in the presence of phenol. S. S. WOOLF.

Rendering fatty and organic bodies soluble. R. VIDAL (B.P. 289,002, 22.12.27. Fr., 19.4.27).—Oleic or other fatty acid is treated with alkali hypochlorites or hypobromites and the product is decomposed with mineral acid. The modified fatty acid so formed is more reactive than the original fatty acid, and may be used in the production of water-soluble pastes in conjunction with many types of organic substances.

S. S. WOOLF.

Production of creams having curative properties. S. WEISSBEIN (B.P. 309,012, 2.1.28).—Fatty acids or fats, *e.g.*, stearic acid, are treated in the presence of colloid protectors, *e.g.*, glycerol and saponin, with a quantity of alkali insufficient for complete neutralisation or saponification, and during the treatment a concentrated solution of a curative salt, *e.g.*, "Oeynhausens salt," containing saponin is stirred in.

L. A. COLES.

Prevention of rancidity in vegetable oils when forming part of plastic compositions for coating fabric, paper, etc. G. E. SCHARFF, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 308,700, 23.11.27).—To such compositions consisting principally of nitrocellulose, camphor, vegetable oil, and pigments or fillers, the addition of about 2% of finely-ground magnesium oxide, calculated on the weight of the vegetable oil, is made.

E. HOLMES.

Stuffing of leather (B.P. 307,775).—See XV. **Keeping quality of margarine, butter, etc.** (B.P. 308,405).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Spreading rates and durability of low- and high-grade paints. ANON. (Amer. Paint & Varnish Manufs. Assoc., April, 1929, Circ. No. 347, 312—320).—The analysis of several low-grade paints demonstrates such disadvantages as high proportions of extender in the pigment, up to 35% of water in the vehicle, etc. Comparative spreading power tests indicate the superiority of high-grade paints over the apparently cheap ones, the low original cost of the latter being outweighed by the heavy expense of initial application and subsequent upkeep, due to poor spreading power and durability.

S. S. WOOLF.

Additive quality of oil absorption [of pigments]. J. T. BALDWIN (Ind. Eng. Chem., 1929, 21, 326—329).—Determinations of oil absorption of various pigment mixtures indicate that, in general, oil absorption is additive, but that where active pigments, *e.g.*, red lead or zinc oxide, are present, the straight-line law is modified, probably by the extent of soap formation and by the rate of diffusion of the soap through the oil to the surface of the inert pigment. "Oil" absorption is considered to be a function of the specific surface of the pigment, and of the affinity of the latter for the particular wetting liquid. The relationship is expressed by $L = S/A^n$, where L is the liquid absorption, S is a surface constant depending on the effective surface of

the powder, A is the adhesion tension, and n is a constant (approx. 0.75). Some applications of this relationship are indicated.

S. S. WOOLF.

Lithopone darkening. J. H. GOSHORN and C. K. BLACK (Ind. Eng. Chem., 1929, 21, 348—349).—The phenomena of the darkening of lithopone pigments on exposure to ultra-violet light, especially in the presence of water, and the re-whitening of the darkened pigments if kept in the dark were examined by the exposure to ultra-violet light of lithopones, pure and contaminated, zinc oxide, zinc oxide intimately mixed with sulphur, zinc oxide covered with aqueous hydrogen sulphide, etc. The free sulphur in exposed lithopone was determined by extraction with carbon tetrachloride. From the results of these and other tests it is considered that the darkening of lithopone is due to reduction to metallic zinc, and probably free sulphur, whilst the re-whitening is due to the oxidation of zinc. Free sulphur takes part in the re-darkening observed on further exposure to light. Metals present, even in the quantities used in driers, cause darkening both by the formation of metallic zinc and of dark-coloured sulphides.

S. S. WOOLF.

Colouring of cellulose acetate solutions. C. F. J. NEWBY (J. Soc. Dyers & Col., 1929, 45, 104—107).—Lacquers made from cellulose acetate may be incorporated with lakes and dyes. An enamel made by dissolving cellulose acetate in acetone and adding a plasticiser can be coloured by means of pigments and lakes, dyes, or metallic and coloured bronzes. The pigment colour is first "wetted" with a carrying agent, and then added to the acetate vehicle. Such enamels must be allowed time in which to mature. Dyes cannot satisfactorily replace pigments, and, for example, better results are obtained by first colouring wood with a water-soluble stain and subsequently applying the varnish, than by using a clear-coloured varnish. For colouring cellulose acetate, metal powders may be used in the form of (a) plain colour, (b) colour supercoated with a dyed solution, (c) an admixture with a dyed solution to give an "Abbo" colour, or (d) admixture with a pigment. A small range of colours can be obtained by using a coloured bronze or by superimposing a blue dye solution over a silver-coloured paint. Black can be obtained by using a deep spirit-soluble blue-black.

L. G. LAWRIE.

Degree of unsaturation of the resin acids. IV. B. M. MARGOSCHES, K. FUCHS, and W. RUZICKA (Chem. Umschau, 1929, 36, 115—118; cf. B., 1927, 228, 371, 755).—Suggested structural formulæ for abietic acid are discussed. The concept in Grün's formula of a double linking in the molecule in $\alpha\beta$ -relation to a tertiary carboxyl group is rejected: the difficulty experienced in esterification and the behaviour on reduction indicate a secondary carboxyl grouping, which, in view of the high halogen absorption, must be at a considerable distance from the double linking. A modified formula is constructed in accordance with these conclusions.

E. LEWKOWITSCH.

Ultra-violet light-transmission characteristics of some synthetic resins. D. L. GAMBLE and G. F. A. STUTZ (Ind. Eng. Chem., 1929, 21, 330—333).—The decrease in transmission of ultra-violet light and the

extent of yellowing on exposing resins to ultra-violet light were observed for a number of synthetic and natural resins, and the results are tabulated. The natural resins are, in general, less sensitive and more transparent to ultra-violet light than the synthetic resins. The resins tested are graded as to their opacity and stability, and from these considerations resin esters are considered the most desirable type of resin for protective coatings, whilst "glyptals," phenol-formaldehyde, and coumarone resins are fairly satisfactory. It is pointed out, however, that physical and chemical properties and resistance to weathering in general must also be taken into account in order to arrive at the optimum ultra-violet transmission characteristics for resins.

S. S. WOOLF.

"Alkyd" resins as film-forming materials. R. H. KIENLE and C. S. FERGUSON (Ind. Eng. Chem., 1929, 21, 349—352).—The properties of "alkyd" resins (*i.e.*, those based on the interaction of the higher polyhydric alcohols with polybasic acids) are described, and their suitability for use in air-drying and baking lacquers is indicated. The oxygen-convertible resins (*i.e.*, those containing oxidisable unsaturated fatty acids in addition to polybasic acids) are considered to be of great importance as air-drying materials, since they possess the quick drying and hardening characteristics of nitrocellulose lacquers and the film-building properties of oleo-resinous varnishes. Their drying reaction is primarily one of oxidation, heat merely accelerating the process, whereas the "glyptal" type of resin requires heat to develop its maximum properties, *e.g.*, toughness, adhesion, flexibility, and oil resistance. S. S. WOOLF.

Corrosion of painted steel. EVANS.—See X. **Lead soaps.** RAGG. **Drying oils.** CHAMBERLIN and others.—See XII. **Pigment reinforcement of rubber.** LUNN. **Prussian-blue etc. in rubber.** SCOTT. **Titanium dioxide in rubber industry.** DITMAR.—See XIV.

PATENTS.

Coating composition. C. E. BRADLEY and W. A. GIBBONS, Assrs. to NAUGATUCK CHEM. CO. (U.S.P. 1,703,920, 5.3.29. Appl., 2.7.21).—A solution of halogenated vulcanised rubber in benzol or solvent naphtha is claimed.

S. S. WOOLF.

Calcination of lithopone. W. J. LINDSAY, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,704,482—3, 5.3.29. Appl., [A] 20.4.27, [B] 21.9.21).—In (A) apparatus is described for carrying-out the process of (B) in which lithopone is heated solely by the passage therethrough and in contact therewith of a non-reactive gas heated at a temperature above that at which the lithopone is to be calcined.

S. S. WOOLF.

Anti-corrosive [varnish] material. M. TOCH, Assr. to STANDARD VARNISH WORK (U.S.P. 1,706,333, 19.3.29. Appl., 8.5.25).—Petrolatum is incorporated with zinc chromate.

H. ROYAL-DAWSON.

Manufacture of resins for lacquers, varnishes, etc. IMPERIAL CHEM. INDUSTRIES, LTD., N. STRAFFORD, and E. E. WALKER (B.P. 308,048, 15.2.28).—A thermo-hardening phenol-aldehyde resin from which free phenols and condensation products of low mol. wt. have

been removed (*cf.* B.P. 296,514; B., 1928, 826) is plasticised by the addition of a "glyptal" resin that has been modified by incorporation with fatty acids, *e.g.*, of linseed or tung oil, or with oils, *e.g.*, castor oil, in the presence or absence of a hardening catalyst. The compound resin may be used as such for moulding powders etc., or in solution as low-temperature hardening lacquers. In the latter application, the transparency and hardness of the finish obtained are improved by the incorporation in the lacquer of alcoholic sulphuric acid.

S. S. WOOLF.

Phenol resin and its manufacture. F. GROFF, Assr. to BAKELITE CORP. (U.S.P. 1,693,112, 27.11.28. Appl., 21.2.27).—30—80% of phenol-formaldehyde "resole" or a natural resin is added to a partially reacted phenol-furfuraldehyde mixture before gelatinisation occurs, and heating is continued at 145—150°.

C. HOLLINS.

Phenolic resin composition. V. H. TURKINGTON, Assr. to BAKELITE CORP. (U.S.P. 1,695,566, 18.12.28. Appl., 24.5.26).—A thermo-hardening resin, either phenol-aldehyde, urea-aldehyde, or "glyptal," is plasticised by adding butyl phthalate to give varnish compositions yielding flexible films.

C. HOLLINS.

Manufacture of condensation products [synthetic resins]. J. A. NIEUWLAND, Assr. to CANADIAN ELECTRO PRODUCTS CO., LTD. (U.S.P. 1,707,941, 2.4.29. Appl., 25.7.21).—See B.P. 183,830; B., 1924, 265.

[Sand-blast] treatment of copal or similar gums. J. C. VAN DER TAELEN, and SOC. COLONIALE ANVERSOISE SOC. ANON. (B.P. 309,254, 30.1.28).

Alkaline-earth titanates (B.P. 308,516).—See VII. **Lacquered wire** (B.P. 308,508).—See XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Constitution and mechanical properties of rubber. L. HOCK and G. FROMANTI (Kautschuk, 1929, 5, 81—82).—The iodine values of smoked sheet rubber and Congo rubber are so nearly equal that the difference in quality of such grades can hardly be attributed to their relative degree of unsaturation. Surface tension phenomena are probably involved in such differences.

D. F. TWISS.

Vulcanisation and constitution of rubber. H. LOEWEN (Kautschuk, 1929, 5, 77—79).—A criticism of the value and foundations of certain recent theories as to the constitution of rubber, with especial reference to the "needle-shaped" molecules of Lindmayer (B., 1929, 294).

D. F. TWISS.

China clay as reinforcing agent in rubber compounding. T. J. DRAKELEY and W. F. O. POLLETT (Trans. Inst. Rubber Ind., 1929, 4, 424—460).—Six grades of clay were examined as to their influence as compounding ingredients in a mixture of rubber (100), zinc oxide (5), sulphur (4), and diphenylguanidine (1). None retarded vulcanisation, but some which were alkaline in reaction had an accelerating effect equal to that of an equivalent quantity of sodium carbonate. A small proportion of stearic acid did not appreciably affect the accelerating action of clays, but it practically eliminated the retardation by carbon-black. Up to 18 vols. (rubber 100 vols.) the clays increased the tensile

strength considerably, the more refined grades exceeding the effect of an equal volume of gas-black; the highest percentage at which any clay produced an increase in resilient energy was 25 vols., this proportion being lower for poorer grades. The clays gave a maximum increase in resilient energy higher than that given by gas-black, but this reinforced at higher volume proportions and also increased the rigidity of the mixture to a greater extent.

D. F. TWISS.

Pigment reinforcement [of rubber]. R. W. LUNN (Trans. Inst. Rubber Ind., 1929, 4, 396—412).—An attempt is made to apportion the resilient energy for a vulcanised rubber mixture amongst its ingredients, particularly the rubber and filler or reinforcing agent ("pigment"). Making assumptions necessary for this apportionment, carbon-black causes an increase in the total energy up to high concentrations; even barium sulphate (precipitated) acts as a reinforcing agent up to about 60 vols. per 100 vols. of rubber. The curves plotting the relation of contributed resilient energy (for different vol.-% of "filler") against elongation of the rubber mixture are remarkably similar up to the breaking point for barium sulphate, zinc oxide, and therm-atomic carbon in proportions up to 50 vols. The contribution to the resilient energy of the mixture, calculated per unit volume of filler, decreases with increase in the concentration and tends towards zero; above 50 vols. per 100 of rubber the contribution per unit volume is almost equal for all the "pigments" examined.

D. F. TWISS.

Behaviour of Prussian-blue and some other ferrocyanides in rubber. J. R. SCOTT (Trans. Inst. Rubber Ind., 1929, 4, 374—395).—Although of comparable particle size, Prussian-blue and Turnbull's-blue do not give identical behaviour in rubber, the former, for example, dispersing much more readily. Rubber containing Prussian-blue, when heated or milled, becomes very soft on account of oxidation; the presence of sulphur or hydroquinone inhibits this oxidation, and permits the customary hardening on storage. Turnbull's-blue is less favourable to the oxidation and zinc and lead ferrocyanides are still more so. The refractive index of Prussian- and Turnbull's-blue approximates closely to that of rubber; mixtures with rubber are consequently transparent. Vulcanisation destroys the blue colour, which becomes partially restored, however, on subsequent exposure to air. All the ferrocyanides named retard vulcanisation and reduce the tensile strength, increase the permanent set, and accelerate the "perishing" of the vulcanised rubber. Volatile decomposition products of a poisonous nature are also formed from the two blues during vulcanisation. These colours are not suitable for rubber intended to be vulcanised by heat. In connexion with the plasticity measurements a new equation is given for the rate of deformation or plastic flow of unvulcanised rubber under pressure between parallel plates.

D. F. TWISS.

Titanium dioxide in the rubber industry. R. DITMAR (Kautschuk, 1929, 5, 79—81).—Titanium dioxide is non-poisonous and has great covering power. Up to 5 or 6 % in rubber it behaves as an inert filler, but beyond this proportion it has a plastifying effect. In spite of its

opacity, some organic accelerators and antioxidants lead to discoloration of rubber pigmented with it. It can be used satisfactorily in solutions for articles to be made by dipping and cold vulcanisation.

D. F. TWISS.

Effect of antioxidants in typical rubber stocks. M. C. REED (Ind. Eng. Chem., 1929, 21, 316—318).—Results of tests on various commercial grades of rubber mixtures, *e.g.*, tyre treads, inner tubes, steam hose, and sponge show that under conditions of natural or artificial ageing, the maintenance of quality is definitely favoured by the presence of an antioxidant; the effect of such preservatives is of especial value in stocks likely to become hot in service. Ageing at 90° both with and without the presence of oxygen should be of assistance in separating the factors of oxidation and over-vulcanisation when compounding rubber for heat-resistance purposes.

D. F. TWISS.

Effect of mixtures of crude ozokerite and agerite on the ageing of rubber mixtures. F. KIRCHHOF (Caoutchouc et Gutta-Percha, 1929, 26, 14501—14503).—Refined ozokerite is inferior to the crude product in its protective influence against the ageing of rubber mixtures. Mixtures of crude ozokerite with some commercial antioxidants, formed by fusing the constituents together, are remarkably effective in preserving rubber; *e.g.*, 0.1% of a commercial antioxidant previously blended with crude ozokerite in the proportion 3:100 provides as marked protective action in a rubber mixture as 5 times as large a quantity of the same antioxidant introduced alone in the ordinary manner.

D. F. TWISS.

Tackiness of unvulcanised rubber. T. L. GARNER (Trans. Inst. Rubber Ind., 1929, 4, 413—423).—Experiments on the effect of mastication, air, light, and antioxidants, respectively, on rubber and rubber solutions show that the processes of tackiness development (or loss of viscosity) and oxidation run concurrently, but even in the absence of oxygen the viscosity of dissolved rubber is reduced by ultra-violet light. The effect of light is partly polymerisation and partly depolymerisation, the latter preponderating in an ordinary atmosphere whilst in an atmosphere containing nitrogen with only a small proportion of oxygen the rubber, although becoming equally tacky, is in part rendered insoluble. The relative effect of various antioxidants in retarding the decrease in viscosity of irradiated rubber stands in no simple relation to their effect on actual oxidation. By favouring the formation of an insoluble polymeride on the surface of exposed rubber, antioxidants indeed may accelerate loss of surface tackiness.

D. F. TWISS.

PATENTS.

Preservation of rubber latex. J. MCGAVACK and R. A. SHIVE, ASSRS. to NAUGATUCK CHEM. Co. (U.S.P. 1,699,368—9, 15.1.29. Appl., 8.4.27 and 2.3.28).—Latex is stabilised by treatment with a preservative in presence of an acidic substance giving a latex of p_H 4.5—7. *E.g.*, 1 litre of natural latex is treated with 3—5 g. of the reaction product of cinnamic acid, butyl alcohol, and sulphuric or chlorosulphonic acid, and 10—15 c.c. of 38% formaldehyde are added. In treating ammonia-preserved latex the ammonia is removed by blowing,

neutralising, *e.g.*, with acetic or boric acid, or by treatment with excess, *e.g.*, 30–35 c.c. of formaldehyde solution. Other reaction products of aromatic acids sulphonated in presence of aliphatic alcohols, or of phenols and hydrocarbons sulphonated in presence of fatty acids, *e.g.*, of *p*-cresol and oleic acid in presence of sulphuric or chlorosulphonic acid, may be used. R. BRIGHTMAN.

Anti-oxidant or age-resister [for rubber]. A. M. CLIFFORD, Asst. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,694,529, 11.12.28. Appl. 13.12.27).—Dinaphthylamines, especially the $\beta\beta$ -compound, are anti-agers for rubber. C. HOLLINS.

Manufacture of articles from aqueous dispersions containing organic materials. DUNLOP RUBBER Co., LTD., W. H. CHAPMAN, and F. H. LANE (B.P. 308,626, 24.9.27).—Products obtained by deposition from aqueous dispersions of organic substances, *e.g.*, rubber, are dried or partly dried, with or without simultaneous moulding, by pressing the products (which may be removed for the purpose from their deposition bases) against or between surfaces through which water may escape; the surfaces may be composed of absorbent materials, *e.g.*, porous ceramic or fibrous material. D. F. TWISS.

Vulcanised rubber composition and its manufacture. W. P. TER HORST, Asst. to RUBBER SERVICE LABS. Co. (U.S.P. 1,698,569, 8.1.29. Appl. 18.6.27).—Pentamethylenephénylguanidine, obtained, *e.g.*, from phenylcyanamide and piperidine, is claimed as a vulcanisation accelerator. C. HOLLINS.

Composite product [rubber cement]. H. GRAY, Asst. to B. F. GOODRICH Co. (U.S.P. 1,697,275, 1.1.29. Appl. 27.4.27).—Turpentine thickened by treatment with stannous sulphate, trichloroacetic acid, sulphur chloride, or other substance generating strong acid yields a cement for bonding rubber to metal. C. HOLLINS.

Rubber coatings (B.P. 282,737).—See X. (U.S.P. 1,703,920).—See XIII.

XV.—LEATHER; GLUE.

Tannery laboratory equipment. C. H. SPIERS (J. Soc. Leather Trades' Chem., 1929, 13, 8–10).—The use of stainless steel balance pans; of titration apparatus consisting of a burette fitted at its upper end with a cork and 3-way tube, one limb of which is connected to a reservoir of the reagent and the other to a rubber suction tube closed with a spring clip; and of pipettes to deliver specified amounts of a pickling liquor for titration with 0.1N-alkali is advocated. A comparator is also described in which the second row of tubes is replaced by a sliding rack carrying alternatively placed tubes containing pure water and different standard tubes. D. WOODROFFE.

Moisture in leather. H. BRADLEY, A. T. MCKAY, and B. WORSWICK (J. Soc. Leather Trades' Chem., 1929, 13, 10–24).—The moisture in leather varies with the relative humidity (R.H.) of the atmosphere in which the leather is placed, and the type of leather. Temperature has little effect apart from that on the R.H. value. Samples of different leathers were placed in atmospheres of different R.H.; when equilibrium

had been established the sample was weighed and the moisture content taken as the increase in weight from zero humidity. The results showed that leather absorbs more moisture than cotton, but less than wool. The order of diminishing absorptivity was wool, sole leather, patent, insole, box calf, special-process sole leather, wood, curried vegetable-tanned splits, cotton. Leathers with the higher content of grease had the lower absorptive power. Leather gave up its moisture slowly at low humidities, but dry leather absorbed moisture rapidly at those humidities. At higher humidities drying out and absorption took place at about the same rate. D. WOODROFFE.

Determination of fat in leather. F. G. A. ENNA (J. Soc. Leather Trades' Chem., 1929, 13, 37–38).—The vacuum oven suggested by Woodroffe (B., 1929, 257) for drying off fat extracts from leather is criticised. The author uses an oven filled with carbon dioxide to prevent oxidation of highly unsaturated oils. D. WOODROFFE.

Effect of heat on wetted vegetable-tanned leather. III. W. J. CHATER (J. Soc. Leather Trades' Chem., 1929, 13, 24–36; cf. B., 1929, 257, 295).—Shrinkage curves have been obtained for pieces of vegetable-tanned sheepskin, which had been treated with solutions of borax, limed calfskin, and pieces of calf pelt, which had been treated with solutions of different p_H values, respectively. It is shown that the treatment affects the type of curve obtained. D. WOODROFFE.

PATENTS.

Stuffing of leather. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 307,775, 13.12.27).—Leather is stuffed, before or after dyeing, with sulphonated fatty acid derived from wool fat or with an aqueous emulsion of the same. The sulphonated product may be prepared as in B.P. 307,776 (B., 1929, 442).

D. WOODROFFE.

Rapid vegetable-tanning process. G. PEACE, and BRIT. INDIA CORP., LTD. (B.P. 307,987, 21.12.27).—Delimed hides are pickled, *e.g.*, in an aqueous solution of salt and sulphuric acid, depickled with an aqueous solution of the sodium salt of a weak acid such as sodium thiosulphate, washed until they show p_H 3.5–5.0, and tanned in liquors at this p_H range using strong extract liquors and mechanical agitation to shorten the time of tanning. D. WOODROFFE.

Production of flour-like horn etc. by grinding. H. OEXMANN (B.P. 297,841, 28.9.28. Ger., 30.9.27).—Cylindrical blocks of pressed horn mass are peripherally ground in the dry state by means of discs of natural or artificial resins, casein, or other albuminous substances, or cellulose derivatives, by intermittent grinding movements, and are continually turned to secure good cooling. D. WOODROFFE.

Shoe creams (B.P. 308,996).—See II. **Water-soluble products** (B.P. 307,776).—See XII.

XVI.—AGRICULTURE.

Rigidity in weak clay suspensions. R. K. SCHOFIELD and B. A. KEEN (Nature, 1929, 123, 492–493).—In a suspension of the clay fraction of soil in dilute hydrochloric acid, sharp ramifying fissures containing

clear liquid develop when the concentration of the suspension exceeds a certain critical value. The density difference sets up a circulation, which is completed by the deposition of floccules in a ring around the exit of the chimneys (conical fissures). The suspension acquires rigidity; the flocculation layer of more dilute suspensions shows rigidity only when a layer of sufficiently great concentration has been formed. The phenomenon is influenced by the amount of electrolyte present.

A. A. ELDRIDGE.

Determination and application of the electrical resistance and p_H value in irrigation soil surveys. A. STEAD (S. Afr. J. Sci., 1928, 25, 149—155).—The amount of brak in soils is assessed in the field by the simultaneous measurement of p_H values (quinhydrone electrode) and the electrical resistance of the wetted soil. Allowance is made in the latter determination for the soil type (sand, loam, clay) and for the temperature. The importance of depth sampling in soils for proposed irrigation is emphasised. Brak may occur at a depth which normally does not influence surface vegetation, but irrigation tends to raise this towards the surface.

A. G. POLLARD.

Microbiological activities in the soil of an upland bog in eastern North Carolina. I. V. SHUNK (Soil Sci., 1929, 27, 283—303).—Examination of a sedge-grass bog soil showed that nitrification could be initiated in 6—10 weeks by liming and drainage. Liming largely increased the bacterial numbers, but fungi were not appreciably affected. The rate of production of carbon dioxide was markedly increased by liming, and was also largely influenced by the soil moisture content. There was an optimum moisture content above and below which carbon dioxide production was retarded.

A. G. POLLARD.

Influences of the development of higher plants on the micro-organisms of the soil. I. Historical and introductory. R. L. STARKEY (Soil Sci., 1929, 27, 319—334).—The numbers of filamentous fungi, actinomycetes, and bacteria in soils were greater in the immediate vicinity of the plant roots than at a distance. The absolute numbers and relative proportions of these organisms near to plant roots varied with different plants. Carbon dioxide production was greater in soils containing growing plant roots than in those free of plants.

A. G. POLLARD.

Fungicidal action of ultra-violet radiation. H. R. FULTON and W. W. COBLENTZ (J. Agric. Res., 1929, 38, 159—168).—Spores of 27 miscellaneous species of fungi were exposed for 1 min. to ultra-violet radiations, from a mercury-tungsten arc at a distance of 6 in. There was complete killing of the spores of 16 species and a survival of less than 1% in the case of 4 species. Such dosage causes no injury to oranges. The limitation to the use of ultra-violet rays for complete disinfection of fruits lies in their lack of penetration below the surface. The shorter wave-length components of the ultra-violet spectrum have the greatest germicidal action.

E. A. LUNT.

Effect of moisture content and cropping on exchangeable calcium and magnesium, with particular reference to rice soil. W. H. METZGER

(Soil Sci., 1929, 27, 305—318).—Long-continued cropping of a rice soil resulted in much larger exchangeable calcium and magnesium contents in the surface soil than in the subsoil. Maintenance of a high moisture content in soils increased the contents of exchangeable magnesium without influencing the exchangeable calcium content except in cases of flooding, when this was slightly decreased. Comparison of the exchangeable calcium and magnesium in a fallowed soil with the same cropped with wheat showed no appreciable differences.

A. G. POLLARD.

Interchange of the phosphoric acid of the soil with arsenic acid. C. ANTONIANI and G. FONIO (Atti R. Accad. Lincei, 1929, [vi], 9, 344—350).—The phosphate ion of soil which has been treated with sodium phosphate behaves differently towards water and dilute arsenic acid solution, which takes up less of the phosphate and is to some extent retained by the soil.

T. H. POPE.

The "effect-law" of growth factors. O. TORNAU and K. MEYER (J. Landw., 1929, 77, 65—96).—Results of cultural experiments by the Mitscherlich method are examined to verify the nature of the descending portions of growth curves of plants produced by excessive nitrogenous feeding, and the alleged constancy of the "effect factor" with a number of different plants. The rate of descent of the growth curve from the maximum following the application of large dressings of ammonium nitrate differed with different plants, mustard and spurrey being much more sensitive than maize and oats to these conditions. The values of the "effect factor" were in general agreement with those of Mitscherlich, but the nitrogen content of the soil as indicated by the growth curve varied with different plants. Each plant appears to possess a specific power of assimilation for nitrogen. Examination of the nitrogen contents of different plants indicated different capacities and different "saturation-conditions" for nitrogen, and these are related to the maximum production of organic matter. The limit of increased growth resulting from the successively increased supplies of any one nutrient is determined by the raising of the "saturation-condition" of the plant.

A. G. POLLARD.

Value of increasing applications of nitrogen to different species of winter rye. J. WEIGERT and F. FÜRST (Z. Pflanz. Düng., 1929, 8B, 161—202).—The results of nine years' manuring trials with rye are recorded. The effect of manuring and rainfall on the yield and quality of straw and grain, and the general development of numerous species of winter rye is discussed.

A. G. POLLARD.

Correlation between soil salinity and flowering date in cotton. J. A. HARRIS (J. Agric. Res., 1929, 38, 109—112).—The existence of a low negative correlation between soil resistance and period required for flowering is indicated, thus suggesting the retardation of the flowering date by soil salinity.

E. A. LUNT.

Certain phases of the interrelationship between soil and plant. I. Availability of mineral plant nutrients in relation to degree of dispersion. W. THOMAS (Soil Sci., 1929, 27, 249—270).—Consideration is given to published work on the nature of soil colloids and dialysis as a basis of the mechanism of the

assimilation of mineral nutrients by plants. Comber's views on the colloidal adsorption of nutrients are considered insufficiently proved in many instances. Evidence in support of the conception that dissolution must precede the intake of nutrients by plants is discussed.

A. G. POLLARD.

Fertiliser trials with sodium nitrate, Chile salt-petre, and iodine on carrots. DENSCH, STEINFATT, and GÜNTHER (Z. Pflanz. Düng., 1929, 8B, 205—211).—Increased leaf production resulted from the application of iodine manures to carrots. Assimilation of iodine from iodides by plants probably occurs only in the presence of iodates. In other crops root injury was observed after iodine manuring. The amount of iodine in Chile saltpetre is too small to have any appreciable effect on plant growth.

A. G. POLLARD.

Computation of no-filler fertiliser mixtures. A. B. BEAUMONT and H. R. KNUDSEN (Ind. Eng. Chem., 1929, 21, 385—388).—Algebraic methods are given for calculating mixture ratios required to give a mixed fertiliser of a given grade (i.e., percentage of N, P_2O_5 , and K_2O), without any filler being added, from the various fertiliser materials available. With single-element components only, e.g., ammonium sulphate, the problem is easily solved by arithmetic. Examples are given of the algebraic solution of problems with one or more "double-element" compounds with "single-element" compounds and with four "single-element" compounds. In these cases one of the fertilising "elements" is present in two forms, and the ratio between these two forms necessary to give the required composition without filler is obtained by solving two simultaneous equations.

C. IRWIN.

Hygroscopicity of fertiliser materials and mixtures. J. R. ADAMS and A. R. MERZ (Ind. Eng. Chem., 1929, 21, 305—307).—The tendency of a substance to absorb water from the atmosphere is directly proportional to the difference between the vapour pressure of its saturated solution and that of water. The determination of such vapour pressures gives the most accurate measurement of hygroscopicity. Determinations at different temperatures for the saturated solutions of a number of fertiliser compounds are tabulated. A further series of determinations was made on two component mixtures. These are usually more hygroscopic than the most hygroscopic component, but exceptions occur where a double salt or compound is formed as between calcium nitrate and urea.

C. IRWIN.

Rôle of nitrogen fertilisers in the treatment of fish-ponds with artificial manures. KUHNERT (Z. Pflanz. Düng., 1929, 8B, 202—205).—Published work on this question is discussed in the light of the writer's demonstration of the increased fish yield resulting from the use of nitrogenous and other fertilisers.

A. G. POLLARD.

Phosphorus deficiency in forage feeds of range cattle. S. G. SCOTT (J. Agric. Res., 1929, 38, 113—130).—The osteomalacia and bone-chewing diseases prevalent among cattle in certain districts of Montana is shown to be due to phosphorus deficiency in the forage crops of such districts. The phosphorus content of the soil in such areas shows no subnormality. E. A. LUNT.

Toxic property of sulphur. WILLIAMS and YOUNG.—See VII.

PATENTS.

Manufacture of fertilisers. A. B. KENSINGTON (B.P. 301,105, 25.8.27).—Bird manure is mixed with sufficient absorbent organic material, e.g., peat moss, to render the material sufficiently porous to be dried to a brittle mass, and with an acid or an acid salt to neutralise any free ammonia. The mixture is dried with agitation and pulverised.

A. R. POWELL.

Production of granular fertilisers. C. C. SMITH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,299, 31.3.28).—A hot, concentrated slurry of the constituents of the fertilisers described in B.P. 305,760 (B., 1929, 298) is sprayed down a tower in which it is brought in contact with an ascending stream of cold air, and the granular product collecting at the bottom is passed through a rotary dryer and is then sieved.

L. A. COLES.

Stimulating the germination of seeds and other vegetable matter. M. POPOFF, Assr. to BAYERISCHE STICKSTOFF WERKE A.-G. (U.S.P. 1,703,362, 26.2.29. Appl., 18.3.25. Ger., 23.5.24).—A portion of the seeds is treated with a solution containing magnesium chloride and sulphate, and the remainder with a solution of potassium bromide (for rye), magnesium sulphate and manganese sulphate (for wheat and oats), or magnesium sulphate and manganese nitrate (for barley); the two portions are mixed together and sown.

A. R. POWELL.

Preparation for use as an additive to screened house and street refuse for producing a fertilising and vermin-destroying agent and for preventing diseases of crops, or for use in the pure state as a cauterising agent for seeds. F. BAERNER (B.P. 308,328, 19.12.27).—Dry-ground and slaked lime (3 pts.) is mixed with 1 pt. of finely-ground and "glowed" infusorial silica or 1 pt. of silicic acid powder, the latter being previously saturated with aqueous formaldehyde solution and then with carbon disulphide.

A. G. POLLARD.

Herbicidal [and fireproofing] products. COMP. DE PROD. CHIM. ET ELECTROMÉTALLURGIQUES ALAIS, FROGES & CAMARGUE (B.P. 273,268, 2.6.27. Fr., 25.6.26).—The products, which also possess fireproofing properties when used for treating combustible material, comprise mixtures of 1 pt. of calcium chlorate with 1—5 pts. of calcium chloride, with or without the addition of ferrous sulphate, phenols, etc. [Stat. ref.]

L. A. COLES.

Treatment of seed. A. KLAGES (U.S.P. 1,706,695, 26.3.29. Appl., 17.5.26. Ger., 27.10.23).—See B.P. 274,974; B., 1927, 792.

Organic refuse (B.P. 300,607).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Correct procedure in sulphitation of thin [sugar] juice. II. Sulphitation. E. THIELEPAPE and P. MEIER (Z. Ver. deut. Zucker-Ind., 1929, 79, 176—193; cf. B., 1928, 498).—Since the removal of excess lime is important in minimising incrustation, laboratory carbonatations and sulphitations of juices

from the first presses were carried out with special reference to this and to the improvement of colour. Lime was most completely removed when the juice was filtered between carbonatation and sulphitation, and when the latter was continued to slight acidity and the excess neutralised by sodium carbonate. The limy material precipitated by this neutralisation contained little, if any, calcium sulphite. The de-liming effect is not merely due to acidification and neutralisation, since hydrochloric acid cannot replace sulphurous acid. In a comparative test a juice contained 0.0056% of lime at a final alkalinity of 0.024, and 0.0016% at alkalinity 0.032 when treated by the author's method, against 0.0197% at alkalinity 0.020 when treated by Weisberg's method. Equally good results were obtained by adding the required amount of sodium carbonate to the juice at the second carbonatation, and, after filtration, reducing the alkalinity to about 0.02—0.03 by sulphurous acid. Reducing the amount of sodium carbonate from 0.14% to 0.08% increased the lime from 0.0024% to 0.0219%. The latter amount was reduced to 0.0109% by heating under pressure for 30 min. at 120—124°. Similar treatment is applicable to thick juice. No inversion was observed in juice of acidity 0.020 when kept for 20 min. at 95°. Marked reduction of colour was occasionally noted. F. E. DAY.

Cane-wax complex in juices from cane-sugar mills. C. F. BARDORF (Ind. Eng. Chem., 1929, 21, 366—367; cf. B., 1928, 540).—The migration of the cane-wax in cane-sugar mills resembles that in the refinery. F. R. ENNOS.

Low-temperature injury to stored sugar cane. G. B. SARTORIS (J. Agric. Res., 1929, 38, 195—203).—Experimental evidence is cited showing that the cold-storage of sugar-cane seed as a regular plantation routine is not practicable. The optimum temperature for the storage of sugar cane is 8—10°. E. A. LUNT.

Applications of the nitrite method. IV. Significance of the hydrocyanic acid number in the analysis of mixtures of sugars. Determination of sugars in marmalade. F. LIPICH (Z. anal. Chem., 1929, 77, 3—12).—The composition of mixtures of invert sugar, dextrose, and sucrose may be calculated from the polarimetric readings and hydrocyanic acid numbers (B., 1929, 412, 414) before and after inversion. The amount and composition of the sugars present in marmalade may be obtained by making these measurements on an aqueous extract. J. S. CARTER.

Self-emptying suction flask for sugar determinations. G. PITMAN (Ind. Eng. Chem. [Anal.], 1929, 1, 112).—Wash liquids are continuously removed by means of a bent tube reaching to the bottom of the filter flask and connected at its upper end with the suction outlet. J. S. CARTER.

Decomposition of starch. WREDE.—See XVIII.

PATENTS.

Purification of diffused sugar syrup. K. KOMERS and K. CUKER (B.P. 308,031, 1.2.28).—Diffusion juice is strongly aerated for a short time, e.g., 5 min., whereby dark oxidation products are formed, which are then

precipitated by addition of lime (0.1—0.2%) or calcium salts such as phosphate, excess of lime being carefully avoided. The process may be repeated so long as aeration produces further darkening. J. H. LANE.

Removal of hydrochloric acid from sugar solutions. A. CLASSEN (B.P. 307,998, 31.12.27).—The sugar solutions or syrups are distilled together with inert organic liquids such as dichloroethylene, preferably under reduced pressure. The permeation of the sugar solution by the vapour of the organic liquid boiling below it causes the hydrochloric acid to pass over into the distillate. J. H. LANE.

XVIII.—FERMENTATION INDUSTRIES.

Determination of alcohol in wines and spirits. L. SEMICHON and FLANZY (Ann. Falsif., 1929, 22, 139—152).—20 c.c. of wine are diluted with about 30 c.c. of water and rather more than half the mixture is distilled, the distillate being made up to 100 c.c. Into a cooled mixture of 20 c.c. of potassium dichromate solution (33.83 g. per litre, so that 1 c.c. will liberate with sulphuric acid 1% of alcohol under the conditions of experiment) and 10 c.c. of sulphuric acid, *d* 1.84, are poured 5 c.c. of the alcoholic solution. After 5—10 min. oxidation is complete, and the liquid is titrated with ferrous ammonium sulphate (135.31 g. in 700—800 c.c. of water to which is added 20 c.c. of sulphuric acid and the whole made up to 1 litre; 2 c.c. correspond to 1 c.c. of the dichromate solution) to the point of formation of Turnbull's blue. Then the degrees of alcohol in the wine are $(20 - n/2)$, where *n* is the number of c.c. in the titration. This method gives at least as great a degree of accuracy as the French official method, takes but 20 min., and requires only a 20 c.c. sample.

D. G. HEWER.

Erratum.—B., 1929, 145, col. 1, lines 2, 13, and 14 from bottom, for "still wines" read "brandies."

Preparation of banana vinegar. H. VON LOESECKE (Ind. Eng. Chem., 1929, 21, 175—176).—Banana mash made from the pulp and peel of ripe bananas and containing about 16% of fermentable sugars is pasteurised at 75° for 45 min. and inoculated, after cooling, with *Saccharomyces ellipsoideus*. The mash is incubated for 3 days at 20—23° and then used as an inoculator for larger quantities of mash which are incubated for 14—20 days. The mixture is filter-pressed and the filtrate centrifuged to remove suspended yeast. The "banana cider" thus formed contains 6.5—10% of alcohol and is about 56% of the weight of fruit taken; it is treated with 25% of its volume of strong vinegar and incubated at 30° for 75 days. The product is filtered and the filtrate clarified with 2% of kieselguhr and pasteurised for 1 min. at 60°. The resulting vinegar is light amber in colour and possesses an agreeable aroma and flavour. A. R. POWELL.

Rapid dehydration of alcohol using barium oxide and metallic calcium. G. F. SMITH (Ind. Eng. Chem. [Anal.], 1929, 1, 72—74).—Systematic investigations show that 92—93% alcohol is simply, economically, and, relatively, rapidly dehydrated by treatment with barium oxide. The last 0.5% of water is removable by metallic calcium. J. S. CARTER.

Liquefaction and decomposition of starch by biolase and its use in the paper industry. H. WREDE (Papier-Fabr., 1929, 27, 197—202).—Processes for the production of soluble starches by the action of dilute mineral acids, bases, oxidising agents (including chlorine and activin), and enzyme preparations are described and their advantages and disadvantages discussed. In particular, the diastatic action of biolase (from the lupin plant) on different kinds of starch, and means whereby the extent of its action may be controlled are described. Biolase is the most energetic of the enzyme preparations, and has a temperature range of 20° to 80°; its optimum activity is at 50—55°. In suitable apparatus starch pastes of up to 50% concentration and of high binding power may be prepared by the use of not more than 1% of biolase, whilst for the decomposition of 20% pastes $\frac{1}{2}$ — $\frac{3}{4}$ % suffices. Its action on potato starch is too energetic, and losses occur through the formation of maltoses etc., hence maize starch is preferred on account of its greater resistance to such attack. Details for the preparation of soluble starch pastes of relatively high concentrations which are liquid when cold, or gelatinised when cold but liquid when warm, are given, and the advantages of the use of such pastes in different branches of the paper industry are discussed. B. P. RIDGE.

PATENTS.

Apparatus for the cultivation of micro-organisms and for carrying out aerobic fermentation. J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 309,207, 7.11.27).—A pitched nutrient solution is allowed to flow slowly down a labyrinthial arrangement of slightly inclined flat surfaces which may be provided with means for regulating the rate of flow. It is preferable to dispose the flat surfaces in a closed vessel through which a current of purified oxygen, air, or other gas containing oxygen is caused to pass on the counterflow principle.

C. RANKEN.

Distilling apparatus. J. McDougall (B.P. 309,317, 18.4.28).—In an apparatus of the Coffey type, the vapour coming from the analyser is cooled either before it enters the rectifier or by water-coils in the lower part of the rectifier. In this way the extra heat which cannot be taken up by the incoming wash in the coils of the rectifier is extracted from the spirit vapour, thus enabling liquors of a spirit strength higher than 5% of absolute alcohol to be treated without loss of spirit. In addition, the wash after leaving the rectifier, where it has been heated by its own vapour, is heated to its b.p. or above in a heater before being passed on to the analyser. The heater takes the form of a pipe-coil contained in a chamber through which passes the heating medium consisting of the spent liquor issuing from the analysing column.

C. RANKEN.

Apparatus for treatment of waste organic mixtures and the like. G. T. REICH (U.S.P. 1,698,171, 8.1.29. Appl., 1.4.24).—Organic waste material, e.g., distillery slop from fermentation of molasses, containing nitrogen and/or potassium, is fed with ammonium sulphate into an agitating vessel and treated with sulphuric acid. The mixture, which may be freed from part of its potassium sulphate by centrifuging, passes into retorts enclosed completely in gas-combustion

chambers, and the dried mixture is discharged and conveyed to chambers where hydrochloric acid is removed by agitation with further sulphuric acid and the treated residue, containing, e.g., 6% of nitrogen and 6.5% of potash, is discharged. R. BRIGHTMAN.

Production of yeast. S. SAK (B.P. 308,324, 20.9.27).—During the production of yeast, with or without the formation of alcohol and with the addition of nutrients to the fermenting wort, the relation between the quantity by wt. of yeast and the quantity by wt. of at least one of the nutrients, e.g., sugar, is kept constant by the controlled withdrawal or addition of yeast to the fermenting wort. C. RANKEN.

Manufacture of durable yeast. E. C. R. MARKS. FROM NORDDEUTS. HEFEIND. A.-G. (B.P. 308,471, 21.3.28).—Yeast, prior to being dried, is deprived as much as possible of its content of protein by being kept in motion for 6—12 hrs. in a solution of alcohol, containing 0.25—1% of alcohol by vol. The alcoholic solution may consist of the alcohol-containing wort which remains during the production of pressed yeast. C. RANKEN.

Cheese (U.S.P. 1,704,458).—See XIX.

XIX.—FOODS.

Determination of maize flour in mixtures with wheat flour. A. V. MILSKI (Ukraine Chem. J., 1928, 3, [Tech.], 183—208).—Wheat flour has a protein content of 11.12—13.63% of which 45.3—55.2% is represented by gliadin, whilst 8.93—12.07% of maize flour is represented by proteins, of which 56.8—62.0% is zein. The tryptophan content of gliadin is 1.01—1.22%, but this constituent of wheat flour cannot be made use of in the determination of admixed maize, as the colorimetric method of Tillmans and Alt (A., 1926, 189) is not applicable to such mixtures. The solubilities of wheat and of maize proteins are approximately equal in 80% alcohol, whilst in 96% alcohol maize protein is about 14 times more soluble than that of wheat. The following method based on this difference is proposed. The flour (37.5 g.) is extracted under reflux with 150 c.c. of 96% alcohol during 1 hr., alcohol is then added to make the volume up to 250 c.c., and the nitrogen content of the filtrate is determined by Kjeldahl's method. The percentage content, X , of maize flour in the mixture is given by $X = (A - 26)/5.84$, where A = mg. of nitrogen in the extract from 100 g. of air-dry flour. The experimental error in the analysis of mixtures containing 3—30% of maize flour is 5.5—3.1%.

R. TRUSZKOWSKI.

Vitamins in canned foods. VII. **Effect of storage on vitamin value of canned spinach.** W. H. EDDY, E. F. KOHMAN, and N. HALLIDAY (Ind. Eng. Chem., 1929, 21, 347).—Little change in the vitamin content of canned spinach was found after 3 years' storage.

F. R. ENNOS.

Cacao butter. KAUFMANN; AUFRECHT. **Anti-rachitic vitamins in milk and butter.** COWARD.—See XII. **Forage feeds.** SCOTT.—See XVI. **Sugars in marmalade.** LIPPICH.—See XVII.

PATENTS.

Improving the keeping quality of margarine, butter, etc. A. BOEHRINGER (B.P. 308,405, 26.1.28).—

Alkali lactates, or buffer solutions containing lactic acid and lactates, in which the proportion of volatile acids does not exceed 0.5% and is preferably below 0.25%, and which are free or practically free from iron, are added to margarine etc. L. A. COLES.

Manufacture of cheese. J. R. BREHM (U.S.P. 1,704,458, 5.3.29. Appl., 6.4.26).—For fermenting a mixture of milk and hydrolised protein decomposition product a pure culture of *Lactobacillus acidophilus* is used, which has been repeatedly grown in milk containing 1% of the original dried protein. The organism is grown until it reaches its maximum rate of growth at 37°, and is then inoculated to a new medium each time the milk is coagulated, or daily if the coagulation period is less than one day. F. G. CLARKE.

Treatment of foods. J. W. D. CHESNEY, Assr. to SOLAR RES. CORP. (U.S.P. 1,704,173, 5.3.29. Appl., 30.4.28).—Sun rays of wave-lengths of 2900–3700 Å. are concentrated and directed upon food to increase its antirachitic properties. F. G. CLARKE.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Diethyl ether. I. Products of autoxidation. H. KING (J.C.S., 1929, 738–750).—Titration of deteriorated ether with 0.1*N*-potassium permanganate in presence of *N*-sulphuric acid and manganese sulphate determines the amount of free and labile hydrogen peroxide present; in four cases the amount varied from 2.8 to 5.6 g. per litre. Distillation of such an ether through an efficient column causes no loss of peroxide. Qualitative detection of hydrogen peroxide in ether is carried out by shaking a mixture of ether (9 c.c.), water (1 c.c.), 2*N*-sulphuric acid (1 drop), and 0.5*N*-potassium dichromate (1 drop) for 30 sec.; the ethereal layer shows a faint blue colour at a dilution of 1 in 400,000. The test is untrustworthy in presence of acetaldehyde. Treatment of an ethereal solution of hydrogen peroxide with acetaldehyde and subsequent evaporation in a vacuum yields di- α -hydroxyethyl peroxide (I), which could not be obtained crystalline. When this is decomposed by gentle boiling with dilute sulphuric acid, the evolved acetaldehyde absorbed in standard sodium hydrogen sulphite solution and determined by back-titration with iodine, and the residual hydrogen peroxide redetermined by permanganate, a ratio of 1.82–1.91 : 1 for aldehyde : peroxide is found. Further treatment of I with acetaldehyde and subsequent redetermination of the ratio gives values of 2.1–2.2 : 1. Similar determinations of the aldehyde-peroxide ratio in deteriorated ethers gives values of 1.13–1.24 : 1. These are not materially affected by previous washing with sodium hydrogen carbonate solution to remove formic and acetic acids. The permanganate titration on the distillation residue of washed and unwashed ether is usually larger than direct titration by about 10%, indicating the presence of a small amount of a more stable peroxide. Treatment of deteriorated ether with acetaldehyde causes an increase of the aldehyde-peroxide ratio to 2.1–2.2 : 1. This fixation of acetaldehyde by the peroxide present in ether is consistent with Wieland and Winkler's view of an acetaldehyde-hydrogen peroxide combination (A., 1923, i, 650), but does not agree with Clover's

formulation (A., 1922, i, 619). It is suggested that the peroxide present is essentially α -hydroxyethyl hydrogen peroxide and not a mixture of I and hydrogen peroxide (cf. Wieland and Winkler, *loc. cit.*), since three-fold extraction of the ether with one tenth of its volume of water gives an extract with an aldehyde-peroxide ratio of 1.43 : 1. The distribution ratios of acetaldehyde, I, and 1% hydrogen peroxide, between ether and water are 1 : 2.45, 1.5 : 1 (cf. Clover, *loc. cit.*), and 1 : 16 (Clover, *loc. cit.*), respectively. Formaldehyde could not be detected among the decomposition products of the ether peroxide when distilled with *N*-sulphuric acid; *s*-dihydroxydimethyl peroxide (II) affords formaldehyde under the same conditions. Aqueous solutions of I and II give a blue coloration with a 10% alcoholic tincture of guaiacum or a 1% solution of benzidine in 50% alcohol, in presence of peroxidase. A mixture of acetaldehyde or formaldehyde and aqueous hydrogen peroxide gives a direct reaction with benzidine, but not with guaiacum. The reaction becomes negative when the solutions are kept for 2–3 days (cf. Wieland and Winkler, *loc. cit.*), indicating that the coloration is due to an intermediate peroxide consisting of molecular proportions of the aldehyde and hydrogen peroxide. A positive test is given by an aqueous solution of the peroxide from deteriorated ether; heptaldehyde and benzaldehyde suspended in water also give positive tests owing to their peroxide content. Butyl, isooctyl, and amyl alcohols show a reaction with benzidine or guaiacum in presence of peroxidase, but methyl, ethyl, isopropyl, and benzyl alcohols do not. In many of these tests a drop of a solution of a ferrous or ferric salt can replace the enzyme.

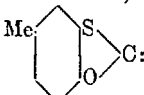
H. BURTON.

Examination of tablets of acetylsalicylic acid containing magnesia. A. HOFFMANN (Dansk Tidsskr. Farm., 1929, 3, 81–102).—The tablets should be tested for their content of acetylsalicylic acid and for salicylic acid produced by hydrolysis of this. Methods available for the former determination are: (a) warming with 0.5*N*-sodium hydroxide, followed by rapid titration of the cooled liquid with 0.1*N*-hydrochloric acid, using alizarin-yellow as indicator; (b) extraction of the acetylsalicylic acid with ether, the dissolved acid being hydrolysed by warming with 0.5*N*-sodium hydroxide after removal of the ether, and the excess of alkali titrated with 0.5*N*-hydrochloric acid, using phenolphthalein as indicator; (c) the material is warmed with sodium hydroxide, excess of hydrochloric acid added, and the whole warmed for 10 min., any separated salicylic acid being redissolved by the addition of alcohol and determined in an aliquot part of the solution by Kolt-hoff's bromate method (A., 1921, ii, 466), with the modification that the liquid is allowed to remain 30 min. before addition of the potassium iodide. To determine free salicylic acid the tablet is treated with 1 c.c. of 4*N*-sulphuric acid, 10 c.c. of a mixture of ether and pentane (1 : 1) are added, and the whole is shaken. The organic layer is separated, and 5 c.c. of this are evaporated. The residue is dissolved in 20 c.c. of 25% alcohol, and after addition of 0.5 c.c. of 2% ferric chloride solution the colour produced is compared with that of similar standard solutions of the acid.

H. F. HARWOOD.

Evaluation of liquor cresoli saponatus. H. KAISER (Apoth. Ztg., 1928, 68, 626—627; Chem. Zentr., 1928, ii, 2047).—A simple procedure based on direct distillation is described. A. A. ELDRIDGE.

Synthetic drugs. IV. Synthesis of sulphur compounds. V. Preparation of benzyl compounds. H. P. KAUFMANN (Arch. Pharm., 1929, 267, 192—211, 212—219; cf. A., 1927, 155, 663, 1083).—IV. [With E. WEBER.] Kaufmann and Oehring's method of "rhodanation" (A., 1926, 392) is improved and its usefulness in preparing sulphur derivatives discussed. The solvent used is methyl alcohol or a volatile ester; it is saturated with sodium bromide or ammonium chloride according as the halogen to be added is bromine or chlorine; and a great excess of thiocyanate is added. The use of such solvents allows the reaction to be performed at a low temperature so that the thiocyanoderivative can be isolated in cases where formerly the thiazole was produced (cf. Kaufmann, A., 1928, 653). *p*-Thiocyananiline is obtained in excellent yield either by adding bromine to a solution of aniline and an excess of sodium thiocyanate in methyl alcohol saturated with sodium bromide, or by passing chlorine into a similar solution containing ammonium thiocyanate and ammonium chloride. *p*-Thiocyanacetanilide cannot be directly obtained from acetanilide in this way. Phenacetin likewise remains unattached, but *p*-phenetidine is smoothly "rhodanated" in methyl alcohol to the 2-thiocyano-derivative, m.p. 67—68°, which, however, is rapidly converted at about its m.p. into 2-amino-6-ethoxybenzthiazole, and yields the diacetyl derivative, m.p. 245°, of this compound on acetylation. 2-Thiocyanop-phenetidine gives 2 : 2'-diamino-5 : 5'-diethoxydiphenyl disulphide, m.p. 101°, when cautiously warmed with dilute alcoholic sodium carbonate solution. The following thiocyanoderivatives, prepared by direct "rhodanation," are also described: formyl-*p*-thiocyanophenylhydrazine, m.p. 132°, and the analogous acetyl, m.p. 171°; benzoyl, m.p. 164°; and phthaloyl compounds, m.p. 213°; acetyl-*pp'*-dithiocyanodiphenylhydrazine, m.p. 160°, and the analogous benzoyl compound, m.p. 160°. 4-Thiocyano-1-hydroxyquinoline (4-thiocyanocarbostyryl) has m.p. 141°; 4-thiocyano-8-hydroxyquinoline has m.p. 134°. Thymol yields 4-thiocyano-6-isopropyl-*m*-cresol, already described by Kaufmann and Gärtner (A., 1924, i, 840), which is a potent disinfectant. *o*- and *m*-Cresols yield the 5-thiocyano- (?-6-cyano- from *m*-cresol) derivatives, m.p. 71° and 76°, respectively. *p*-Cresol yields the

compound  m.p. 105°, hydrolysed by alcoholic hydrochloric acid to the cyclic carbonate of the mercaptocresol, m.p. 83°.

V. [With O. RITTER.] The pharmacological properties of compounds containing benzyl groups are exhaustively discussed, with copious literature references, particularly to the developments from Macht's work on benzyl alcohol. The preparation of the following compounds is described. 4-Dibenzylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone, m.p. 102°; *N*- α -bromoisovaleryl-*N'*-benzylcarbamide, m.p. 134° (from benzylcarbamide and α -bromoisovaleryl bromide); *pp'*-diethoxydiphenyl-

phenylethenylamidine, m.p. 113°, obtained by heating phenylacet-*p*-phenetidine, m.p. 128.5—130°, with phosphorus oxychloride in benzene solution; *oo'*-dibenzylphenolphthalein, m.p. 175° (diacetyl derivative, m.p. 134°), prepared in the usual way from *o*-benzylphenol; *pp'*-dibenzylidiphenylphthalide, m.p. 123°; *p*-thiocyano-N-benzylaniline, m.p. 78°, by direct "rhodanation," yielding on treatment with alcoholic potassium hydroxide the corresponding disulphide, m.p. 92°; *p*-thiocyano-N-benzylmethylaniline, m.p. 63° (disulphide, m.p. 86—87°); *p*-thiocyano-N-benzylethylaniline, m.p. 54° (disulphide, m.p. 76°). W. A. SILVESTER.

Variation in activity of different samples of strophanthin. F. WOKES (Quart. J. Pharm., 1928, 1, 513—516).—Nine commercial samples of strophanthin were compared with the official ouabain of the United States Pharmacopœia X by the cat method. The average potency of eight samples was 47.3% of the standard; seven samples were within 25% of this average. F. C. HAFOLD.

Detection of holocaine. L. EKKERT (Pharm. Zentr., 1929, 79, 120).—Holocaine may be distinguished from novocaine, stovaine, and other related compounds by the different colorations given with resorcinol and sulphuric acid. S. I. LEVY.

Commercial utilisation of Java citronella oil. B. O'DONOGHUE, J. DRUM, and H. RYAN (Sci. Proc. Roy. Dubl. Soc., 1928, 19, 113—120).—The more valuable constituents of Java citronella oil (citronellal, geraniol, and citronellol) may be obtained as follows: the crude oil (d_{4}^{15} 0.8918, α_D $-2^\circ 48'$, acetyltable content 83.3%, aldehyde content 45%) is treated with 30% sodium bisulphite solution for 4 hrs. at 0°, the citronellal bisulphite, which contains 1—2% of the double bisulphite, is collected, washed with ether, decomposed with dilute alkali, and the citronellal steam-distilled. The aldehyde is further purified by distillation in a vacuum, but probably contains some isopulegol; the product has b.p. 205—206°, $n_D^{18.3}$ 1.472, $d_{4}^{19.5}$ 0.8845, α_D $+2^\circ 24'$, and the yield is about 43%. The oil remaining after removal of the citronellal is hydrolysed with alcoholic potassium hydroxide for 1 hr., and geraniol is separated as the double compound with calcium chloride; after washing with light petroleum, the compound is decomposed with water, and the alcohol, b.p. 114—115°/12 mm., $n_D^{18.3}$ 1.4768, $d_{4}^{19.5}$ 0.8811, α_D 0°, is obtained by steam-distillation. The residual oil still contains some geraniol together with citronellol and terpenes; attempts to separate the citronellol as the phthalate were unsuccessful. Citronellol, containing some isopulegol, is obtained by reduction of citronellal with iron and acetic acid at 15°; the use of 5% sodium amalgam in faintly acid (acetic acid) solution below 5° is preferable and gives a product of b.p. 110—112°/10 mm., $n_D^{18.3}$ 1.468, $d_{4}^{19.5}$ 0.8542, α_D $+2^\circ 49'$, in 70% yield; aluminium amalgam in aqueous-alcoholic solution at 60—70° also affords citronellol in 70—75% yield. Preliminary experiments on electrolytic reduction of citronellal indicate that with lead electrodes and sodium hydroxide solution as electrolyte, using a current of 8—9 amp. for 10 hrs. at 50—60°, an 80% yield of citronellol can be obtained readily. The preparation of geranyl acetate,

b.p. 127—129°/15 mm., $n_D^{18.3}$ 1.4637, $d_{15}^{19.5}$ 0.907, α_D 0°, citronellyl acetate, b.p. 119—121°/15 mm., $n_D^{18.3}$ 1.457, $d_{15}^{19.5}$ 0.9035, α_D +0° 30', and of geranyl methyl ether are described. C. W. SHOPPEE.

Detection of small quantities of alcohol in cassia oil. SCHIMMEL & Co. (Deut. Parfümerieztg., 1928, 14, 405—407; Chem. Zentr., 1928, ii, 2079).—The flash point is determined in a Pensky-Martens apparatus. Tables give the flash points of cinnamaldehyde and cassia oil with varying amounts of added alcohol.

A. A. ELDRIDGE.

Determination of ethereal oils in gentian spirits. T. VON FELLEBERG (Mitt. Lebensmittelunters. Hyg., 1928, 19, 242—251; Chem. Zentr., 1928, ii, 2079).—The spirit (5 c.c.), diluted to 30% of alcohol, is shaken with light petroleum (b.p. 29—45°, 5 c.c.), the extract is heated with 15*N*-sulphuric acid (1 c.c.) at 100° for 1 min., and the brown residual liquid is compared in a microcolorimeter with a specified (inorganic) comparison solution.

A. A. ELDRIDGE.

Pine-stump oil. SCHIMMEL & Co. (Ber. Schimmel, 1928, 100; Chem. Zentr., 1928, ii, 2077).—The oil, obtained by distillation in steam, had d_{15}^{15} 0.8599, α_D +22° 43', n_D^{20} 1.46993; it contains *d*- α -pinene and β -pinene (35—40%) and 40—50% of Δ^3 -carene.

A. A. ELDRIDGE.

Dill top oil. SCHIMMEL & Co. (Ber. Schimmel, 1928, 23; Chem. Zentr., 1928, ii, 2077).—The oil has d_{15}^{15} 0.8752, α_D +108° 35', n_D^{20} 1.47691. The chief constituent is phellandrene; carvone is apparently absent.

A. A. ELDRIDGE.

Cistus leaf oil. SCHIMMEL & Co. (Ber. Schimmel, 1928, 16; Chem. Zentr., 1928, ii, 2076—2077).—The oil of Spanish cistus leaves had d_{15}^{20} 0.9560, n_D^{20} 1.49592, acid value 28.0, ester value 42.9. A. A. ELDRIDGE.

Botha-grass oil. SCHIMMEL & Co. (Ber. Schimmel, 1928, 8; Chem. Zentr., 1928, ii, 2076).—Two samples of the oil (apparently identical with Rao, Sudborough, and Watson's bode-grass oil) had d_{15}^{15} 0.9321, 0.9303; α_D -18° 28', -20° 45'; n_D^{20} 1.48693, 1.48587; acid value 2.2, 2.2; ester value 46.7, 40.1; ester value after acetylation 112.9, 110.1. A. A. ELDRIDGE.

[Oil of] Carline thistle. SCHIMMEL & Co. (Ber. Schimmel, 1928, 24; Chem. Zentr., 1928, ii, 2077).—Oil from the root of *Carlina acaulis*, L., has d_{15}^{15} 1.0334, α_D -5° 22', n_D^{20} 1.56548, acid value 1.1, ester value 7.5, ester value after acetylation 24.3.

A. A. ELDRIDGE.

Oil of Eucalyptus dives. SCHIMMEL & Co. (Ber. Schimmel, 1928, 46; Chem. Zentr., 1928, ii, 2077).—The following constituents were isolated: 4-terpineol (b.p. 208—212°/760 mm.; 79—81°/5—6 mm.; d_{15}^{15} 0.9275, α_D -22° 30'; n_D^{20} 1.47740; α -naphthylurethane, m.p. 105°, 1:4-terpin, m.p. 137—138°. Oxidation with permanganate at a low temperature gave the glycerol of 4-terpineol, m.p. 113—115°. The sublimate from this, m.p. 129°, when distilled in steam with hydrochloric acid, gave carvenone (semicarbazone, m.p. 197—198°) and *p*-cymene. Oxidation with alkaline permanganate gave a mixture of acids, m.p. 190—195°, passing by loss of water into the dilactone, m.p. 64—66°; the acid mixture contained the dihydroxy-

dicarboxylic acid $C_{10}H_{16}O_6$, m.p. 200°. The new alcohol is converted by hydrogen chloride into terpinene dihydrochloride, m.p. 51°. Piperitone is not present.

A. A. ELDRIDGE.

Oil of Dacrydium Franklinii, Hook. SCHIMMEL & Co. (Ber. Schimmel, 1928, 23; Chem. Zentr., 1928, ii, 2077).—The oil of the Huon pine had d_{15}^{15} 1.0406—1.0470, α_D +1° 21' to -1° 16', n_D^{20} 1.53037—1.53446.

A. A. ELDRIDGE.

Oil of Cryptomeria japonica. SCHIMMEL & Co. (Ber. Schimmel, 1928, 23; Chem. Zentr., 1928, ii, 2077).—Oil from the root wood of the Japanese cedar had b.p. 270—300°, d_{15}^{15} 0.9325—0.9431, α_D -8° 20' to -20° 49', n_D^{20} 1.50910—1.51075, acid value 0—0.3, ester value 0.9—9.3, ester value after acetylation 36.4—57.9.

A. A. ELDRIDGE.

Terpenes and terpene alcohols. I. Vapour pressure-temperature relationships. O. A. PICKETT and J. M. PETERSON (Ind. Eng. Chem., 1929, 21, 325—326).—Data are given for α - and β -pinene, dipentene, terpinolene, fenchyl alcohol, and α -terpineol, determinations being made by a modification of the Ramsay and Young method.

F. R. ENNOS.

Thiocyanogen value of fats. KAUFMANN and BROCKE.—See XII. Vapour pressure of nicotine. YOUNG and NELSON.—See XXIII.

PATENTS.

Preservation of ether. E. MALLINCKRODT, JUN., and H. V. FARR, ASSRS. to MALLINCKRODT CHEM. WORKS (U.S.P. 1,697,320, 1.1.29. Appl., 17.11.27).—The tinned interior surface of the container is oxidised, e.g., with potassium permanganate, or coated with varnishes insoluble in ether or with lead or other metal which does not catalyse the oxidation of ether.

R. BRIGHTMAN.

Medicinal compound. C. N. ANDERSON, ASSR. to PARKE, DAVIS & Co. (U.S.P. 1,703,377, 26.2.29. Appl., 6.2.28).—Tetrachloroethylene in anthelmintic capsules for administration to animals is stabilised against actinic rays by incorporation with an oil-soluble red dye, e.g., Oil Soluble Red O.

R. BRIGHTMAN.

Purification of organic compounds [chlorophenarsazine]. D. B. BRADNER (U.S.P. 1,696,539, 25.12.28. Appl., 25.6.25).—Crude chlorophenarsazine, made for example from diphenylamine and arsenic trichloride at 160—250°, is washed with water only. C. HOLLINS.

Manufacture of soluble plant extracts. F. GOEDECKE (B.P. 308,403, 24.1.28).—Inositolhexaphosphoric acid and vitamins are extracted together from comminuted plants by means of very dilute hydrochloric acid, and the former are precipitated by addition of calcium carbonate as neutral salts, from which they are recovered by means of (oxalic) acid. The vitamin solution may be mixed with the pure inositolhexaphosphoric acid in solution and concentrated in a vacuum for use in foodstuffs.

C. HOLLINS.

Basic quinine solution. A. LIEBRECHT, ASSR. to CHEM.-PHARM. A.-G. BAD HOMBURG (U.S.P. 1,692,900, 27.11.28. Appl., 17.1.28. Ger., 4.11.25).—Quinine base is dissolved in solutions of quinine hydrochloride, formate, lactate, etc., preferably with the addition of

solubilisers such as antipyrine, isovaleramide. In presence of antipyrine quinine base is soluble in water. The solutions are suitable for injections. C. HOLLINS.

New emetine salts. CHEM. FABR. VORM. SANDOZ (B.P. 283,533, 12.1.28. Switz., 12.1.27).—Emetine salts of bile acids, especially cholic and deoxycholic acids, are described. C. HOLLINS.

Manufacture of vaccines. W. WEICHLEIN (B.P. 282,780, 24.12.27. Ger., 24.12.26).—Immunising sera of high strength are produced by combining the virus or germ culture in physiological salt solution with aniline dyes (magenta, brilliant-green, etc.) at 37° during 24–48 hrs. Examples are vaccines for swine erysipelas and for chicken cholera. C. HOLLINS.

Manufacture of soporifics containing at least one alkynyl group. M. BOCKMÜHL, R. SCHWABE, and G. EHRHART, Assrs. to WINTHROP CHEM. CO. (U.S.P. 1,682,062, 28.8.28. Appl., 15.12.26. Ger., 11.6.25).—See B.P. 285,598; B., 1928, 389.

Manufacture of complex antimony compounds. H. HAHN, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,703,732, 26.2.29. Appl., 28.11.27. Ger., 22.4.26).—See B.P. 288,370; B., 1928, 427.

Production of pyridine derivatives [3:5-di-iodo-2-hydroxypyridine]. M. DOHRN and R. DIRKSEN, Assrs. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,706,775, 26.3.29. Appl., 23.2.28. Ger., 31.3.27).—See B.P. 288,133; B., 1929, 37.

Alkyl- and aralkyl-resorcinolcarboxylic acids (U.S.P. 1,697,332).—See III. Curative creams (B.P. 309,012).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic action from metals, woods, etc. J. G. STRACHAN (J. Roy. Tech. Coll., Glasgow, 1929, 2, [1], 20–29).—In close proximity to a photographic plate zinc produces a blurred image in the dark after 2 hrs.; the intensity of the image is increased by highly polishing the zinc or by amalgamating it (although mercury alone produces no effect) and is diminished by rasping the surface. Boiled linseed oil, alcohol, and benzene have a similar action to zinc, but light petroleum is without action (cf. Russell, B., 1897, 825; 1904, 998).

A. R. POWELL.

Determination of ripening silver in photographic emulsions. F. WEGERT and F. LÜHR (Z. Elektrochem., 1929, 35, 209).—Polemical against Kieser (B., 1929, 227).

H. T. S. BRITTON.

PATENTS.

Production of photographic colour prints and transparencies. J. C. CARTER (U.S.P. 1,706,107, 8.1.27).—Positives are printed from the separate colour-impression negatives on to collodion emulsion plates, and these are dyed their respective colours, stripped, and superposed on a suitable base to give the colour print. J. W. GLASSETT.

Making of negatives. W. HEINECKE (U.S.P. 1,700,262, 29.1.29. Appl., 6.5.27).—Impressions are made from engraved plates or the like on a sensitised film, which is then developed in a bath containing

sodium sulphite, formaldehyde, and quinol, rinsed, dipped into an alkali bicarbonate solution to remove the ink used in making the impression, and finally fixed. A. R. POWELL.

XXII.—EXPLOSIVES; MATCHES.

Specifications for cellulose for use in the manufacture of smokeless powder. F. OLSEN (Ind. Eng. Chem., 1929, 21, 354–356).—The current specifications for cellulose to be used in the preparation of nitro-cellulose have been drawn up with particular reference to cotton linters. There are, however, other sources of cellulose which, while not complying with these specifications—particularly that of the limiting amount of alkali-soluble material—yet yield an equally stable product; other trustworthy criteria would seem to be called for which are adapted to the particular form of cellulose concerned. F. R. ENNOS.

Determination of diphenylamine and diphenylnitrosoamine in the presence of their derivatives. H. RYAN, J. KEANE, and J. DUNNE (Sci. Proc. Roy. Dubl. Soc., 1928, 19, 85–100).—Methods suggested by Berger and by Dreger (A., 1909, ii, 708) for determining the “safe-period” of nitrocellulose powders stabilised by diphenylamine (I) are discussed critically. The present authors claim that accurate results for the total reserve of stability in the powder may be obtained by the following method: after extraction with alcohol, the extractive is reduced with stannous chloride and hydrochloric acid in alcoholic solution, the alcohol removed, and the diphenylamine distilled in steam. The distillate is extracted with chloroform, and diphenylamine is determined volumetrically with bromine. Diphenylnitrosoamine (II) is thereby quantitatively converted into (I), and nitro-derivatives of (I) into bases the hydrochlorides of which are non-volatile in steam. In this method it is found that bromine water cannot be used, as the apparent absorption varies with the light conditions and the concentration of the reagent. These difficulties may be overcome by using a chloroform solution of bromine at 15° in indirect sunlight, when (I) affords a tetrabromide, (II) a dibromide, and 4-nitrodiphenylamine (III) a tribromide; 4-nitrodiphenylnitrosoamine (IV) and 2:4-dinitrodiphenylamine (V) give monobromo-compounds, whilst 2:4'-dinitrodiphenylnitrosoamine (VI), 2:4:2':4'-tetranitrodiphenylamine (VII), and 2:4:6:2':4':6'-hexanitrodiphenylamine (VIII) exhibit no appreciable bromine absorption. When binary mixtures of the above nitro- or nitroso-derivatives are brominated in chloroform solution the apparent bromine absorption is greater than the theoretical value by 10%; if this deduction be made the bromination appears to be normal. Figures are also given for the quantitative action of bromine in alcoholic solution (cf. Dreger's method) on I, II, III, and a mixture of VI and 4:4'-dinitrodiphenylnitrosoamine (IX). The volatility in steam at 100° and 250° of several of the above compounds and their mixtures are recorded. It is found that I is wholly, and II partly (66%), distillable in steam at 100° from an acid solution, and that II distills to the extent of 75% from an alkaline solution (cf. Berger's method). C. W. SHOPPEE.

Testing of explosives for use in fiery coal mines (Safety in Mines Res. Board Paper No. 51, 1929, 50 pp.).—Comparative tests with coal gas, light petroleum vapour, benzene vapour, and methane showed that methane was the most suitable for use in the gallery test for explosives. New tests for permitted explosives are proposed which it is hoped will result in more reliable control of coal-mine explosives. S. BINNING.

Explosive gas mixtures. PRAUSNITZ.—See II.
Erosion of guns. GREAVES and others.—See X.

PATENTS.

Explosives for use in mines containing firedamp and like gases. A. SEGAY (B.P. 299,462, 23.10.28. Belg., 27.10.27).—Potassium perchlorate, or nitroglycerin with or without potassium perchlorate, is added to ammonium nitrate, sodium or potassium chloride, and trinitronaphthalene, the last-named being in such amount that it is completely burnt by the oxygen in the ammonium nitrate, or at least by the total oxygen present in the mixture. S. BINNING.

High-explosive composition. F. OLSEN (U.S.P. 1,705,874, 19.3.29. Appl., 30.10.23).—The composition includes ammonium perchlorate, barium nitrate, vegetable ivory meal, ferrosilicon, trinitrotoluene, and graphite. H. ROYAL-DAWSON.

Porous material for receptacles for explosive gases (B.P. 293,697).—See II.

XXIII.—SANITATION; WATER PURIFICATION.

Treatment of town refuse by fermentation in closed chambers (Beccari method). J. BORDAS (Bull. Soc. d'Encour., 1929, 128, 170—172).—Household refuse is sorted and materials such as glass, rags, scrap iron, etc. are removed. The remainder is placed in closed chambers and allowed to ferment. The temperature reaches 75°, which is said to be sufficient to destroy all pathogenic organisms and to prevent loss of nitrogen by inhibiting the action of denitrifying bacteria. The gases evolved are passed through a tower containing clay soil mixed with iron sulphate to fix any ammoniacal products prior to discharge through a tall chimney. The fermentation is complete in 40—60 days, and the final sludge is inoffensive and suitable for use as manure. The process has been used in Italy for several years, its chief disadvantage being the size of the installation required owing to the slow rate of fermentation. C. JEPSON.

Losses caused by heating liquefied sewage solids. H. HEUKELEKIAN (Ind. Eng. Chem., 1929, 21, 324—325).—The resolution of solid sludge into soluble products by fermentation is preliminary to further decomposition into gas. When the liquid portion of the digesting sludge is heated to 100—105°, e.g., in evaporation to dryness, a loss of carbon and nitrogen occurs which may be as high as 25% of the total solids. The products of "liquefaction" are apparently stable and may be disposed of without nuisance if the collection and utilisation of gas is not desired. C. JEPSON.

Control of scum in sewage tanks. A. M. BUSWELL (Ind. Eng. Chem., 1929, 21, 322—323).—If the water layer below the scum or foam, which frequently forms in the digestion of sewage sludge and interferes with

the collection of the gases evolved, be slowly pumped on to the surface thereof, the circulation produced disintegrates the scum or reduces the amount of foam, and once this has been accomplished it is sufficient to carry out the operation for 5—10 min. per day to prevent a recurrence of the trouble. The power costs should not be high. C. JEPSON.

Chlorination relieves ponding on sewage filter beds. F. K. WING and R. C. WILLIAMS (Eng. News-Rec., 1929, 102, 621—622).—The addition of chlorine to the sewage applied to fixed-spray percolating filters, in sufficient quantity to leave a residual amount of 0.5—5.0 p.p.m., proved effective in freeing supply-pipes, nozzles, and bed surface of slimy growths which interfered with the operation of the filter. The addition was continuous for 6 days, and the chlorine cost \$110 per acre, which might have been reduced if the addition had been confined to night periods when the flow was low and the sewage weak. C. JEPSON.

Unusual methods of water purification. M. E. FLENTJE (J. New Eng. Water Works' Assoc., 1929, 43, 38—43).—The New Rochelle Water Co., N.Y., owns four reservoirs, each with its own small watershed, which are operated in pairs to supply the high- and low-service distribution systems. On the high-service system the water flows from one reservoir to another through a concrete flume and then through 300 ft. of 36 in. tile. Turbidity troubles in the first reservoir were remedied by installing an alum feeding plant at the head of the 36 in. pipe line, and utilising the high velocities therein as a mixing agent and the second reservoir as a settlement tank. As the supplies are unfiltered the presence of water-fleas and crustacea must be guarded against; chlorination followed by dechlorination with sodium thiosulphate and soda ash has been found to be effective. At another plant "whiting" has been substituted for soda ash or lime as a means of providing sufficient alkalinity to react with the alum used to aid in the removal of colour, the average increase in hardness resulting from its use being 11 p.p.m. C. JEPSON.

Decolorisation [of water] by storage in clean-bottomed reservoirs. K. R. KENNISON (J. New Eng. Water Works' Assoc., 1929, 43, 60—75).—Observations made at Wachusett reservoir and Suntaug lake, covering periods in several years when the amount of inflow to these reservoirs was low compared with the total volume of water and the effects of other extraneous influences were reduced to a minimum, show that about 20% of the colour present is removed by storage month by month. This reduction follows the usual "die-away curve" the equation for which would probably be stated as $C_t/C = 10^{-kt}$, where C is the initial colour, C_t the colour remaining after t months, and k the decolorisation constant, which in the case of a rate of 20% would be approx. 0.1. C. JEPSON.

Chemical aspects of stream pollution by phenol. E. J. THERIAULT (Ind. Eng. Chem., 1929, 21, 343—346).—By using a simplified procedure based on Gibbs' method (A., 1927, 475, 688), in which the colour produced with 2:6-dibromobenzoquinonechloroimide under certain conditions with regard to pH value etc. is used as a means of determining phenol directly when

the quantity present exceeds 50 pts. per billion (0.005 mg./100 c.c.) or in the products of distillation when the quantity present is smaller, it has been shown that unpleasant tastes become apparent when phenol is present to the extent of 1 pt. per billion and are unmistakable when the concentration reaches 5 pts. Since purely domestic sewage contains 100–500 pts. with an average of 250 pts. per billion of phenol, a 250-times dilution with water is the limit at which the taste should be apparent. Such water would be obviously very highly polluted, but is not unknown as a source of drinking water. A large proportion of the phenol present in raw water is removed during the usual purification processes, and in any case unpleasant tastes should not be ascribed to pollution of faecal origin without careful consideration of all other possible sources thereof (cf. A., 1927, 688; B., 1928, 504).

C. JEPSON.

Determination of traces of phenol in water.

H. BACH (Gas- u. Wasserfach, 1929, 72, 375–377).—A review of methods for determining minute amounts of phenol, including a detailed description of the procedure of Vorce (B., 1925, 861). Essen drinking water, which in December, 1928, tasted strongly of chlorophenol, was tested in this way and contained 0.008 mg. of phenol per litre.

R. H. GRIFFITH.

Determination of small amounts of dissolved oxygen [in water]. F. R. McCrumb and W. R. KENNY (J. Amer. Water Works' Assoc., 1929, 21, 400–407).—The Winkler method of determining dissolved oxygen is unsatisfactory when the quantity present is small, *e.g.*, in the control of the oxygen content of boiler water for corrosion prevention purposes, and is unsuitable for carrying out rapid determination. Manganese with a valency in excess of two produces a colour with *o*-tolidine solution (as used in the determination of chlorine), and as the valency of bivalent manganese is increased in alkaline solution by oxygen, it is proposed to make this the basis of a colorimetric method of determining dissolved oxygen. The sample is siphoned into a stoppered bottle holding exactly 65 c.c., 0.25 c.c. of manganous chloride [$4\text{H}_2\text{O}$] (400 g. per litre) and 0.25 c.c. of potassium hydroxide (700 g. per litre) are added, the bottle is sealed without entrainment of air, and the contents are well shaken, allowed to settle, and shaken again before finally settling. One c.c. of concentrated hydrochloric acid and 1 c.c. of *o*-tolidine solution (10 g. of *o*-tolidine+30 c.c. of hydrochloric acid per litre) are introduced at the bottom of the bottle, which is again sealed and shaken. After 5 min. a portion is withdrawn and its colour compared with previously prepared standards representing from 0.00 to 0.50 c.c. of oxygen per litre. It is essential that the p_H value should not exceed 2.0, and that the tests be carried out between 30° and 40°. Ferric iron in excess of 10 p.p.m. interferes, and it is advisable to prevent the introduction of rust, *e.g.*, when dealing with boiler waters. Consistent results are claimed for very low values of oxygen and fairly accurate ones when dilution is practised.

C. JEPSON.

Vapour pressure of fumigants. IV. Vapour pressure of nicotine. H. D. YOUNG and O. A. NELSON Ind. Eng. Chem., 1929, 21, 321–322; cf. B., 1929,

114).—The vapour pressures were determined below 100° by a method in which a current of air was employed. Values at temperatures from 102.3° to 146.9° were found by a static method. The vapour pressure-temperature curve $\log P = 8.0935 - 2695.5/T$ expresses the experimental results.

H. INGLESON.

Absorption of oxygen by alkaline tannates. FAGER and REYNOLDS.—See VII.

PATENTS.

Treatment of organic refuse. L. BOGGIANI-PICO (B.P. 300,607, 13.9.28. Italy, 16.11.27).—The refuse is fermented in a closed chamber under aerobic conditions produced by a supply of air introduced beneath a false bottom and also distributed throughout the material by means of suitably placed nozzles. The mass is moistened as required by a spray of sewage, and the process regulated by observations of temperature and humidity. The gases evolved are collected and utilised, and the final product obtained after about 30 days is an inoffensive powder which, after the mechanical removal of materials such as glass, bones, metals, etc., may be utilised as a manure. C. JEPSON.

Distillation apparatus [for small amounts of water]. W. J. STONE & Co., LTD., and R. C. B. SWINDALE (B.P. 308,956, 30.12.27).—An evaporating pan is fitted with an easily detachable condensing chamber in the form of a hollow cone which is surrounded on its outside with cooling water and fitted at its base with a frusto-conical sheet of metal so as to form an annular channel in which the condensate formed on the interior surface of the cone may be collected. The annular channel is protected from the heat of the steam by another similar but somewhat larger cone fitted beneath it.

C. JEPSON.

Distillation of water for the make-up feed; in steam supply plants. A.-G. BROWN, BOVERIE & CIE. (B.P. 283,158, 3.1.28. Ger., 5.1.27. Addn. to B.P. 262,396).—Steam, superheated by flue gases from the boiler, is passed through the water to be evaporated to which it loses its superheat. The increased volume of steam produced is again superheated and passed through a second evaporator, where the process is repeated. This is continued until a sufficient volume of water has been evaporated, when a portion is condensed, the condensate being used as feed water for the boiler and the heat thereof to raise the temperature of the water to be evaporated, whilst the remainder is again superheated for use as working steam. C. JEPSON.

Treating wool-washing water. AKTIEBOLAGET SEPARATOR, and W. S. FLIGHT (B.P. 308,634, 22.12.27).—After allowing to settle for a short time in order to remove sand, the liquid is clarified in one or more centrifugal bowls provided with separate outlets for continuous discharge of the water, clay matter, and/or wool grease.

F. R. ENNOS.

Composition to be used as sterilising and germicidal agent. J. H. BUCHANAN, ASST. to AMER. BOTTLERS OF CARBONATED BEVERAGES (U.S.P. 1,702,629, 19.2.29. Appl., 15.11.26).—Addition of 1–3% of sodium chloride to 1–3% sodium hydroxide solutions gives increased sterilising and germicidal efficiency. R. BRIGHTMAN.

Fertiliser etc. from refuse (B.P. 308,328).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 21, 1929.

I.—GENERAL ; PLANT ; MACHINERY.

Modification of Hempel gas pipette. G. H. W. LUCAS (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 79).—To prevent reagents, *e.g.*, alkaline pyrogallol, used in Hempel absorption pipettes from running up the capillary tube of the pipette and entering the measuring burette, using mercury, a small bulb is blown in the capillary tubing, thus causing bubbles to be broken.

C. A. KING.

See also A., May, 528, **Determination of water by distillation** (BOLLER). 533, **Gauge for measurement of high vacua** (STANLEY). **Efficiency of fractional distillation apparatus** (GRIMM). **Apparatus for fractional distillation** (PICHARD). **Vacuum distillation** (BURCH). 613, **Apparatus for filtration and precipitation with subsequent filtration in nitrogen** (GRABE and FREUND).

PATENTS.

Producer furnaces for boiler heating and like purposes. T. R. WOLLASTON (B.P. 305,288, 8.11.27).—A gas producer for low-grade fuels is in communication with a combustion furnace by means of a side passage to which air is also admitted. The producer itself is provided with crusher-like rolls at the bottom for discharging clinker, also with suspended inclined fire-bars adapted to be joggled by the rotation of the rolls.

B. M. VENABLES.

Manufacture of [refractory] linings of furnaces. D. F. CAMPBELL, and ELECTRIC FURNACE CO., LTD. (B.P. 303,574, 16.4.28).—The lining is formed either of bricks or loose material round a core of electrically conducting material the m.p. of which is higher than the fritting point of the lining, the whole being raised to the latter temperature by means of alternating currents in a coil surrounding the furnace, and the core afterwards withdrawn whole. Materials mentioned as suitable for the core are graphite, tungsten, nickel-chrome, and heat-resisting steel.

B. M. VENABLES.

Carrying out chemical reactions and furnace suitable therefore. R. VETTERLEIN (B.P. 309,734, 11.4.28).—A multi-stage furnace built like a Herreshoff roaster is used for carrying out chemical reactions (in a continuous manner) between neutral or acid salts and liquid reagents. The uppermost hearth of the roaster is preferably charged with solid material only, forming a powder seal, the liquid reagent being added to the second hearth through the hollow shaft. Examples of reactions that may be effected are: sodium or potassium sulphate or bisulphate from the chloride and sulphuric acid; the production of soda and purely metallurgical reactions are excluded.

B. M. VENABLES.

Carrying out of reactions at high pressures and temperatures. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 309,057, 2.12.27).—High-pressure reactions, *e.g.*, the destructive hydrogenation of carbonaceous materials, are carried out in a jacketed apparatus, the inner wall of which consists of a special steel highly resistant to chemical action, and the outer wall of another special steel resistant to mechanical stress.

A. B. MANNING.

Apparatus for effecting chemical reactions under pressure. SYNTHETIC AMMONIA & NITRATES, LTD., and A. RULE (B.P. 309,258, 12.2.28).—High-pressure reactions, in particular the hydrogenation of oils, tars, etc., are carried out in a vertical tower, of considerable height in relation to its diameter, the liquid in which is continuously agitated by the passage of the hydrogen which is admitted with the liquid at the bottom of the tower.

A. B. MANNING.

Heating of materials. J. S. MORGAN (B.P. 305,106, 28.7.27).—The finely-divided material to be heated (or cooled) is mixed with heated (or cold) shot, the whole of the heat being supplied (or removed) by the shot. The shot may also be used to effect grinding of the material and to form a gas-tight seal. The process may be worked intermittently, or with more than one stage of heating. The separation of the shot at the end (or between stages) of the process may be effected by screening, by magnetic means, or pneumatically.

B. M. VENABLES.

Heat interchanger. F. H. WILLCOX and J. C. HAYES, JUN., Assrs. to FREYN ENGINEERING CO. (U.S.P. 1,707,777, 2.4.29. Appl., 11.4.27).—A heat interchanger has vertical tubes for the gas to be heated, and is provided with inlet and outlet conduits and headers so positioned and shaped as to distribute the gas evenly. Flushing jets are also placed in the delivery header for flushing the tubes.

B. M. VENABLES.

Heat exchanger. J. P. RATHBUN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,708,031, 9.4.29. Appl., 28.10.27).—A baffle within a heat exchanger of the shell and nest-of-tubes type is provided with a liquid seal between it and the shell.

B. M. VENABLES.

Heat-exchanging apparatus. W. E. KOCHS & CO., LTD. (B.P. 309,798, 21.7.28).—Heat-transfer tubes are constructed with a section having the shape of a falling drop or pear, or other stream-line section, so that the external fluid stream will remain in contact with the whole surface of the tubes. A preferred arrangement is vertical with the gas approaching the wide end of the section. The rows of tubes may be staggered or not, as desired.

B. M. VENABLES.

Drying apparatus. I. G. FARBERIND A.-G. (B.P. 293,028, 19.6.28. Ger., 30.6.27).—A Huillard apparatus (in which a band of fine-mesh wire netting picks up a paste and conveys it zig-zag fashion against a stream of heated air) has its rollers mounted on springs, so that they can yield in two directions, against the pull of the band and axially. B. M. VENABLES.

Oven for drying or baking briquettes and other moulded products. T. NAGEL (B.P. 283,123, 3.1.28. U.S., 3.1.27).—The briquettes are allowed to slide down a succession of sloping chutes, disposed at an angle approximately equal to the angle of repose of the material; each chute terminates close to the commencement of the next, but the direction changes at each step, so that the briquettes are turned over at each transfer. They are heated by an upward current of gases, and cooled by other gases in the lower chutes. B. M. VENABLES.

Apparatus for treating and drying coal, cereals, chemical substances, etc. J. H. GOURLEY (B.P. 308,548, 19.9.28).—In apparatus comprising a number of rotating drums or retarders in staggered vertical relation, around which heated air or other drying agent zig-zags upwardly countercurrent to the falling material, the drums are heated internally, and balanced flaps are provided extending from the nearest side of the casing, the flaps acting both as scrapers for sticky material and as baffles preventing flow of the drying gas up that side of the casing. The inlet and outlet for the material are provided with valves. B. M. VENABLES.

Drying and impregnating *in vacuo* particularly applicable to manufacture of insulating materials. H. GRIFFITHS and E. PASSBURG (B.P. 308,641, 23.12.27).—The major portion of the moisture is removed under a vacuum of the same order as the vapour tension of water at ordinary condenser temperatures (*e.g.*, 24 mm. of mercury for 22°), and the drying is finished by increasing the vacuum, which may be effected by removing the moisture from the internal atmosphere either by deposition on a condensing surface maintained at about 0°, by absorption in a cooled liquid such as brine, or by dehydrating substances such as silica gel. Alternatively, the high vacuum may be obtained simply by the use of efficient vacuum pumps, *e.g.*, of the steam ejector type. In either case impregnation is afterwards effected under pressure. B. M. VENABLES.

Heat-insulating material. HENLEY'S TYRE & RUBBER CO., LTD., and J. TRAXLER (B.P. 309,662, 6.2.28).—Articles such as pipes and receptacles are coated before assembly with a rubber composition capable of becoming porous when heated under pressure, *e.g.*, rubber 47%, granulated cork 35%, sulphur 3%, barytes 14½%, accelerator ½%. Vulcanisation is effected preferably after the material has been applied. D. F. TWISS.

Disintegrators. GEBR. BÜHLER (B.P. 301,896, 12.11.28. Ger., 9.12.27).—Accumulations of material at points between the screen and the enclosing casing are prevented by providing air slots at those points through which strong currents of air are induced by the operation of the disintegrator. B. M. VENABLES.

Crushing mill. A. H. STEBBINS (U.S.P. 1,708,195, 9.4.29. Appl., 15.12.27).—The ground material from a mill in an air-borne system is drawn through a pipe horizontally then upwardly, and the elbow in the pipe is provided with a number of apertures which admit air and break up any lodgments of dust. B. M. VENABLES.

Gyratory crusher. T. C. COOKE (U.S.P. 1,706,975, 26.3.29. Appl., 27.10.27).—The driven eccentric ring is provided with anti-friction members [roller bearings] both outwardly and inwardly. B. M. VENABLES.

Rotary [gyratory] crusher heads or the like. J. A. HETU (B.P. 305,398, 17.3.28).—The mantle of a gyratory crusher is provided with a conical interior and fits on a conical part of the driving shaft, with or without the interposition of a conical bush. On any slip taking place the mantle is pressed more tightly down the shaft by means of a pair of cam-faced washers one of which is prevented from rotation with respect to the mantle and to the shaft, respectively. The head is thus self-tightening. B. M. VENABLES.

Formation of agglomerates or aggregates from pulverised materials. J. S. MORGAN (B.P. 305,051, 29.7.27).—Aggregates of solid carbonaceous or other powdered material are formed by causing it and a binder to impinge upon a solid surface. The rapid relative motion necessary may be produced mechanically (centrifugally) or pneumatically, and the solid surface preferably moves slowly to withdraw the agglomerated material. The binding agent may be produced by condensing the vapour or finely-divided spray of oil, and the vapour may also be used as the pneumatic carrying agent. In some cases addition of external binding agent is not necessary. B. M. VENABLES.

Rotary screens or separators. W. and R. H. REID, and W. REID, JUN. (B.P. 303,970, 23.8.28).—The apparatus is in the form of a trommel, the screening surface comprising a number of rollers parallel to the axis. The end spiders (driven by the shaft) are formed into sun-pinions, each roller being provided with a pair of planet-pinions at each end, one of each pair gearing with outer, fixed, internally-toothed rings and the other of each pair with the sun-pinions. The rollers rotate about their own axis, also about the central shaft, but at a slower speed than the end spiders; internal scoops may be provided rotating with shaft and spiders, and therefore having movement relative to the screening surface. B. M. VENABLES.

Dry separation of masses of materials. H. M. SUTTON, W. L. and E. G. STEELE (B.P. 307,526, 10.11.27).—A reciprocating table, having a crinkled perforated deck and perforated tapering riffles, is described, which is pervious to upward air currents. Adjustable "pneumatic skimmers" or horizontal cross-jets of air are provided above the table. B. M. VENABLES.

Separation of solid materials of different sp. gr. G. RAW (B.P. 305,068, 28.10.27 and 31.7.28).—A bed of materials is stratified by pulsating air pressure coming through a pervious table without substantial net movement of the air. The pressure waves are caused to grow synchronously by creating the pulsation at one

end of the wind box and having a wall at the other end accurately positioned to reflect the waves back to the pulsating device, the frequency of the applied pulsations being related by a formula to the time it takes a wave to travel the length of the box. The waves are preferably caused by the simultaneous operation of a number of valves well distributed over the end of the wind box.

B. M. VENABLES.

Centrifugal machines. SHARPLES SPECIALTY CO., Assees. of L. D. JONES (B.P. 299,018, 18.10.28. U.S., 19.10.27).—A small machine suitable for continuous operation, *e.g.*, purifying lubricating oil etc., is constructed with an electric motor on the same axis and below the bowl, and with only two bearings, one of which, nearest the commutator of the motor, is rigid radially and the other merely restrained radially. The bowl is easily detachable for cleaning.

B. M. VENABLES.

Centrifugal machine. J. J. MUNSON (U.S.P. 1,703,404, 26.2.29. Appl., 23.6.26).—A continuously-acting centrifugal machine is constructed with a number of planetary rotating baskets.

B. M. VENABLES.

Centrifugal purifying and dehydrating apparatus. EMPSON CENTRIFUGALS, LTD., and F. A. G. NOEL (B.P. 308,233, 19.12.27).—The heavier matter collected in a centrifugal machine is caused to flow continuously away by constructing the drum with no "abruption" on its inner surfaces, and the cover and sealing cone with smooth surfaces, thus reducing the resistance to upward flow of heavy material, while at the same time the resistance to inward flow of purified lighter liquid is increased.

B. M. VENABLES.

Centrifugal separator drums. F. KRUPP A.-G. (B.P. 299,454, 18.10.28. Ger., 28.10.27).—In a centrifugal separator, the fluid after passing through one or more concentric separating chambers passes inwardly through a filter medium. The filter medium is secured between conical inner and outer members, the former of which is pressed down by screwing means attached to the shaft.

B. M. VENABLES.

Centrifugal fixture. C. G. HAWLEY, Assr. to CENTRIFIX CORP. (U.S.P. 1,700,261, 29.1.29. Appl., 4.5.25).—In a non-rotating centrifugal separator the fluid passes from an inlet at one end of a casing to an outlet at the other, through two sets of whirling elements both operating in the same direction, one of which is longitudinally "tuyèred" and the other radially.

B. M. VENABLES.

Filtering apparatus. J. B. VERNAY (B.P. 297,394, 20.9.28. Ger., 20.9.27).—The filter is of the type in which a number of filter chambers are arranged in a horizontal circle around a vertical shaft. The chambers are overweighted on their radial supporting shafts so that they tip up in succession when permitted to do so and discharge their contents, aided by compressed air. The supporting shafts are hollow, and serve to withdraw filtrate under vacuum, and to supply compressed air to discharge the cakes. Charging of the chambers is done intermittently by a measuring device. To save ground space the filter may be in two stories, the thickened pulp from the upper storey discharging into the lower.

B. M. VENABLES.

Operation and construction of filter presses. M. WILDERMAN (B.P. 307,525, 10.11.27).—The filter plates are made double, so that in addition to the comparatively wide spaces for cake there are formed narrower spaces for filtrate. The plates may be perforated right through and used with cloths or filter paper, or may be made of porous ebomite or other porous material with impervious rim. Several variations of construction are described.

B. M. VENABLES.

Apparatus for straining liquids. R. A. and J. B. BLAKEBOROUGH, and J. LINDSAY (B.P. 309,814, 13.9.28).—A duplex strainer is mounted on a sliding carrier so that while one element is in use the other may be removed for cleaning.

B. M. VENABLES.

Emulsifiers, mixers, etc. C. S. II. SNOW (B.P. 305,711, 10.11.27).—A rotor with vertical axis comprises spokes driving upper and lower annular discs with concentric corrugations and runs with fine clearances between upper and lower fixed annuli, which are maintained the correct distance apart by a sleeve of stiff gauze or perforated metal. The whole is submerged in a container for the material which enters the centre of the rotor from above and below, passes through the sinuous spaces between the discs, and leaves through the gauze. Additional devices are provided, notably scoops for preventing excessive whirlpool action and returning the fluid to the centre. Air may be admitted through a central sleeve surrounding the driving shaft.

B. M. VENABLES.

Concentration or drying of liquids [*e.g.*, aqueous dispersions]. DUNLOP RUBBER CO., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 307,315, 3.11.27).—In a machine for producing simultaneous evaporation and mixing of difficult liquids such as rubber mixes, a comparatively small quantity of liquid is placed in a vessel which oscillates in all directions about a point within the vessel, so that both the exposed wetted surface on the interior vessel and the free surface of the liquid are continually being changed. A jacket for heating or cooling is provided, also inlet and outlet for a drying medium to and from the interior of the vessel.

B. M. VENABLES.

Recovery of volatile liquids. R. RILEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,141, 1.3.28).—Mixtures of dry air with, *e.g.*, acetic acid vapour are scrubbed with a countercurrent of water in quantity sufficient to saturate the air with water vapour.

L. A. COLES.

Treating liquids with gases or vapours. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 309,206, 5.11.27).—Organic liquids (*e.g.*, paraffin wax) are treated with oxidising gases by blowing the mixture upwards through a reaction vessel filled with inert or catalytic fillers; the foam is drawn off at the top and returned to the bottom of the same vessel, or passed through other vessels in series. The material may be heated or cooled during the return journeys.

B. M. VENABLES.

Absorption tower. J. A. CAMPBELL (U.S.P. 1,703,571, 26.2.29. Appl., 27.7.26).—The lowest floor of an absorption tower is supported by a shoulder within the shell casing. The other floors are supported from the lowest by means of cylindrical spacers smaller

than the shell. There is communication from the top space of the apparatus to the annular space between the top spacer and the shell; also means for pressing the top floor, and consequently the other floors and spacers, downwards towards the supporting shoulder.

B. M. VENABLES.

Filter [for gases]. A. JORDAHL and K. G. RUNBACK, ASSRS. to MIDWEST STEEL & SUPPLY CO., INC. (U.S.P. 1,708,065, 9.4.29. Appl., 28.12.23).—A filter for air or gas has a primary filter freely exposed to the atmosphere on the inlet side and situated on one side of a quiescent air chamber; on the other side of the chamber is a secondary filter of larger area and having pockets for the collection of the dust settled out in the quiescent chamber. The larger area of the secondary filter may be obtained by zig-zagging the filter medium.

B. M. VENABLES.

Filters for air and other gas. (SIR) H. AUSTIN (B.P. 307,540, 10.12.27).—The air to be cleaned passes edgewise through a pile of plates that are kept moist by oil or other liquid, the plates being corrugated transversely to the flow of air so that the narrow passages between have abrupt turns at intervals. A preferred construction comprises a pile of annular plates assembled in an inner container with horizontal axis, and with axial inlet and peripheral outlet. An outer casing contains the filter and also a bath of oil which is flushed over the plates by rotating the inner container and plates slowly or intermittently.

B. M. VENABLES.

Rotatable air or gas filter of the plate type. A. SCHIRP (B.P. 307,694, 5.10.28. Ger., 15.3.28).—In a filter comprising a number of closely-spaced plates through which the gas passes edgewise, bundles of the plates are assembled in frames which are wholly or partly of chill-cast metal, and the frames are secured to a chain which passes over small sprockets at each end of its run, so that in the operative position the bundles lie close to each other (grooves and tongues are provided in the frames for sealing purposes), but on passing over the sprockets the frames open out and the plates may be flushed while passing round the lower sprocket.

B. M. VENABLES.

Apparatus for separating solid impurities from air and gases. H. S. POCHIN and C. H. W. CHELTNAM (B.P. 305,693, 4.11.27 and 20.1.28).—The dirty gas is blown by a fan through an outlet tube of rectangular cross-section and bent, and in which the impurities stratify; the outer layer of gas with the heavier particles are taken off by a branch to a cyclone separator, the inner layers pass on to a fabric filter, which may also receive the partially-cleaned gas from the cyclone. The proportion going to each branch may be regulated by a flap within the bent tube, and the centre about which the tube is bent is situated on the inlet side of the fan.

B. M. VENABLES.

Air cleaner. H. R. DAVIES, ASSR. to INDUSTRIAL RES. CORP. (U.S.P. 1,708,122, 9.4.29. Appl., 29.5.29. Renewed 29.8.28).—A number of imperforated, inverted, truncated conical baffle plates are suspended from the closed top (base) of an inverted, truncated-conical casing, to which the air is admitted near the top periphery (outside the largest baffle), and exhausted through

the top centre. An upright cone extends from the bottom (small end) of the casing upwards towards the baffles.

B. M. VENABLES.

Air filtering and like apparatus. O. STOTT, and MATTHEWS & YATES, LTD. (B.P. 303,961, 10.11.27).—A number of cylindrical or other shaped units are mounted in pairs on either side of a dividing wall with apertures through the wall within the units. The end walls of the units are impervious, but the cylindrical walls are of filter medium, so that the air passes inwards through the cylindrical filter surfaces on one side of the wall, through the wall, and out through the filter surfaces in the other side of the wall. The units are easily detachable for cleaning.

B. M. VENABLES.

Production of compressed gases from liquefied gases. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 280,568, 10.11.27. Ger., 15.12.26).—Liquefied gases are evaporated by permitting heat to enter them, a moderate pressure being thus self-generated. The gases are further compressed into storage cylinders by means of a power-driven compressor, the operation of which may be regulated by the intermediate pressure.

B. M. VENABLES.

Insulation of vessels for storing liquefied gases. Adsorption of gases in liquid gas apparatus. Drying of gases in air-liquefying and gas-separation plant. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. [A] 281,305, [B,C] 305,974—5, 23.11.27. Ger., [A] 27.12.26, [B] 24.11.26, [C] 1.12.26).—(A) Highly porous, inorganic insulating material (e.g., kieselguhr) is loosely packed in the space between the pressure vessel and outer case, so loosely that it does not become compacted under its own weight, which effect may be aided by use of layers of slag wool, cellulose fibres, etc. to act as supports. The interstices of the material are filled with still air so chilled by the stored liquefied gas that it becomes a perfect insulator. (B) Attached to a storage vessel for liquefied gas (which it is intended to convert into compressed gas) is an absorption vessel containing a gel which absorbs any gas evaporated by leakage of heat during periods of rest between the periods of production of compressed gas, and during the latter periods the vaporising liquid withdrawn is passed through a pipe coil within the gel, so that it is chilled and rendered a more effective absorbent. When the storage vessel is empty, the adsorbed gas is recovered by heat. (C) In an air- or other gas-liquefying plant, the gases are dried by passing through a gel, such as silica gel, and the moisture is afterwards removed by a heated gas, e.g., nitrogen, obtained from the same plant. Two (or more) vessels may be used—one drying, the other regenerating. Gases saturated with moisture at 15° may, at the pressure commonly used for the separation of constituents of air, be dried to a dew point of −30 to −40°.

B. M. VENABLES.

Apparatus for testing the viscosity of oils. C. W. B. SHORTO (B.P. 307,602, 13.2.28).—A vertical tube to contain the oil is provided with a tapered internal seating at the bottom, and with a tapered plug an exact fit in the seating. The plug, which is provided with a handle in the form of a rod extending upwards within the tube and longer than the latter,

has convex upper and lower surfaces with an axial hole of exact diameter drilled through the summits of the convexities. A valve or stopper with handle is provided to fit closely to the upper convexity and close the hole. The viscosimeter is operated in the usual way, after taking suitable precautions as to temperature etc., and the time is noted for the oil to drop a certain distance in the tube. B. M. VENABLES.

Apparatus for testing the effect of light and other influences. KELVIN, BOTTOMLEY, & BAIRD, LTD., and F. A. KING (B.P. 309,726, 2.4.28).—A number of "swatch boxes" in which samples of fabrics, paints, etc. may be exposed to light and kept under definite conditions of humidity are arranged round a source of light, such as a carbon arc. They are mounted on trunnions, which are hollow, and serve for a supply of conditioned air to all the boxes in series. When any swatch box is swung down for inspection of a sample, the rotation of the trunnion causes the air stream to be by-passed across that box. B. M. VENABLES.

Fire-extinguishing compound. G. BLENIO (U.S.P. 1,707,597, 2.4.29. Appl., 22.7.22).—A mixture of an aluminium soap with a carbonate is claimed.

H. ROYAL-DAWSON.

Apparatus for introducing air or gaseous fluid into the charge in rotary furnaces. G. BOJNER and A. H. PEHRSON (U.S.P. 1,709,456, 16.4.29. Appl., 21.4.27. Swed., 24.4.26).—See B.P. 269,892; B., 1928, 505.

Heat-transferring apparatus. F. H. BRAMWELL, ASSR. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,708,387, 9.4.29. Appl., 23.7.26. U.K., 22.7.25).—See B.P. 248,999; B., 1926, 405.

Calorimetric bomb. J. DARROCH (U.S.P. 1,708,873, 9.4.29. Appl., 22.12.23. U.K., 13.8.23).—See B.P. 208,492; B., 1924, 320.

Apparatus for [wet] separation of the constituents of pulp. T. G. MARTYN (U.S.P. 1,709,676, 16.4.29. Appl., 24.12.26. S. Afr., 7.1.26).—See B.P. 264,129; B., 1927, 319.

Catalytic apparatus. SELDEN CO., ASSEES. of A. O. JAEGER (B.P. 283,887, 7.1.28. U.S., 19.1.27).—See U.S.P. 1,660,511; B., 1928, 320.

Furnace walls. F. B. BIGELOW (B.P. 310,305, 23.1.28).

[Distribution of fuel and air to] furnaces for pulverised coal. S. LÖFFLER (B.P. 297,835, 25.9.28. Ger., 29.9.27).

[Water] heating apparatus. BASTIAN-MORLEY & CO., ASSEES. of J. P. MORLEY (B.P. 288,594, 5.10.27. U.S., 13.4.27).

Heat-insulating processes, devices, and materials. E. SCHMIDT and E. DYCKERHOFF (B.P. 310,572, 2.2.28. Addn. to B.P. 266,177).

Absorption refrigerating apparatus. ELECTROLUX, LTD., ASSEES. of A. LENNING (B.P. 283,938 and 284,193, [A] 20.1.28, [B] 23.1.28. U.S., [A] 20.1.27, [B] 22.1.27).

Absorption refrigerating systems. ELECTROLUX, LTD., ASSEES. of PLATEN-MÜNTERS REFRIGERATING

SYSTEM AKTIEBOLAG (B.P. 283,937, 20.1.28. Swed., 20.1.27).

Filter material (B.P. 308,187).—See V. Separation of materials (B.P. 284,307).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coking test of Chinese and Japanese coals. T. SHIMMURA (J. Fuel Soc. Japan, 1929, 8, 48—50).—Application of the Lessing test shows that strongly coking coals give swollen, black cokes, whilst weakly coking coals give grey, unswollen cokes. When examined by methods of solvent analysis, the α -compounds, carbonised in the Lessing apparatus, produced a black, compact, powdery coke, β -compounds mostly gave a highly swollen coke, and γ -compounds fused easily, forming black flakes of mushroom appearance. It is concluded that the swelling properties and general appearance of the cokes obtained in the Lessing test bear a close relationship to the coking constituents of the coal. C. B. MARSON.

Heat expenditure in the coking process [for coals]. E. TERRES (Gas- u. Wasserfach, 1929, 72, 361—369).—Fuel needs for different types of carbonising units vary widely, being about 955 kg.-cal. per kg. of coal for intermittent ovens, and generally 540—580 kg.-cal., but sometimes as low as 420—480 kg.-cal. in coke ovens of the most modern type. In determining the thermal effect of coking, a calorimetric bomb is used which contains an electrically-heated quartz vessel; the total energy consumption needed to produce a given temperature rise, with and without a coal charge, is measured. The tar and gas formed are removed continuously, and if information is also required as to the heat of the reaction it is only necessary to pass the distillation products through a second calorimeter and to allow for the work done by these substances in expanding against atmospheric pressure (cf. Terres and Walter, B., 1927, 177; Terres and Meier, B., 1928, 508). Curves are constructed showing the heat consumption and the heat of reaction at temperatures from 600° to 1100° for gas coals from the Ruhr and the Saar, two coking coals from the Ruhr, and a Boldon gas coal. With the Saar coal the heat consumption rose slowly from 700° to 870°, then fell slightly until 940°, and finally rose rapidly; these fluctuations are explained by the curve showing the heat of reaction, which increases suddenly at 870°, but falls again from its maximum at about 940°. The same general form of curve is obtained with other coals, but the intensity of the effect varies considerably, and the heat of reaction may have a negative or a positive value. New coal has a smaller heat requirement than one which has been stored for some time, and the moisture content has also a large effect which does not arise simply from evaporation of water, but because the water-gas reaction comes into play during carbonisation. The relation between heat of reaction and heat consumption on carbonisation at 1000° takes the form of a smooth curve for 28 different coals, and it is found that when the heat of reaction is nil, the heat consumption is 325 kg.-cal. per kg. The nett heat requirement is made up of the total heat effect of the coking process,

the thermal result of the recuperator systems for gas and air, and the radiation and convection losses from the setting. These losses are bigger than is generally realised, and may account for 15–35% of the fuel used. The time which a charge needs for complete carbonisation will depend on these heat requirements and also on the thermal conductivity of the coal and coke. Experiments in this connexion have been carried out, using the method of Heyn and Baur (Mitt. Materialprüf., 1914, Nos. 2 and 3), with coals and cokes of different sizes, and it appears that the dimensions of the particles have an important effect. The values for coal are always very small, but coke gives appreciably larger results, and measurements can be made at higher temperatures. Curves and methods for calculating the mean heat conductivity for a range of temperatures are given for different coals, and the information which can be derived from them with relation to coking time and design of ovens is discussed.

R. H. GRIFFITH.

Apparatus for determining the softening range and the degree of softening of coking coals. G. AGDE and L. VON LYNCKER (Brennstoff-Chem., 1929, 10, 86–87).—The apparatus resembles a penetrometer, a weighted needle, the movement of which can be measured on a graduated dial, resting on the coal sample. The latter is contained in a metal tube, supported vertically in an electrically heated salt bath, the temperature of which is read with a thermometer or thermocouple. The tube is 10 mm. in external diameter, and contains 3 c.c. of coal (1–2 mm. size) resting on 1.5 c.c. of sand. The needle usually carries a 100 g. weight and the rate of heating is 5° per min. By reading the dial pointer for every 5° rise in temperature the softening range and the extent of softening are determined.

W. T. K. BRAUNHOLTZ.

Fixation of sulphur in coal briquettes. R. KADA and K. OGATA (J. Fuel Soc. Japan, 1929, 8, 52–55).—Experiments are described in which calcium oxide, calcium carbonate, magnesium oxide, sodium carbonate, barium carbonate, and ferric oxide are added to the briquettes in order to fix the sulphur and retain it in the ash. An addition of 1–2% of ferric oxide together with some slaked lime was found to be the most efficacious.

C. B. MARSON.

Rough estimation of the content of tar coke in lump coke. G. AGDE and L. VON LYNCKER (Brennstoff-Chem., 1929, 10, 88–89).—Tar coke comprises the solid residue formed by the coking of decomposition products of bitumen which have not been removed by distillation during carbonisation. It is derived from both the extractable and non-extractable bitumen, but the quantity obtained from coal after extraction is very small if the coal is carbonised under greatly reduced pressure. The content of tar coke may thus be roughly estimated by weighing the coke produced by carbonising the non-extracted coal under normal crucible conditions and subtracting from this the weight of coke obtained by carbonising under diminished pressure at 1020° an equivalent quantity of extracted coal. A normal coke contains about 6–8% (not more than 10%) of tar coke.

W. T. K. BRAUNHOLTZ.

Properties of coal for gas producers. S. UCHIDA (J. Fuel Soc. Japan, 1929, 8, 50–52).—It is suggested that in addition to the usual data from the analysis and calorific value determinations, a knowledge of the size of the coal, its caking power, the fusing temperature of the ash, and the reactivity of the coke is necessary to obtain the best working results.

C. B. MARSON.

Apparatus and method for determining the progress of gas evolution from coking coals. G. AGDE and L. VON LYNCKER (Brennstoff-Chem., 1929, 10, 89–90).—The coal (3 g., $\frac{1}{2}$ –1 mm. size), contained in an elongated silica crucible, is placed in a porcelain tube sealed at one end. The tube is heated in a vertical, electric tube furnace, and the temperature, read by a thermocouple just below the sealed end of the porcelain tube, is raised by at least 7° per min. up to the maximum of 1200°. The upper end of the porcelain tube is cooled and contains a plug of asbestos, previously heated to redness, to prevent condensed products dropping back on to the heated coal. The gas is scrubbed by passing it through a bottle containing cotton wool and passes to a 3-way tap, by means of which it is collected and measured alternately every 5 min. in two graduated burettes. Curves are drawn with temperatures as abscissæ and the gas collected in 5 min. as ordinates, an example being given for a raw coal, its oily and solid bitumens, and the extracted coal.

W. T. K. BRAUNHOLTZ.

Petroleum wash-oil thickening in the scrubbing of coke-oven gas. H. M. ULLMANN, D. S. CHAMBERLIN, C. W. SIMMONS, and M. A. THORPE (Ind. Eng. Chem., 1929, 21, 313–314).—Although the sulphur content of a wash oil on thickening increased from 0.182% to 0.967%, no thickening was caused by hydrogen sulphide or sulphur alone under any conditions. Addition of light petroleum to spent oil gave a precipitate containing 9.25% S and 6.17% O, and the substance appears to be derived from the gas which is being stripped, carried in a state of fine suspension. This suspended material was fully investigated by using a Cottrell precipitator, which separated 3.17 g. of a brown tarry substance, containing 12.5% S, per 100 cub. ft. of gas. No thickening occurred in wash oil which dealt with gas that had been cleaned in this way, but the addition of a coal tar, collected from sumps on the plant, produced thickening in an unused oil.

R. H. GRIFFITH.

Blau gas and its industrial applications. L. DAL PRATO (Zymologica, 1929, 4, 35–51).—Descriptions are given of the various processes for obtaining this gas (cf. B., 1925, 621), and of its uses in the synthesis of alcohol and other products, as a substitute for acetylene in autogenous welding, and as an automobile fuel.

T. H. POPE.

Action of sulphuric acid on olefines etc. W. R. ORMANDY and E. C. CRAVEN (J.S.C.I., 1928, 47, 317–320 T).—The olefines from ethylene to pentene have been treated with excess of concentrated sulphuric acid at temperatures below 25°, and the reaction products examined. Inspection of the oil which separated from the acid layer, of oily products extracted by pentane after dilution with water, and of alcohols remaining dissolved showed that high percentages of the mixtures

were colourless, mobile oils having the following mean values: d_{40}^{20} 0.73—0.76, n_D^{20} 1.41—1.43, n_L 0.34, b.p. range 80—350°, mol. wt. 170—200, and containing 85% C and 15% H. The oils extracted by pentane were unsaturated in character: some cyclic olefines and substances of high mol. wt. obtained from cracked spirit appear to give similar results. Treatment of alcohols belonging to the same series yields substances of the same type, but no paraffin-like oils separate with a smaller molecule than that of propyl alcohol, and the cyclohexanols give rise to cyclic compounds of high b.p. and viscosity.

R. H. GRIFFITH.

Production of alcohols from butenes and pentenes through interaction with sulphuric acid. J. F. NORRIS and H. S. DAVIS (J.S.C.I., 1929, 48, 70—71 r).—The authors take issue with the conclusions of Ormandy and Craven (cf. preceding abstract) that alcohols cannot be made from the butenes and pentenes by sulphuric acid; *sec.*- and *tert.*-butyl and amyl alcohols are being manufactured in the United States in this way from olefines made from petroleum products. A review of the conditions necessary for their production is given.

Methods of extraction of pyridine bases. C. AB-DER-HALDEN (Chim. et Ind., 1929, 21, 708—710).—Modern methods of coal-tar distillation employing steam may yield a light oil containing 35—40% of tar acids and 5—7% of bases. The compounds formed by their union are not completely decomposed by sulphuric acid, and the tar acids must first be removed with soda. Pyridine is then obtained as pyridine sulphate which may be further treated: (1) by neutralisation with ammonia in presence of pure benzol followed by distillation of the latter; (2) by addition of the equivalent of caustic soda followed by distillation with live steam. Light pyridine comes over at 93—96° as soluble hydrate. The heavy bases which commence to distil at 96° are insoluble in water. The light pyridine is dehydrated with 50% caustic soda solution; (3) by neutralisation with sodium carbonate. Two layers are formed, the lower one containing sodium sulphate and light pyridine. This is distilled without fractionation. The upper layer is fractionated and gives in succession aqueous pyridine, anhydrous light pyridine, and heavy bases. The aqueous fractions are used for the dissolution of further sodium carbonate. This method is cheaper than (2) in reagents, but consumes more heat.

C. IRWIN.

Volumetric determination of phenols by means of glycerin in distillation tars of brown coals. B. P. LUGOVKIN (J. Chem. Ind. Moscow, 1928 5, 798—799).—The tar is shaken with an equal volume of glycerin; after 2—3 min. (or immersion in hot water) two layers are formed, the lower consisting of a solution of phenols in glycerin, from which the phenols may be recovered by dilution with water. The decrease in volume of the tar layer is measured. Paraffins or much (60—70%) water interfere. For viscous distillates the glycerin is diluted with kerosene.

CHEMICAL ABSTRACTS.

Carbonisation of lubricating oils and fuel oils. N. I. TSCHERNOSHUKOV (Ind. Eng. Chem., 1929, 21, 315—316).—Sludge formation in lubricating and other oils during use is due to oxidation reactions of various types,

and three crude oils have been treated with oxygen at 150° and 15 atm., while the formation of asphaltenes was measured at intervals. Similar experiments with floridin tars from the crude oils showed that no relation existed between the behaviour on oxidation and the results provided by the Conradson coking tests. Satisfactory information as to the value of an oil can only be obtained, therefore, by oxidation tests, especially when conditions of high pressure actually exist under working conditions.

R. H. GRIFFITH.

Determination of olefines. P. S. SMIRNOV (Neft. Choz., 1928, 15, 217—221).—Cracked gasoline is added drop by drop to liquid nitric oxide at —15° to —20°; the liquid is then made alkaline with sodium hydroxide and distilled with steam. The distillate, after separation, is dried with sodium sulphate and filtered through silica gel, which is washed with ether. After centrifuging, the ether is removed by distillation, and the top layer (gasoline) is added to that left after treatment with sodium sulphate. The difference between the total volume of gasoline left after treatment and the original volume represents the unsaturated compounds.

CHEMICAL ABSTRACTS.

Characteristics of amorphous wax. L. D. JONES and F. E. BLACHLY (Ind. Eng. Chem., 1929, 21, 318—320).—When amorphous wax is to be precipitated by centrifuging from lubricating stock it must be present in sufficiently coarse form to make rapid separation possible; this can largely be achieved by choice of a suitable diluent. The properties of the wax depend primarily on its origin, but may be modified by the solvent, and a crystalline wax may be obtained from one that is amorphous by removal of associated impurities. An oil solution containing amorphous wax may be cooled to —45.6° without coating the chilling coils, even when the mixture becomes quite viscous, but crystalline wax is always deposited on the cold surface. This is due to the higher surface tension of the amorphous form caused by the presence of some substance of higher b.p., which can, however, be distilled without appreciable change. It is suggested that this substance is of an asphaltic nature, and can be adsorbed by fuller's earth, but is frequently adsorbed by crystalline wax, especially at low temperatures.

R. H. GRIFFITH.

Acids of montan wax. III. H. TROPSCH and H. KOCH (Brennstoff-Chem., 1929, 10, 82—86; cf. B., 1922, 208 A, 659 A).—The fatty acids present in montan wax obtained from mid-German lignite are purified by fractionally distilling their methyl esters under diminished pressure, and are separated from the distillates by fractional precipitation of their magnesium salts. They are identified from their m.p. and by determining their equivalent by titration with alcoholic potassium hydroxide. The presence in the wax of an acid, $C_{31}H_{62}O_2$, and the absence of the acid, $C_{30}H_{60}O_2$, are established. The former, which constitutes 4.7% of the fatty acids present in the wax, has m.p. 88.5—89.0° and appears to be identical with melissic acid occurring in beeswax.

W. T. K. BRAUNHOLTZ.

Treatment of montan wax without acid. V. TOKMANOV (Neft. Choz., 1928, 15, 224—225).—Treatment with sulphuric acid (6% free monohydrate) is preferred

to that with adsorbents in the production of ceresin; adsorbents give a higher yield, but the product contains unsaturated compounds and naphthenes.

CHEMICAL ABSTRACTS.

See also A., May, 534, **Measurement of flame temperature** (GRIFFITHS and AUBREY). 551, **Thermal behaviour of phenols** (HAGEMANN).

PATENTS.

Coke ovens. L. WILPUTTE (B.P. 309,774, 30.5.28. Addn. to B.P. 308,120; B., 1929, 422).—The heating arrangements of the oven described in the main patent are modified, and undesirably intense combustion in the lower part of the flues is avoided, by supplying gas at alternate intervals to the ports opening at different levels within each flue.

A. B. MANNING.

Coke ovens and chamber ovens. J. Y. JOHNSON. From A. WEINDEL (B.P. 309,760, 15.5.28).—Coking chambers which are heated on one side only have sloping passages connecting the opposite side to the discharge chamber for the distillation products. These passages are also inclined to the longitudinal direction of the chamber, and are preferably narrowed conically from the coking chamber to the discharge chamber. Longitudinal grooves extend along the unheated wall and form the starting point of the off-take passages. The lower edges of the grooves are rounded off and the bricks above may be provided with bulbous projections into the chamber. The offtake passages may be arranged just above the level of the charge only or may be distributed over the wall below the surface of the charge. The discharge chambers are provided with drainage gulleys, and may be heated in such a way that only the heavy tar or pitch is run off through the gulleys, whilst the lighter products are led away to the condensing system.

A. B. MANNING.

Coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,707,419, 2.4.29. Appl., 22.4.22).—A regenerative coking oven is provided with steam-superheating channels below the respective coking chambers and extending from side to side of the battery. Steam is introduced into these channels and passes through suitably arranged distributing channels to the coking chambers.

A. B. MANNING.

Coking retort oven. J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,706,476, 26.3.29. Appl., 13.10.22).—A special flue system communicates with vertical combustion flues which constitute the heating walls.

J. A. SUGDEN.

Regenerative coke ovens. W. MUELLER (B.P. 308,994, 24.11.27).—The regenerators arranged below the oven chambers are each divided into a number of separate sections, alternate sections being traversed by gas and air, respectively, simultaneously flowing in the same direction. The gas and air, thus separately preheated, are supplied in a uniform manner to the heating flues over the whole length of the oven. The waste heat gases are withdrawn through the corresponding regenerator sections below the adjacent oven chamber, also in a uniformly distributed manner throughout the length of the oven. In examples, the flames ascending in one heating flue pass over at the top and down the adjacent

flue, a portion of the gas and air being led up through partition flues to the turning point between the adjacent heating flues; or the vertical flues are connected at the top to a horizontal flue and the vertical passages in the partition wall serve as exit flues for the waste gases and as inlet flues for gas and air, the direction of the flames in the heating flues reversing with each reversal of the gas and air supply. Provision is made for heating with rich gases, and some other possible modifications in the mode of heating are described.

A. B. MANNING.

Regenerative coke oven. C. OTTO (U.S.P. 1,707,537, 2.4.29. Appl., 25.4.25. Ger., 26.6.24).—The heating walls of the oven have vertical flues with cross-over conduits connecting together into independent flue systems the tops of flues of alternate heating walls only. Regenerators for preheating weak gas and air extend longitudinally below the ovens. Provision is made also for heating with strong gas.

A. B. MANNING.

Coking of coal in a retorting space. J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,707,427, 2.4.29. Appl., 24.7.22).—The area of the coking chamber is smaller at the bottom than at the top. External heat is applied to the sides of the chamber so that coking is first completed at the bottom. Steam is then passed through the lower portion until the remainder of the charge is coked. The gas production is increased and over-coking of the lower portion of the charge is avoided.

J. A. SUGDEN.

Vertical continuous distillation furnace. J. PIETERS (B.P. 287,934, 29.3.28. Fr., 30.3.27).—A bench of vertical retorts of the type which are heated from flues constructed in the longitudinal walls of the retorts has the combustion zones of the vertical flues bridged and partly obstructed by a number of bricks parallel to the horizontal long axis of the retorts, but occupying only a small proportion of the vertical depth of the flues, so that the heating gases become thoroughly mixed. For a certain distance below the combustion zone the gas and air are kept separate and are preheated by the cooling coke or residue. The gases after combustion are exhausted through a number of ports in the short unheated wall of the bench, which lead into a collecting passage, one for each retort with a valve to each passage.

B. M. VENABLES.

Apparatus for low-temperature carbonisation. P. C. POPE (B.P. 309,387, 9.12.27).—An annular retort is formed by an externally heated, vertical, cylindrical chamber within which is suspended an axially movable, internal stem, which is enlarged at the bottom to be a loose fit to the outer container. After carbonisation is complete the stem with the coke is lowered into a cooling chamber. A pair of hinged scrapers arranged below the container remove any adherent coke from the stem as it is again raised into position. The retort is charged from a hopper through a heat-insulated, conical valve, which slides upon the rod from which the stem is suspended. During carbonisation low-pressure steam is admitted to the coke-cooling chamber, whence it passes up through the narrow passage between the enlarged part of the stem and the container and assists in carrying away from the retort the volatile products of carbonisation.

A. B. MANNING.

Low-temperature carbonisation of fuels. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 309,253, 26.10.28. Addn. to B.P. 301,975; B., 1929, 119).—Before submitting the fuel to the process described in the main patent it is dried at 250–350° by blowing hot scavenging gases through it at such a rate that the fuel is maintained in a state of active movement on its bed; it is then introduced directly, in its preheated state, into the carbonising chamber.

A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. H. A. HUMPHREY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 308,995, 30.11.27).—Solid carbonaceous material is treated with hydrogen, under at least 100 atm. pressure, and at 300–550°, while held in suspension in a liquid medium of high b.p., contained in a vertical tower.

A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 309,228, 3.10.27. Cf. B.P. 302,941; B., 1929, 232).—The hydrogenation is carried out in the presence of small quantities of substances, *e.g.*, sodium isopropyl-naphthalenesulphonate, which contain the radical of a mineral acid in the molecule, and which lower the surface tension of water.

A. B. MANNING.

Hydrogenation of coal, tars, mineral oils, and other carbonaceous materials. IMPERIAL CHEM. INDUSTRIES, LTD., and K. GORDON (B.P. 309,095 and 309,239, [A] 2.12.27, [B] 14.1.28).—The material is subjected to the action of hydrogen at temperatures of 400–500° under pressures of (A) 300–1000 atm. or (B) 1000–5000 atm. or higher. The process may be carried out in stages, and with or without the use of catalysts. The preliminary hydrogenation may be effected with circulation of the hydrogen, but the hyper-pressure treatment is preferably effected without such circulation.

A. B. MANNING.

Manufacture of valuable liquid products from varieties of coal, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 282,384, 9.12.27. Ger., 20.12.26).—In the hydrogenation of coal etc. under high pressures and at elevated temperatures, and in similar processes, in which, however, there should be no appreciable amount of carbon monoxide present, the presence of iron and nickel is excluded in such forms only as are not physically or chemically resistant or which initiate injurious chemical reactions, *e.g.*, the formation of methane or coke. Copper is excluded if sulphur is present. The apparatus, therefore, is constructed of, or coated with, aluminium or iron and nickel alloys having a high content of chromium, molybdenum, tungsten, vanadium, etc. The metals and alloys used are preferably freed from such impurities as oxygen, sulphur, and carbon by treatment with moist hydrogen at high temperatures. When sulphur is absent silver or its alloys may be used.

A. B. MANNING.

Manufacture of activated carbon. E. BERL (B.P. 283,968, 20.1.28. Ger., 21.1.27).—Acid tars, *e.g.*, such as are produced in the sulphuric acid refining of oils and tars, are neutralised with potassium carbonate, hydroxide, or sulphide, and heated to activating temperatures in the presence of air, carbon dioxide, steam,

or mixtures of these. After cooling and extracting the soluble inorganic constituents the material is preferably again heated in an atmosphere free from or poor in oxygen.

A. B. MANNING.

Production of gas. C. W. ANDREWS and H. A. BRASSERT (U.S.P. 1,707,651, 24.29. Appl., 20.5.22).—A unit containing carbonaceous residue is alternately air-blasted and steamed. The generated water-gas, further heated by passing through another unit containing heated carbonaceous residue, serves to carbonise a bed of fresh coal contained in the same unit, thereby producing a mixture of coal gas and water-gas.

J. A. SUGDEN.

Recovery of ammonia [from gas]. F. OSBORNE ASSR. to SEMET-SOLVAY Co. (U.S.P. 1,703,405, 26.2.29. Appl., 12.4.27).—Ammonia gas, *e.g.*, from coke ovens, at 120–130° is cooled successively to 100° and to 80° in a heat interchanger and an electrical precipitator. Condensate is removed, and the issuing gas is raised to 90–100° by heat exchange with fresh gas in the interchanger and passed into a saturator containing sulphuric acid. Ammonium sulphate crystallises out, and is ejected and centrifuged, the mother-liquors being returned with sulphuric acid to the saturator. Vapours from the saturator are cooled to 30°, the condensate of water and light oils being separated, passed through a second precipitator, and thence to an exhaustor and holder.

R. BRIGHTMAN.

Thermal decomposition of hydrocarbons. S. M. NORWOOD, ASSR. to ELECTRO METALLURGICAL Co. (U.S.P. 1,703,949, 5.3.29. Appl., 11.10.27).—An iron catalyst, containing 15–40% Cr, 2–15% Ni, 0.7–3.0% Si, 0.7–3.0% Mn, and not more than 1% C, is used.

C. HOLLINS.

Cracking of [hydrocarbon] oil. G. EGLOFF, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,703,617, 26.2.29. Appl., 15.12.20. Renewed 24.3.27).—Oil, *e.g.*, Kansas fuel oil, is cracked at, *e.g.*, 135 lb./in.² and 400° in a series of stills and expansion chambers. Reflux condensate from the dephlegmators is returned to the heating tubes, and the uncondensed gas from the final condenser and receivers passes through a common header to the compression pump and reservoir, a check valve in the leads to the header preventing back-pressure on the receivers.

R. BRIGHTMAN.

Cracking of heavier hydrocarbon oils. E. C. HERTHEL and H. L. PELZER, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,703,529, 26.2.29. Appl., 28.5.25).—The oil is circulated through heating tubes from a supply tank containing a bed of 10–18 in. of fuller's earth, the return pipe to the tank delivering to the further end below the oil level and above the bed of earth. Vapours escape to a dephlegmator and thence to condenser and receiver, the phlegms being returned to the circulation pipe below the bed of earth. Fresh oil may be fed in at the top of the dephlegmator or through the circulating pump.

R. BRIGHTMAN.

Cracking of emulsified petroleum oil. G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,703,103, 26.2.29. Appl., 1.9.20. Renewed 2.7.28).—The oil is heated to cracking temperature and the condensate and vapours from the dephlegmator are

passed through separate coils at above 100°, *e.g.*, 190°. Uncondensed vapours from either coil are passed through a water condenser at, *e.g.*, 20°, and the water and light oil are separated in the receiver. The condensate is discharged through separate water condensers to receivers for distillate and reflux condensate, the latter being retreated if desired.

R. BRIGHTMAN.

Refining of petroleum oils. O. DIECKMANN (U.S.P. 1,703,615, 26.2.29. Appl., 1.9.21).—The crude oil, after separating any water, is topped to remove benzene and gasoline and is agitated with concentrated sulphuric acid, the acid sludge being drawn off, partly decomposed with live steam, and extracted with refined petroleum oil. The recovered oil is returned to the acid oil and the residue affords, on extraction with tar oil and neutralisation, cylinder oil and a hard pitch free from coke. The acid oil and extract, after further acid washing, are agitated successively with 10–15% of sodium carbonate at 80–90° and with 20–25% of water and fractionated.

R. BRIGHTMAN.

Means for treating oils. C. B. BELKNAP (U.S.P. 1,704,277, 5.3.29. Appl., 7.5.25. Renewed 25.7.28).—Oil is heated in the liquid state under a predetermined pressure, and the vapours are received in a vapour space adjacent the liquid oil.

H. S. GARLICK.

Combining natural gas and hydrocarbon oil for production of gasoline. L. S. WORTHINGTON, Assr. to C. L. THOMPSON (U.S.P. 1,705,348, 12.3.29. Appl., 6.5.24).—The oil is fed into a chamber in which it is maintained at a constant level. It is passed from this chamber through a heater, is then atomised and mixed with a hydrocarbon gas, and re-introduced into the vapour space of the chamber. The mixture of gas and vapour discharged from the upper part of the chamber passes to a condensing system.

A. B. MANNING.

Recovering oil. B. M. CLOUD (U.S.P. 1,697,260, 1.1.29. Appl., 7.3.27).—Hydrocarbon oil is recovered from oil-containing sand and rock, *e.g.*, "exhausted" wells, by injecting carbon monoxide and hydrogen (water-gas), preferably admixed with 15–30% of acetylene.

R. BRIGHTMAN.

Treatment of distillates from cracking of petroleum oils. R. A. HALLORAN, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,704,246, 5.3.29. Appl., 4.10.23).—The pressure of the distillate, which contains more than 0.2% S, is reduced only sufficiently to release water vapour prior to washing with alkali.

F. G. CLARKE.

Acid treatment of lubricating oils. G. F. OLSEN, Assr. to GEN. PETROLEUM CORP. OF CALIFORNIA (U.S.P. 1,704,206, 5.3.29. Appl., 11.10.26).—A confined stream of oil is treated with an enveloping annular stream of acid. The two liquids are then mixed by being passed into a conduit of reduced diameter.

F. G. CLARKE.

Normal-pressure hydrolysis of acid sludge. E. W. ROTH (U.S.P. 1,682,713, 28.8.28. Appl., 13.4.27).—A supernatant layer of acid sludge, or a mixture of acid sludge and solvent oil, *e.g.*, gas oil, of *d* 0.875–0.886, in contact with sulphuric acid of *d* 1.16–1.53, is heated by means of a steam coil immersed in the acid, which keeps the acid layer at simmering temperature

(about 125°). The disappearance of the sludge coincides with cessation of evolution of sulphur dioxide, and the layer of tar is run off, a portion being left until sufficient free carbon has accumulated from successive charges to interfere with the operations when this residue is run to waste. The requisite concentration of acid is maintained by the addition of small amounts of water as required.

R. BRIGHTMAN.

Recovery of oxidation products of solid hydrocarbons, waxes, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 309,875, 7.11.27).—Mixtures obtained by the incomplete oxidation of waxes etc. are subjected to mechanical pressure at a temperature below the m.p. of the initial material, whereby the oxidation products are expressed as oils. Alternatively, fatty acids are first recovered by conversion into soaps and extraction of these with water, and residual higher alcohols etc. are then recovered by expression.

L. A. COLES.

Lubricating mixture. P. C. GOSS (U.S.P. 1,708,058, 9.4.29. Appl., 7.2.27).—A mineral lubricating oil of a petroleum base is emulsified with an aqueous extract of the inner bark of slippery elm.

H. ROYAL-DAWSON.

[Production of] decolorising carbon. E. URBAIN (U.S.P. 1,709,503, 16.4.29. Appl., 29.3.26. Fr., 12.3.25).—See B.P. 249,138; B., 1926, 1003.

Purification of coal or other gases. J. N. REESON and W. L. MOSS (U.S.P. 1,708,590, 9.4.29. Appl., 13.3.25. Austral., 30.4.24).—See B.P. 249,312; B., 1926, 430.

Separation of low-temperature tar into phenols and hydrocarbons. A. CORRELL, Assr. to ZECHE M. STINNES (U.S.P. 1,709,309, 16.4.29. Appl., 7.11.25. Ger., 10.12.24).—See B.P. 245,633; B., 1926, 184.

Distillation of [mineral] oil. A. E. HARNSBERGER (B.P. 310,393, 23.1.28).—See U.S.P. 1,666,597; B., 1928, 396.

Atomisable motor fuel product. ALOX CHEM. CORP. (B.P. 286,260, 10.10.27. U.S., 2.3.27).—See U.S.P. 1,684,125; B., 1929, 10.

Manufacture of agents for emulsifying, purifying, wetting, etc. by sulphonation. K. MARX, K. BRODERSON, and K. BITTNER, Assrs. to I. G. FARBERIND. A.-G. (U.S.P. 1,708,103, 9.4.29. Appl., 18.5.27. Ger., 19.5.26).—See B.P. 271,474; B., 1928, 326.

Burners for pulverulent fuel and/or liquid fuel. L. GROTE (B.P. 310,555, 26.1.28.)

Producer furnaces (B.P. 305,288). **Apparatus for chemical reactions** (B.P. 309,057 and 309,258). **Drying of coal etc.** (B.P. 308,548). **Agglomerates from pulverised materials** (B.P. 305,051). **Centrifugal machine** (B.P. 299,018). **Treating liquids with gases or vapours** (B.P. 309,206). **Viscosity of oils** (B.P. 307,602).—See I. **Ammonium sulphate** (B.P. 307,037).—See VII. **Castor oil-mineral oil mixtures** (B.P. 308,502).—See XII.

III.—ORGANIC INTERMEDIATES.

See A., May, 521, Anodic behaviour of diphenyl-acetic acid (KATAGISHI and others). Electrolytic

reduction of aldehydes (SHIMA). 548, Hydrogenation of phenanthrene (SCHROETER and others). 550, Perylene and its derivatives (FUNK and others). 555, Di- and tetra-hydro- β -naphthoic acids (LÉON and CHARRO). 579, Acenaphthiminazoles and acenaphthoxazoles (SIRCAR and GUHA-RAY).

Blau gas. DAL PRATO. Action of sulphuric acid on olefines (ORMANDY and CRAVEN; NORRIS and DAVIS). Extraction of pyridine bases. AB-DER-HALDEN. Determination of phenols. LUGOVKIN.—See II. Methyl and ethyl alcohols in sugar beet pulp. JEDLIČKA.—See XVII. Anhydrous alcohol. VON KEUSSLER.—See XVIII.

PATENTS.

Manufacture of organic compounds containing oxygen. I. G. FARBENIND. A.-G. (B.P. 280,522, 28.10.27. Ger., 15.11.26).—In the hydrogenation of oxides of carbon, the use of an iron oxide catalyst containing small amounts of oxides or hydroxides of alkali or alkaline-earth metals below 300° leads to oxygenated products. Copper oxide (*e.g.*, 33%) may be added. C. HOLLINS.

Recovery of acetone from gases containing it. W. R. ORMANDY (B.P. 308,885, 14.3.28).—The gases are scrubbed with moderately concentrated sulphuric or phosphoric acid, which is diluted before distilling off the absorbed acetone. C. HOLLINS.

Manufacture of condensation product from *m*-cresol and acetone. SCHERING-KAHLBAUM A.-G., and H. JORDAN (B.P. 308,741, 28.10.27. Addn. to B.P. 273,684; B., 1929, 236).—When the process of the prior patent is carried out under milder conditions (lower temperature, lower concentration of acid, or with a diluent) the product is 2:2'-*dihydroxy*-4:4'-*dimethyl*- $\beta\beta$ -*diphenylpropane*, m.p. 131–132° (*diacetate*, m.p. 84–85°). C. HOLLINS.

Production of purified styrene. I. OSTRONISLEN-SKY, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,703,950, 5.3.29. Appl., 7.5.24).—Styrene is separated from ethylbenzene by polymerising for 16–48 hrs. at 130–140°, distilling off the ethylbenzene, and depolymerising the styrol at 350–500°. C. HOLLINS.

Manufacture of benzonitrile and other aryl cyanides [nitriles]. R. B. TRUSLER, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,702,711, 19.2.29. Appl., 20.6.27).—Aryl halides which do not contain substituents, *e.g.*, amino-, carboxylic, or hydroxyl groups, which are reactive under the conditions of reaction, are heated at 300° with zinc cyanide or other metallic cyanide in presence of copper, nickel, or cobalt bromide as catalyst. R. BRIGHTMAN.

Manufacture of 2-aminonaphthalene-3-carboxylic [2-amino-3-naphthoic] acid. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 282,450, 19.12.27).—2:3-Hydroxynaphthoic acid (or a salt) is heated with ferrous sulphate and 25% ammonia solution at 200–210° (pressure 40 atm.) to give an iron compound $C_{10}H_6 \begin{smallmatrix} \text{NH} \cdot \text{Fe} \\ \text{CO} \cdot \text{O} \end{smallmatrix}$, which on decomposition with hot caustic alkali yields 2-amino-3-naphthoic acid. C. HOLLINS.

Oxidation of hydrocarbons. E. C. R. MARKS. From ALOX CHEM. CORP. (B.P. 309,382, 6.10.27).—See U.S.P. 1,690,768–9; B., 1929, 199.

Catalytic oxidation of organic compounds. A. O. JAEGER, Assr. to SELDEN CO. (U.S.P. 1,709,853, 23.4.29. Appl., 3.6.27).—See B.P. 291,419; B., 1929, 275.

Preparation of maleic and succinic acids from furfuraldehyde by electrolysis. T. YABUTA, Assr. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,709,207, 16.4.29. Appl., 31.3.26. Japan, 22.6.25).—See B.P. 253,877; B., 1927, 268.

Manufacture of aniline and other arylamines. W. J. HALE and J. W. BRITTON, Assrs. to DOW CHEM. CO. (Re-issue 17,280, 23.4.29, of U.S.P. 1,607,824, 23.11.26).—See B., 1927, 101.

Preparation of dinitrohalogenaryls [halogeno-dinitro-benzenesulphonamides and -benzamides]. E. FISCHER, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,709,256, 16.4.29. Appl., 12.10.27. Ger., 18.10.26).—See B.P. 279,134; B., 1928, 440.

Manufacture of mononitrated diphtaloyl-acridones. W. MIEG and A. JOB, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,709,945, 23.4.29. Appl., 23.5.27. Ger., 27.5.26).—See B.P. 295,645; B., 1928, 781.

Manufacture of naphthaquinone derivatives. A. WAHL and R. LANTZ, Assrs. to SOC. ANON. DES MAT. COL. & PROD. CHIM. DE ST.-DENIS (U.S.P. 1,708,001, 9.4.29. Appl., 22.10.23. Fr., 30.10.22).—See B.P. 206,142; B., 1924, 902.

Manufacture of 1:8-naphthoxyphenanthiophen [*perinaphththioindoxyl*] compounds. J. MÜLLER and M. SCHUBERT, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,709,277, 16.4.29. Appl., 5.10.27. Ger., 12.10.26).—See B.P. 300,771; B., 1929, 200.

IV.—DYESTUFFS.

See A., May, 554, Synthesis of *m*-hydroxyazo-dyes (BUCHERER and HOFFMANN). 567, Differently coloured conditions of anthraquinonylcarboxylic acids and anthraquinol- α -carboxylactones (SCHOLL and others). Derivatives of 3:4-phenanthraquinone (FIESER). 568, Perylene and its derivatives (ZINKE and others). 569 and 574, Plant colouring matters (KARRER and BACHMANN; KARRER and WIDMER). 579, Dyes from acenaphthenequinone (SIRCAR and GUHA-RAY). 580, Pyocyanine (WREDE and STRACK).

PATENTS.

Triarylmethane dyes. IMPERIAL CHEM. INDUSTRIES, LTD., E. H. RODD, and F. L. SHARP (B.P. 308,906, 18.4.28).—Pararosaniline, rosaniline, or a homologue is condensed with an anisidine or phenetidine and sulphonated to give new acid blues. Good yields are obtained even with New Magenta. C. HOLLINS.

Manufacture of substantive dyes of the stilbene series. A. RHEINER, Assr. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,708,897, 9.4.29. Appl., 13.12.26. Ger., 19.12.25).—See B.P. 263,192; B., 1928, 46.

Manufacture of vat dyes of the anthracene series. H. SCHEYER, Assr. to GRASELLI DYESTUFF CORP.

(U.S.P. 1,709,956, 23.4.29. Appl., 26.9.27. Ger., 30.9.26).—See B.P. 299,972; B., 1929, 12.

Manufacture of dihydro-*p*-thioazines of the anthraquinone series. G. KRÄNZLEIN and M. CORELL, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,709,986, 23.4.29. Appl., 15.12.26. Ger., 17.12.25).—See B.P. 263,178; B., 1928, 398.

Manufacture of azo dyes. L. OSWALD, Assr. to J. R. GEIGY SOC. ANON. (U.S.P. 1,709,734, 16.4.29. Appl., 14.7.27. Ger., 29.7.26).—See B.P. 275,220; B., 1928, 399.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Electrical conduction in textiles. III. Anomalous properties. E. J. MURPHY (J. Physical Chem., 1929, 33, 509—532; cf. B., 1929, 318).—The insulation resistance of cotton samples of different types has been measured as a function of applied *P.D.* and humidity, together with the residual *E.M.F.* in cotton at various humidities, and the distribution of resistance in cotton threads. The insulation resistance decreases with an increase in applied *P.D.* under all conditions, and in the range 20—80% R.H. can be represented by the equation $\log_{10} R = -8.5 \times 10^{-2} H + k$, where *H* is % R.H. and *k* is a constant. The passage of an electric current through cotton tends, in general, to increase its resistance and above 70—80% R.H. the rate of change is rapid but below 70—20% it is slow. The increase in resistance is shown to take place in the textile itself and not at the contacts with the electrodes, and the current tends to cause a non-uniform distribution of the resistance of the textile, which depends on the nature of the electrode; with brass it becomes concentrated in the anodic region, whilst with platinum electrodes the greatest resistance occurs in the middle of the textile thread. This non-uniform distribution may remain for hours after the removal of the applied *P.D.* These facts are explained by the effects of the products of electrolysis on the conductivity of the aqueous solutions through which conduction takes place, and their bearing on the measurement of resistance in textiles and other moisture-absorbing materials is discussed. Views similar to those previously advanced to account for *D-C* resistivity and *A-C* capacity and conductivity (*loc. cit.*) are now used to explain the anomalous properties of conduction in textiles.

L. S. THEOBALD.

Relation of moisture content of wool to that of air. V. LÖHRMANN (Kisérlet Köz., 1927, 30, 472—482). The moisture content of wool depends on that of the air, and not on the quality of the wool.

CHEMICAL ABSTRACTS.

Enzymic degradation of cellulose and cotton. O. FAUST and P. KARRER (Helv. Chim. Acta, 1929, 12, 414—417).—Enzymic decomposition of an artificial-silk cellulose with snail cellulase proceeds to a somewhat greater extent when mercerised, provided maturing is of the order of one day. With various forms of cotton at 36° and *p*_H 5.28, increased degradation again occurs with the mercerised material, and proceeds to a greater extent when mercerisation is carried out without stretching the fibre.

H. BURTON.

Properties of acetone-soluble acetylcellulose. K. WERNER and H. ENGELMANN (Z. angew. Chem., 1929, 42, 438—444).—The variation in the viscosity, tensile strength, solubility, and other properties of acetylcellulose of varying acetyl content (50—60% OAc) has been investigated in dried films 0.1 mm. thick prepared from a 13% acetone solution of acetylcellulose. The main results are as follows. The absorption of water in 24 hrs. at the ordinary temperature is inversely proportional to the acetyl content (varying from 1.2% with a specimen containing 60.9% OAc to 25.4% with one containing 42.4%), but with alcohol (containing 10% of ether) a maximum absorption occurs with a 50% acetyl content. Simultaneous determinations of the viscosity of various samples in 2% formic acid and 10% acetone solutions and of the tensile strength of the films have been made. In the former solvent the tensile strength is directly proportional to the viscosity. Samples of low acetyl content, and which, therefore, absorb much water, show a very large decrease in tensile strength on wetting, the latter varying from a 57% decrease with a specimen containing 51.3% OAc to 17% with one containing 60.9%, *i.e.*, one in which very little hydrolysis has occurred. A similar behaviour is shown by nitrocellulose films, the tensile strength increasing with the viscosity, but both the decrease in tensile strength on wetting and the quantity of water absorbed (both of which depend on the nitrogen content of the sample) are much smaller in magnitude than in the case of acetylcellulose, the values for a highly viscous sample containing 12% N (8% and 1%, respectively) scarcely equalling those of an acetylcellulose containing 61% OAc (17% and 1.2%, respectively). Both cellophane and completely hydrolysed acetylcellulose (transparite) have a very high tensile strength (14 kg./mm.²) and show only a very low absorption of alcohol (0.6%), but absorb a large quantity of water and hence exhibit a very large decrease in tensile strength (90—100%) on wetting. On the basis of these results the nature of solutions of acetylcellulose in acetone and other solvents is discussed, and it is concluded that the colloidal character of the solution depends very closely on the acetyl content, which in turn depends on the degree of hydrolysis, and hence on the presence of free hydroxyl groups, in the surface of the triacetylcellulose micelle in a similar manner to that postulated by Highfield (B., 1926, 188) for nitrocellulose solutions, the solvent power depending on the presence of polar and non-polar groups in both the solute and the surface of the micelle. A minimum viscosity is thus a criterion for a homogeneous sol. Hence the minimum viscosity and maximum solubility in acetone occur with a sample containing 58.9% OAc, in which only one of the acetyl groups in the surface of the micelle is hydrolysed, giving an apparent composition $3C_6H_7O_2Ac_2.C_6H_7O_2(OH)Ac_2$ (acetyl 59.5%), but the acetyl value has purely a statistical significance. The technical importance of the results in the preparation of good acetylcellulose products is indicated.

J. W. BAKER.

Structural matter of varieties of cabbage. II. H. PRINGSHEIM and C. R. FORDYCE (Ber., 1929, 62, [B], 831—832; cf. A., 1928, 1227).—The polysaccharide obtained previously from the leaves and stalks of the

white cabbage has been isolated from the leaves and stalks of the red cabbage and from cauliflower. The swollen polysaccharide is readily soluble in Schweizer's solution, and does not appear separable into components by fractional treatment with the reagent in presence of sodium hydroxide. The Röntgen diagram of the polysaccharide is nearly identical with that of cellulose. H. WREN.

See also A., May, 544, Hydrolysis of cellulose (WILLSTÄTTER and ZECHMEISTER).

Fibre plants. TOBLER.—See XVI.

PATENTS.

Preparation from flax or other plants of fibre suitable for spinning by machinery of the kind used in cotton mills. VICKERS, LTD., and O. D. LUCAS (B.P. 309,213, 8.12.27).—Plant fibres, *e.g.*, flax, preferably unretted, are subjected to a chemical treatment of such a nature that the cellulose of the fibre remains substantially unaffected whilst the cementing agent at the nodal points is attacked sufficiently to permit the ultimate fibres to be separated in an unbroken condition by a subsequent mechanical treatment. A suitable treatment consists in digesting the raw material at 150–170° for 1–4 hrs. with 3–5% caustic soda solution containing 1½% of Turkey-red oil and ½% of linseed oil (on the weight of fibre), and finally treating the washed fibre with cold dilute acid, *e.g.*, ½–2% hydrochloric acid. With flax straw the raw material, without previous scutching, is first digested for about ½ hr. in water at 182° to produce furfuraldehyde, which is recovered by distillation. D. J. NORMAN.

Washing or cleaning of wool. E. C. DUHAMEL, and COMP. GEN. DES IND. TEXT. (B.P. 307,199, 20.2.28. Addn. to B.P. 251,669; B., 1928, 187).—In the process of the prior patent a quantity of weak suint liquor exceeding 1 litre per kg. of wool treated is intermittently introduced into the bath, while any excess of liquor is withdrawn therefrom, these two operations being so conducted that the concentration of suint in the bath always corresponds with *d* 1.02 or above.

D. J. NORMAN.

Treatment of wool. V. G. WALSH and E. V. HAYES-GRATZE (B.P. 306,916, 28.9.27).—Wool is cleaned and scoured by treatment with a water-miscible, sulphonated vegetable oil containing no free alkali. [Stat. ref.]

D. J. NORMAN.

Filter material for hot, or acid, or alkaline gases and liquids. M. LEHMANN (B.P. 308,187, 19.12.27).—The filter material has as its base wild silk, which covers all classes of silk not produced by the silkworm. It is stated that dry distillation does not commence until 180° is reached, and that the silk is resistant to acids and alkalis of moderate concentration; dilute acid even improves it. B. M. VENABLES.

Manufacture of cellulose esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 309,203, 3.10.27).—The preparation of organic esters of cellulose is facilitated if the esterification is conducted in the presence of an alkylated naphthalenesulphonic acid or a salt thereof, *e.g.*, 0.7 pt. of isopropyl-naphthalenesulphonic acid per 100 pts. of cellulose. As protective colloids such

compounds as the addition products of sulphuric acid and oleic acid may be used. D. J. NORMAN.

Spinning of artificial silk and the like. J. HUETNER, R. J. H. GAEBEL, and NITRA ART-SILK CO., LTD. (B.P. 309,204, 6.10.27).—In the process of B.P. 303,421 (B., 1929, 208) some of the filaments stretch to a greater extent than others, and tend to separate from the main thread. This tendency is counteracted by causing the liquid set in motion by the travel of the thread to flow towards the bundle of filaments at one or more points, and preferably at or immediately before the point where the thread leaves the bath.

D. J. NORMAN.

Manufacture of hollow artificial textile threads. H. KARPLUS (U.S.P. 1,707,164, 26.3.29. Appl., 27.8.26. Czechoslov., 17.9.25).—Liquid or solid organic, non-crystalline substances, *e.g.*, oils, waxes, soaps, which cannot form bubble-forming vapours, are incorporated as emulsions or suspensions with the spinning solution, and are subsequently removed from the textile threads by suitable solvents. F. R. ENNOS.

Production of fine-fibre artificial silk. A. WAGNER, Assr. to F. KUTTNER (U.S.P. 1,706,717, 26.3.29. Appl., 25.3.27. Ger., 11.6.26).—Threads of cuprammonium silk after being treated with aluminium sulphate solution are wound on to spools, washed with water to remove soluble salts, treated with sulphuric acid to free it from copper, and finally again washed with water, soaped, and dried. F. R. ENNOS.

Reeling of artificial threads. I. G. FARBENIND. A.-G. (B.P. 297,112, 30.8.28. Ger., 15.9.27).—The centrifugal cake of artificial silk is supported during the reeling operation by inserting an elastic bag mounted on a base plate into the centre of the cake and then inflating the bag so that it presses uniformly against the inner surface of the cake. D. J. NORMAN.

Manufacture of [non-curling] adhesive paper. G. H. WILKINSON and J. MITT (B.P. 308,444, 2.3.28).—As an adhesive for the paper a mixture comprising 25–40 pts. of glue, 50–70 pts. of water, and 30–50 pts. of clay, chalk, terra alba, French white, plumbago, or any mixture of these is used. D. J. NORMAN.

Waterproof paper. Greaseproof and waterproof paper. A. L. CLAPP, Assr. to BENNETT, INC. (U.S.P. 1,706,840—1, 26.3.29. Appl., 27.2.26).—Paper pulp is mixed with (A) a soluble, straight-chain fatty acid soap, *e.g.*, sodium stearate, and sodium silicate, or (B) glue, an aqueous dispersion of a waterproofing material, *e.g.*, paraffin wax, rosin, etc., and sodium silicate. Alum or aluminium sulphate is then added in sufficient quantity to precipitate the soap and/or sodium silicate, and to fix the glue and waterproofing material in the pulp, which is then run off on a paper machine. F. R. ENNOS.

Saturating paper. R. P. ROSE and H. E. CUDE, Assrs. to GEN. RUBBER CO. (U.S.P. 1,705,537, 19.3.29. Appl., 11.12.25).—Cotton fibre is mildly cooked to remove fats, waxes, and other non-cellulosic material without impairing the strength of the fibre, and the paper made from this pulp is used for absorbing a plastic substance from a dispersion thereof. D. J. NORMAN.

Carbon paper. M. BANDLI (B.P. 307,714, 11.10.27).—Solutions of cellulose derivatives, *e.g.*, an acetone solution of cellulose acetate, in admixture with suitable softening agents and optionally containing undissolved cellulose in a fine state of division, are used for applying the colouring medium in the manufacture of carbon paper.

D. J. NORMAN.

De-inking of paper. H. C. FISHER, Assr. to RICHARDSON Co. (U.S.P. 1,707,604, 2.4.29. Appl., 28.9.27).—After soaking the pulp in a solution of alkali and of the sodium salt of a sulphonic acid to loosen the inky material, the whole is agitated with a volatile liquid immiscible with water, *e.g.*, benzene, to form an emulsion. It is then deposited on the surface of water maintained at a temperature sufficient to vaporise the volatile liquid, when the inky matter floats on the surface and is skimmed off, and the pulp sinks to the bottom.

F. R. ENNOS.

De-inking of fibrous material. J. DE WITT (U.S.P. 1,705,907, 19.3.29. Appl., 14.7.27).—The paper is soaked in soap solution for approximately 2 hrs., shredded for 15 min., allowed to remain for 1 hr., and shredded for a further 30 min. The pulp is then shredded and washed for 15 min. and allowed to drain. Suitable apparatus is described.

D. J. NORMAN.

Vegetable parchment. W. HARRISON, and BRIT. VEGETABLE PARCHMENT MILLS, LTD. (B.P. 307,108, 3.12.27 and 16.8.28).—Thin vegetable parchment, which is supple, free from minute pinholes, and superior in mechanical strength and equal in appearance to paper of the same weight made from rag pulp, is obtained by using as the base a paper containing a proportion, *e.g.*, 70–75%, of boiled and bleached graminaceous fibres such as sparto.

D. J. NORMAN.

Treating or retting fibre-bearing plants. M. WADDELL and H. C. WATSON, Assrs. to WATSON-WADDELL, LTD. (U.S.P. 1,708,812, 9.4.29. Appl., 31.10.27. U.K., 14.9.27).—See B.P. 302,300; B., 1929, 167.

Degumming [of natural silk]. G. H. ELLIS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,709,662, 16.4.29. Appl., 30.11.25. U.K., 30.10.25).—See B.P. 264,936; B., 1927, 247.

Manufacture of cellulose. H. WALKER, Assr. to G. H. WALKER, M. H. EPSTEIN, and E. MARKS (U.S.P. 1,709,824, 16.4.29. Appl., 12.8.26).—See B.P. 267,226; B., 1927, 361.

Manufacture of fatty acid [acetic] esters of cellulose of high viscosity. H. DREYFUS (U.S.P. 1,708,787, 9.4.29. Appl., 18.4.23. U.K., 24.5.22).—See B.P. 207,562; B., 1924, 129.

Manufacture of paper. G. A. MOURLAQUE (U.S.P. 1,710,375, 23.4.29. Appl., 4.10.27. Fr., 22.8.27).—See B.P. 299,965; B., 1929, 14.

Stencil sheet. S. HORII (Re-issue 17,267, 9.4.29, of U.S.P. 1,587,954, 8.6.26).—See B., 1926, 627.

Rotary drying cylinders [for fabrics, paper, etc.]. J. B. NORTON, and LANG BRIDGE, LTD. (B.P. 309,732, 10.4.28).

[Conveying] means for waterproofing sacks, bags, etc. L. MOTLEY (B.P. 309,636, 7.1.28).

[Pumping] apparatus for use in manufacture of artificial silk or other operations in which liquids are supplied under pressure. BRIT. CELANESE, LTD., and E. KINSELLA (B.P. 310,384, 21.12.27).

Nozzles for spinning artificial silk. RUTH-ALDO Co., INC., Assees. of E. ORIOLI (B.P. 303,782, 3.8.28. Fr., 9.1.28).

Recovery of acetone from gases (B.P. 308,885).—See III. **Bleaching of pulp** (U.S.P. 1,705,897).—See VI. **Coated fabrics etc.** (B.P. 309,391).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

[Colour-]lake formation and cotton dyeing. E. BAUR and H. SCHNYDER (Z. Elektrochem., 1929, 35, 254–263).—Numerous curves are given showing the amounts of various dyes taken up from dye solutions of widely differing concentrations by (i) aluminium hydroxide; (ii) tannin complexes, formed by the interaction of tannin with the hydroxides of either aluminium, chromium, or copper; and (iii) various types of yarns, including cotton and mercerised cotton.

H. T. S. BRITTON.

Coal-tar dyes in wine. MAROTTA.—See XVIII.

PATENTS.

Bleaching of pulp. L. BRADLEY and E. P. McKEEFE (U.S.P. 1,705,897, 19.3.29. Appl., 16.3.22).—Chemical wood pulp resulting from the digestion of wood with cooking liquors containing sodium compounds is treated first with dilute acid and then with an alkaline hypochlorite solution.

D. J. NORMAN.

Mercerisation of cotton. E. C. R. MARKS. From CHEM. FABR. STOCKHAUSEN & Co. (B.P. 308,116, 24.5.28).—The wetting capacity of mercerising liquors is considerably increased, resulting in a more uniform mercerisation and permitting of the use of less concentrated liquors, *e.g.*, of d 1.2–1.22, if the sulphuric compounds of oils, fats, or fatty acids described in B.P. 293,480 and 293,717 (B., 1928, 678, 718), either alone or in admixture with aromatic or hydroaromatic sulphonc acids (*e.g.*, alkylated naphthalenesulphonic acids) or hydrocarbons, chlorinated hydrocarbons, hydrogenated naphthalenes or phenols, etc. are added to the mercerising bath (0.5–2 g. per litre) or used for pretreating the fabric.

D. J. NORMAN.

Carroting animal hair and wool. E. BÖHM (U.S.P. 1,710,565, 23.4.29. Appl., 6.1.28. Austr., 9.2.27).—See B.P. 285,028; B., 1928, 520.

Printing with vat dyes mixed with cellulose esters or ethers. E. PFEFFER, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,710,007, 23.4.29. Appl., 15.10.27. Ger., 26.10.26).—See B.P. 279,864; B., 1929, 15.

Treatment of cellulose acetate artificial silk. A. J. HALL, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,709,470, 16.4.29. Appl., 8.6.28. U.K., 8.6.26).—See B.P. 277,089; B., 1927, 964.

Machines for dyeing, impregnating, or like treatment of fabrics. L. MELLERSH-JACKSON. From

MASCHINENFABR. BENNINGER A.-G. (B.P. 309,702, 17.3.28).

Testing the effect of light etc. (B.P. 309,726).—See I.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Manufacture of synthetic nitric acid. M. KALTENBACH (Chim. et Ind., 1929, 21, 701—707).—Liquid ammonia for nitric acid manufacture can be transported, at any rate over private railways, in ordinary tank-wagons if these are insulated and the ammonia is cooled to -30° before filling. These tank-wagons are charged from high-pressure reservoirs by a valve which automatically maintains a given pressure difference, and the necessary cooling is obtained by partial evaporation of the ammonia. The ammonia is stored at the nitric acid plant in insulated tanks connected by safety valves to a gasholder. When the pressure in this rises a liquefaction plant is automatically started. Tank-wagons are discharged into these through a constant-pressure-difference valve proportional to the capacity of the liquefaction set. With this system of working no difficulty arises in keeping joints tight. An automatic arrangement is described for maintaining a constant composition of the air-ammonia mixture, based on the difference in thermal conductivities of air and the mixture. The Parsons cylindrical catalyser is described. It has greater mechanical strength than the flat gauge. A temperature of 825° can thus be maintained or, with a heat exchanger, 925 — 950° . At this temperature the capacity is double that at 725° , and the conversion efficiency is 95—96% against 88—90%. A device for controlling the volume of nitric acid withdrawn from the absorption towers to correspond with their production is described, together with a spraying arrangement for the tower tops. C. IRWIN.

Analysis of fluorspar. G. E. F. LUNDELL and J. T. HOFFMAN (U.S. Bur. Stand. J. Res., 1929, 2, 671—683; Res. Paper No. 51).—Descriptions are given of methods suitable for the determination of carbonates, silica, sulphur, barium, lead, zinc, and calcium fluoride.

F. J. WILKINS.

Metal carbonyls. R. L. MOND (Chim. et Ind., 1929, 21, 681—700, 937—940).—The history of the discovery of nickel carbonyl and the measurement of its thermal and physical properties is recounted. The nickel should be prepared by reduction at 350° and cooled to 50° in a reducing atmosphere. There is no definite evidence of any compound containing nickel carbonyl as such. The blue substance formed by reaction with nitrogen peroxide is probably $\text{Ni}(\text{NO}_2)_2$. An account of the preparation of other carbonyls is also given. To nickel and iron tetracarbonyls and cobalt tricarbonyl cyclic structures are assigned. Such compounds as cobalt tetracarbonyl are dicyclic. It is shown that this view accords well with the chemical properties of the compounds. The comparatively easy formation of nickel carbonyl at the ordinary temperature and its decomposition at 180° is the groundwork of the Mond nickel process. This is described and illustrated. The operations performed in Canada are magnetic concentration, sintering with

coke and lime whereby part of the iron present combines with the silica as a slag, and treatment in a Bessemer converter whereby the remaining iron is removed as oxide. The greater part of the sulphur is driven off and converted into sulphuric acid, using a vanadium catalyst. The matte contains 80% (Ni+Cu). This matte is shipped to Wales, powdered to 60-mesh, and calcined at 700 — 800° in excess of air. The oxides so produced are extracted with dilute sulphuric acid and the undissolved nickel oxide is filtered on rotary vacuum filters. Copper sulphate is crystallised and when nickel has accumulated in the mother-liquor it is extracted as nickel ammonium sulphate. The dried powder is reduced with water-gas, cooled, and passed without contact with air to the "volatilisers." Here it is treated with carbon monoxide. Several operations are necessary to remove the nickel completely. The gas passes through dust filters to the decomposing towers. The only other commercial use for the carbonyls actually developed is as an anti-knock addition to petrol. Other suggestions have included use in nickel plating, for mirrors on the internal walls of Dewar flasks, for making pure metal for transformer cores, etc.

C. IRWIN.

See also A., May, 489, Phosphorescence of zinc sulphides (PREVER). 503, Adsorption of carbon dioxide and ammonia by silica gel (MAGNUS and KIEFFER). 523, Pure hydrogen peroxide (MATHESON). 524, Preparation of phosphorescent zinc sulphide (COUSTAL and PREVET). 525, Preparation of hypophosphoric acid (VOGEL; PROBST). 528, Determination of sulphate in fluorides (GINSBERG and HOLDER). 529, Determination of fluosilicate and fluorine ions (BAYLE and AMY). Determination of phosphoric acid (ISHIBASHI). 531, Analysis by electrolysis with mercury cathode (MOLDENHAUER and others). 600, Basic titanium salicylate (PICHON). 614, Microdetermination of carbon dioxide in air (KROGH and REHBERG).

Alkalies as germicides. LEVINE and BUCHANAN.—See XXIII.

PATENTS.

Gaseous oxidation of ammonia and gases containing ammonia. F. JOST (B.P. 300,562, 14.11.28, Ger., 15.11.27).—The excess heat of reaction produced when using a high concentration of ammonia and oxidising gases at optimum yield temperature is removed by employing hollow bodies as contact catalysts, the reaction taking place on the external surfaces while the inner surfaces are cooled. W. G. CAREY.

Production of alkali nitrates from alkali chlorides. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,230, 25.6.28).—An alkali chloride is kept in continuous intimate contact (e.g., by counterflow) with liquid nitrogen tetroxide, preferably containing a small amount of moisture. W. G. CAREY.

Producing a mixture of calcium nitrate and ammonium nitrate. KUNSTDÜNGER-PATENT-VERWERTUNGS A.-G., Assees. of F. G. LILJENROTH (B.P. 310,276, 30.10.28, Swed., 5.6.28, Addn. to B.P. 301,486; B., 1929, 171).—Removal of the calcium sulphate formed in the reaction is facilitated by obtaining the hemihydrate

at suitable temperature and concentration, cooling and diluting the solution to produce gypsum, and assisting crystallisation by the addition of small crystals of gypsum from a former operation. W. G. CAREY.

Production of ammonium sulphate by the treatment with gypsum of an ammoniacal solution used for the scrubbing of gases. UNION CHIM. BELGE, SOC. ANON. (B.P. 307,037, 24.3.28. Belg., 2.3.28).—A solution nearly saturated with ammonium sulphate is treated in a gas scrubber with ammonia and with gases obtained by the carbonisation, the gasification, or the combustion of fuel, and, after removal of the crystalline ammonium sulphate which settles out, the liquor is successively treated with calcium sulphate, filtered, distilled to recover ammonia and volatile acids, concentrated to remove water added with the gypsum and during the washing of the calcium carbonate, and returned to the scrubber. L. A. COLES.

Manufacture of ammonium benzoate. J. A. SPINA, ASSR. to HOOPER ELECTROCHEM. CO. (U.S.P. 1,704,636, 5.3.29. Appl., 1.3.27).—Dry ammonia is combined with dry benzoic acid. C. HOLLINS.

Bleaching of raw heavy spar. K. EBERS (U.S.P. 1,709,612, 16.4.29. Appl., 13.7.25. Ger., 17.7.24).—See B.P. 237,268; B., 1925, 847.

Furnace for chemical reactions (B.P. 309,734). **Insulation of vessels for liquefied gases etc.** (B.P. 281,305, 305,974—5).—See I. **Ammonia from gas** (U.S.P. 1,703,405).—See II. **Crystalline cuprous oxide on copper** (B.P. 309,966).—See X.

VIII.—GLASS; CERAMICS.

Reports to the Special Committee on glass-house pots. I. J. F. HYSLOP. II. P. MARSON. III. W. E. S. TURNER (J. Soc. Glass Tech., 1929, 13, 11—20 r).—Three reports on the subject of pot manufacture are presented. I.—Methods of improving the performance of pots are considered, for which the most important factors in the case of pots made from usual clay mixtures are: (a) finely grinding the raw clay and thoroughly mixing clay and grog, and (b) well soaking the finished pot at high temperatures before using. Better pots would be secured by improving pot-making technique to allow of the use of mixtures containing much sillimanite or of highly aluminous grog.

II.—Defects found in pot clay, grog, and grog-clay mixtures, in the process of pot-making, in pot-arching, and in pot-using are summarised.

III.—Sources of clay suitable for pot-making, the osmotic purification of clay, the heat treatment of pots, and the uses of sillimanite are dealt with.

A. COUSEN.

Influence of cullet on rate of melting and other properties of soda-lime-silica glass. F. W. HODKIN, H. W. HOWES, and W. E. S. TURNER (J. Soc. Glass Tech., 1929, 13, 25—37 r).—Variable results were obtained for the rates of melting of series of colourless and greenish-amber soda-lime glasses as the proportion of cullet employed was increased. The colourless glasses were less rapidly "fined" when the cullet exceeded 60%,

but no such effect was found with the coloured glasses. Working-out tests indicated an increase of viscosity and shortening of the working range when more than 40% of cullet had been used, particularly when this was in the form of large-sized lumps. The silica content of remelted glass was slightly less than that of the cullet used, but this could not be ascribed to volatilisation of soda. A. COUSEN.

Influence of addition of small quantities of alkaline salts on ease of melting and on working properties of soda-lime-silica glasses prepared from cullet. F. W. HODKIN, W. E. S. TURNER, and F. WINKS (J. Soc. Glass Tech., 1929, 13, 47—56 r).—The salt which was the most effective, when added to cullet, in producing easy melting and fining, with least extensive corrosion of refractory materials, while resulting in a product having good working properties, was sodium carbonate; the next best was borax. Both salts increased the melting rate, as compared with cullet alone or cullet-batch mixtures, a similar result being obtained also with saltcake with some carbon. The softest glass was made by the use of sodium nitrate with the cullet. A. COUSEN.

Effect of cullet on the melting and working properties of potash-lead oxide-silica glasses. S. ENGLISH, (MRS.) G. A. GREEN, F. W. HODKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1929, 13, 37—47 r).—Increase of cullet beyond 50% retarded the melting rate, though less markedly than with soda-lime glass, whilst the working viscosity and setting rate were increased. A. COUSEN.

Influence of grain size of batch materials on rate of melting [of glass]. (MRS.) G. A. GREEN, F. W. HODKIN, M. PARKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1929, 13, 57—63 r).—Diminution of the size of sand grains increases the melting rate, and similar results are found by decreasing the size of soda ash and of limestone particles in the batch. A. COUSEN.

Velocity of crystallisation of soda-lime-silica glasses. E. ZSCHIMMER (J. Soc. Glass Tech., 1929, 13, 76—84 r).—The apparatus of Zschimmer and Dietzel (B., 1926, 877) for measuring microscopically the rate of growth of crystals in glass at varying temperatures has been used to draw up a series of charts showing temperature-devitrification velocity relationships for soda-lime glasses containing 12—18% Na₂O and 6—16% CaO. A. COUSEN.

Remelting of glasses of abnormal working properties. (MRS.) G. A. GREEN, F. W. HODKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1929, 13, 64—70 r).—Soda-lime glasses made from batches containing 10—15% of moisture, and which had abnormal melting and working properties, formed satisfactory, homogeneous glasses when broken up and remelted, but these had a still greater viscosity and setting rate. Addition of saltcake up to 2% (with a small proportion of carbon for reducing purposes) to the cullet gave a glass having normal properties on remelting. Similar results were found with cullet made from batch containing coarse limestone. A. COUSEN.

Manganese equilibrium in glasses. W. D. BANCROFT and R. L. NUGENT (J. Physical Chem., 1929,

33, 481—497).—Investigation of the system sodium oxide-boric oxide-manganese dioxide has shown that the pink-violet colour of the manganese borax bead under oxidising conditions is due to tervalent manganese, probably in the form of free manganic oxide; under reducing conditions the manganese in the colourless bead is bivalent, and is probably present as manganous borate. Under ordinary oxidising conditions equilibrium is attained at approx. 40% Mn^{+++} , and is shifted towards the manganic side by an increase in oxygen pressure and alkali content and by a fall in temperature. Similar behaviour has been demonstrated quantitatively for the system litharge-boric oxide, and qualitatively for the system sodium oxide-phosphorus pentoxide. Assuming that a similar equilibrium obtains in silicate systems, various applications to the problems of glass-making are discussed. The loss of colour of rose quartz on heating to 575° is also attributed to a shift in a manganous-manganic equilibrium. Under conditions in which there is 40% Mn^{+++} in borax melts, 100% Fe^{++} is practically obtained, and this difference is discussed together with other points of theoretical interest.

L. S. THEOBALD.

Gases in glass. II. Gas and moisture content of glasses. A. BECKER and H. SALMANG (J. Soc. Glass Tech., 1929, 13, 98—111 r).—Small bubbles appeared during fining or remelting of glass, due to several causes, viz., (1) secondary decomposition of unchanged batch materials present; (2) decomposition of dissolved sulphates by reducing gases; (3) penetration of air from pores in the refractory materials; (4) liberation of hydrogen sulphide from sulphide constituents; (5) reaction between the glass and oxides, carbides, etc. present in any commercial iron in contact with it. Water was only retained in the glass when relatively large quantities of steam had to pass through the molten material, or by transference from the furnace atmosphere to the glass. At high temperatures water vapour was a stronger acid than silicic acid, and acted on glass chemically.

A. COUSEN.

Reduction of glasses in hydrogen. J. T. RANDALL and R. E. LEEDS (J. Soc. Glass Tech., 1929, 13, 16—19 r).—Pure soda-lime glass was unaffected in a hydrogen furnace at 1200°, but glasses containing arsenious, lead, or ferric oxides show reduction of these oxides at temperatures between 500° and 1000°. Glass coloured amber by selenium was unaffected by hydrogen at 1000°. The process is suggested as a method of testing for the presence of the above-named oxides in glass.

A. COUSEN.

Le Chatelier's equation for viscosity of glass. F. W. PRESTON (J. Soc. Glass Tech., 1929, 13, 19—24 r).—A criticism of the proposed double-logarithmic formula for the temperature-viscosity relationships of glass (cf. B., 1925, 242).

A. COUSEN.

Viscosity of some glasses of abnormal working properties. S. ENGLISH and W. E. S. TURNER (J. Soc. Glass Tech., 1929, 13, 70—76 r). Viscosity determinations in the case of abnormal glasses obtained by the melting of batches containing large proportions of moisture or coarse-grained limestone, over the lower ranges down to within 15° of the annealing temperature,

failed to confirm the effects indicated by hand-working of the glass, and no explanation of the apparent divergence could be found.

A. COUSEN.

Detection of iron oxides in glass sands. J. T. RANDALL and R. E. LEEDS (J. Soc. Glass Tech., 1929, 13, 15—16 r).—The iron oxide is reduced by heating the sand in an atmosphere of dry hydrogen at 1000° for $\frac{1}{2}$ hr., and the proportion present is judged from the change of colour.

A. COUSEN.

Manufacture of fused bifocal spectacle lenses. R. J. MONTGOMERY (J. Amer. Ceram. Soc., 1929, 12, 274—303).—The conditions necessary to unite satisfactorily a segment of flint glass to a worked surface of crown glass are very exacting. Besides being of high quality in respect to colour and striæ, and of correct optical properties, the softening points and coefficients of expansion must be adjusted to avoid distortions and strain. An account is given of a partial solution of the problem. In testing for strain it was found necessary to make observations through both the edge and the flats of the lense. "Fogging" at the surface of fusion was troublesome, and the use of a barium glass was necessary. Modifications must be made to improve the quality of a normal barium glass and to prevent tarnishing.

J. A. SUGDEN.

Organic agents as aids to adhesion and suspension of glazes. E. S. FOSTER (J. Amer. Ceram. Soc., 1929, 12, 264—269).—The uses and properties of gums tragacanth, arabic, karaya, and dextrin, and "bindex" are described. Some measurements of the effect of the gums on the transverse strength of a ball clay-silica mixture show that gum arabic is the best adhesive. Some points of terra cotta glaze shop practice are described.

J. A. SUGDEN.

Some properties of glaze slips. E. SCHRAMM and R. F. SHERWOOD (J. Amer. Ceram. Soc., 1929, 12, 270—273).—Data are given for the relationship between viscosity and sp. gr. and p_H of a fritted glaze and a raw porcelain glaze. As regards the action of acids and alkalis on the viscosity it was found that the normal working condition of the slips is near that of minimum viscosity. Finer grinding decreases the viscosity of a slip and gives improved appearance at the same glaze fire.

J. A. SUGDEN.

Plasticity and water absorption of clays. H. B. OAKLEY (Nature, 1929, 113, 714—715).—The water absorption of clays, as determined by the increase in concentration of chlorine ions remaining in an aqueous solution of chloride after removal of the clay, is correlated with the plasticity, relative hardness, and bulk density for a clay with different bases, and with the plasticity for different clays with the same base. The water absorption is greatly influenced by the concentration of the reference salt.

A. A. ELDRIDGE.

Effect of typical slags on firebrick with a method of determination correlated to service. C. E. GRIGSBY (J. Amer. Ceram. Soc., 1929, 12, 241—263).—A large number of fireclay and diaspore materials were tested in the laboratory for resistance to typical slags met with in malleable cast-iron, reverberatory furnace, grey-iron cupola, and "bull-ladle" practice. The

results showed excellent correlation with actual service tests in all cases where high-temperature abrasion was not severe. Abrasion is the chief cause of failure in some cases, and the need for such a test is stressed. The laboratory tests were carried out in a small, rotating, oil-fired furnace. The hearth of the furnace held a small amount of metal on the top of which the slag was violently agitated by the blast from the burner entering through a hole in the lid. The wall of the furnace was built up of the twelve bricks to be tested. A tapping hole was provided so that fresh slag could be added every 4 hrs. The test was run until the bricks were eroded to about half their size (*e.g.*, for 24 hrs.) and the results are reported as volume eroded per unit area. Service tests were carried out in several types of furnaces. It was found that for resistance to slag high in iron oxide (reverberatory furnace) bricks of 50% Al_2O_3 or over are superior to flint-fireclay-grog bricks of low (12–20%) porosity. For slags high in lime (cupola) and in soda ("bull-ladle") the flint-fireclay bricks were superior. Where slagging and abrasion are combined, high-alumina bricks are inferior owing to high porosity. Excessive spalling takes place if the porosity is reduced to the point which will give good resistance to attack. Increase in plastic clay or grog content decreases resistance to both types of attack. In general, it seems that if a brick does not exceed 20–22% porosity and withstands slag attack, it will resist high-temperature abrasion satisfactorily. Composition is more important than physical properties in determining resistance to slag, whilst the reverse seems to be true with abrasion.

J. A. SUGDEN.

Ceramics of highly refractory materials. I. Forms of zirconium dioxide. O. RUFF and F. EBERT (*Z. anorg. Chem.*, 1929, 180, 19–41).—X-Ray investigation shows that the ordinary monoclinic form of zirconium dioxide has the lattice constants $a=5.17$, $b=5.27$, $c=5.31$ Å., and $\beta=80.8^\circ$, and has $d\ 5.68$, but at about 1000° undergoes a reversible transformation into a tetragonal form, having $d\ 6.10$, and $a=5.07$ and $c=5.16$ Å., to which change the destruction of vessels made from the pure oxide when heated at high temperatures must be attributed. The oxide prepared by heating the nitrate, oxalate, or basic chloride below about 600° is tetragonal, but metastable, becoming monoclinic above 600° . If, however, the salt is heated with at least 10 g.-mol.% of magnesium oxide at about 1400° there is formed an oxide with a cubic lattice, which, as it does not change on cooling, is a suitable ceramic mass for the production of articles required to undergo very wide variations in temperature without fracture or deformation. A similar result is obtained by heating the monoclinic oxide at 1700° with as little as 4 g.-mol.% of magnesium oxide. The maximum amount of magnesium oxide which the zirconium oxide lattice will take up corresponds with the formation of a compound $\text{Mg}_2\text{Zr}_3\text{O}_8$. The oxides of scandium, yttrium, and calcium, and cerium dioxide have the same effect as magnesium oxide.

R. CUTHILL.

Effect of water vapour and sulphur dioxide on firing of clays. J. KONARZEWSKI and B. KRYNSKI (*Trans. Ceram. Soc.*, 1929, 28, 18–25).—See B., 1928, 671.

See also A., May, 534, **Platinising glass** (TAYLOR).

PATENTS.

Enamelling composition. R. WEINER (U.S.P. 1,706,866, 26.3.29. Appl., 24.7.25).—A composition suitable for application in a cold condition consists of ground silica, clay, lime, soda, boric acid, broken pieces of glass and porcelain, and water, the whole being cold-ground.

H. ROYAL-DAWSON.

[Readily fusible] vitreous [enamel] composition. F. SKAUPY, H. NACHOD, and G. GAIDIES, Assrs. to GEN. ELECTRIC Co. (U.S.P. 1,708,743, 9.4.29. Appl., 1.12.26. Ger., 11.5.26).—See B.P. 267,815; B., 1927, 365.

Apparatus [lehre] for annealing glassware. AMSLER-MORTON Co., W. A. MORTON, and P. L. GEER (B.P. 309,511 and 309,528, 10.10.27).

Manufacture of plate glass. COMP. RÉUNIES DES GLACES & VERRES SPÉCIAUX DU NORD DE LA FRANCE (B.P. 299,034, 14.8.28. Fr., 21.10.27).

Crucible process for manufacture of plate glass. COMP. RÉUNIES DES GLACES ET VERRES SPÉCIAUX DU NORD DE LA FRANCE, and A. HERMANSEN (B.P. 309,974, 19.1.28).

Manufacture of sheets of glass. J. H. LEMAIRE (B.P. 301,083, 20.11.28. Fr., 25.11.27).

Apparatus for manufacture [sealing edges] of reinforced glass. G. B. RILEY (B.P. 310,065, 21.10.27).

Linings for furnaces (B.P. 303,574).—See I.

IX.—BUILDING MATERIALS.

Source of error in determination of plasticity of hydrated lime. F. C. MATHERS and E. C. GOSNELL (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 59–61).—The lack of uniformity of results from determinations of plasticity is due not so much to the variation in the quantity of water or the manner in which it is added as to the amount of stirring at the time of the final test. It is recommended that the proper proportion of water, determined previously, should be added at the beginning, and that the samples should be stirred vigorously for 2 min. with a steel spatula before using the Vicat needle.

C. A. KING.

Effect of sugar on concrete in large-scale trial. M. N. CLAIR (*Eng. News-Rec.*, 1929, 102, 473).—In the construction of a large floor of 1 in. stone concrete with 1 in. topping, sugar was added to the mix for the latter in proportions from 1% to 0.12% with the intention of retarding the setting overnight. Portions with 0.5% and over, however, failed to set satisfactorily in 25 days, and the 2 in. concrete beneath was affected. Laboratory experiments did not indicate any practical treatment likely to yield a satisfactory result, and these parts of the floor were replaced. Parts with 0.25% of sugar were slightly affected.

C. IRWIN.

Fibre saturation point of wood as obtained from electrical conductivity measurement. A. J. STAMM (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 94–97).—The strength of wood is greatly affected by absorbed water up to the fibre-saturation point, and with changes in the moisture content from zero to this point the electrical conductivity increases a million-fold, a linear relation existing between the logarithm of the conductivity and the moisture content. The usefulness of

the conductivity method depends on a uniform distribution of moisture in the fibres, and small specimens less than a fibre length in thickness were used. The same linear relationship exists at the lower moisture content values for all the species studied, a slight deviation occurring above a moisture content of 30%. Results showed that the extractives from redwood were less hygroscopic than was the wood substance itself. The fibre-saturation point indicated by this method corresponds closely with that determined by other methods.

C. A. KING.

Laboratory methods of testing the toxicity of wood preservatives. H. SCHMITZ (Ind. Eng. Chem. [Anal.], 1929, 1, 76—79).—No single method of determining toxicity is entirely satisfactory. Observation of the period of fungoid growth on impregnated wood is too slow to be of any value, whilst the addition of toxic material to nutrient agar does not always represent conditions parallel to practical considerations, though the trend is towards the latter method. It is now customary to sterilise the culture medium and the preservative separately, to shake the mixture vigorously until it is about to gel, and to pour it quickly into cold Petri dishes. Measurement of the toxicity of these mixtures to *Fomes annosus* is now standard practice in the U.S. Forest Products laboratory. To overcome loss of volatile preservatives during sterilisation it is suggested that a weighed quantity of the preservative be placed in a sealed ampoule which is sterilised in the nutrient, and the ampoule fractured to provide the sterile emulsion.

C. A. KING.

PATENTS.

Rotary [cement] kiln. J. S. FASTING, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,708,693, 9.4.29. Appl., 6.1.26. U.K., 7.1.25).—See B.P. 240,049; B., 1925, 923.

Feeding cement slurry to rotary kilns. N. NIELSEN, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,708,705, 9.4.29. Appl., 17.9.26. U.K., 4.5.26).—See B.P. 258,199; B., 1926, 981.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Thermal conductivities of grey cast irons. J. W. DONALDSON (Proc. Inst. Mech. Eng., 1928, 953—983).—The thermal conductivity of grey cast iron varies from 0.11 to 0.137 g.-cal./cm.²/sec. and decreases as the temperature increases; e.g., iron with 0.65% Si had a conductivity of 0.135 at 100°, falling to 0.114 at 400°. With silicon increased to 1.24% corresponding values were 0.127 and 0.109, and, generally, the influence of silicon, nickel, and to a smaller extent manganese and vanadium is to lower the conductivity. Chromium and tungsten act in the opposite direction, and phosphorus has a negligible influence. Structure is of less importance than composition, though ferrite was a better conductor than eutectoid pearlite, as was noticed by the decomposition of pearlite at 550° raising the conductivity, which decreased later by reason of oxidation of ferrite adjacent to the graphite flakes.

C. A. KING.

Change of sp. gr. of cold-worked iron and steel by tempering. K. TAMARU (Bull. Inst. Phys. Chem. Res., Japan, 1929, 8, 187—196).—The temperature at which strain is relieved in iron and steel, as determined by change in sp. gr., was found to be 400° for all samples examined. Armco iron showed a maximum density after tempering at 150°, and a minimum after tempering at 400°. In steels the values of the maxima and minima decrease with decreasing carbon content and disappear at 0.2% C. The results are explained on the assumption of minute changes in grain size and cleavage within the grains, an increase in the total grain boundary surface causing a reduction in density.

C. J. SMITHELLS.

Physical chemistry of steel making; deoxidation with silicon and the formation of ferrous silicate inclusions in steel. C. H. HERTY, JUN., and G. R. FITTERER (Carnegie Inst. Tech. Min. Met. Invest. Bull. 36, 1928, 1—92).—A phase-equilibrium diagram of the system ferrous oxide-silica has been constructed. Ferrous oxide has m.p. not far above 1355°. Two eutectics contain 22% (1240°) and 35% (1260°) SiO₂, respectively. Deoxidation with silicon consists of the formation of silica particles ($\text{Si} + 2\text{FeO} \rightleftharpoons \text{SiO}_2 + 2\text{Fe}$) and subsequent fluxing of dissolved ferrous oxide by the silica formed, the extent of deoxidation for steel of a particular ferrous oxide content depending on the amount of silicon added and the type of silicate formed. When the metal is saturated with silica, $K = [\text{Si}][\text{FeO}]^2 = 1.49 \times 10^{-4}$. As the concentration of dissolved silica decreases, the amount of deoxidation increases. Steels containing ferrous silicates rich in ferrous oxide forge readily and do not show red-shortness; when inclusions rich in silica are present the steel is red-short.

CHEMICAL ABSTRACTS.

Physical chemistry of steel making. Dickenson's method for the determination of non-metallic inclusions in steel. C. H. HERTY, JUN., G. R. FITTERER, and J. F. ECKEL (Carnegie Inst. Tech. Min. Met. Invest. Bull. 37, 1928, 1—37).—Dickenson's method (B., 1926, 491) is satisfactory for steel "killed" with sulphur or aluminium, but not for rimming steel or steels in which the predominant inclusions are manganous or ferrous oxide. In the system ferrous oxide-silica good results are obtained if the inclusions contain more than 60—65% SiO₂; the compound 2FeO.SiO₂ is completely dissolved by the treatment with nitric acid. Ferrous or manganous oxide, alone or combined, is not recovered; alumina is completely recovered, and good results are obtained with manganous oxide-silica inclusions containing more than 30—40% SiO₂. The compound 2FeO.Al₂O₃ is recoverable. A decrease in the concentration of acid does not affect the results for the more soluble oxides. A modified method is described.

CHEMICAL ABSTRACTS.

Properties of materials at high temperatures. The "creep" strength of a "high nickel-high chromium steel" between 600° and 800°. H. J. TAPSELL and J. REMFRY (Dept. Sci. Ind. Res. Eng. Res., Spec. Rep. No. 15, 1929, 7 pp.).—Steel containing 26.5% Ni, 14% Cr, and 3.59% W, being within the Air Ministry Specification D.T.D. 49, has been subjected

to prolonged tensile stresses at 600°, 700°, and 800° to determine the limiting creep stresses, the estimated values of which were 11 (at 600°), 6 (at 700°), and 2 (at 800°) tons/in.² Inter-crystalline cracks could not be detected at 600°, but were noted at the higher temperatures.

C. A. KING.

Magnetostriction of various steels. J. S. RANKIN (J. Roy. Tech. Coll. Glasgow, 1929, 2, [1], 12—19).—The change in length of steels containing 0.17—0.8% C, due to magnetostriction, increases with the magnetising force to a maximum, and then decreases; this maximum occurs at $H = 350$ c.g.s. units with steels containing 0.17—0.4% C, and at $H = 600$ c.g.s. units, with steels containing 0.6—0.8% C. The increase of length is greater the lower the carbon content in plain carbon steels and the lower the nickel content in nickel steels.

A. R. POWELL.

Rapid determination of the endurance limit [of steel] by measuring the electrical resistance. S. IKEDA (Tech. Rep. Tôhoku Imp. Univ., 1929, 8, [2], 41—70).—Bending tests are carried out in Ono's machine under slowly increasing stresses until the electrical resistance begins to increase. This occurs at a definite stress value which is far below the proportionality limit in static tension tests. Specimens stressed just below the endurance limit so found withstand 10⁷ reversals without fracture. In hypoeutectoidal steels the endurance limit increases linearly with the carbon content, other conditions remaining constant; it also bears a linear relation to the hardness number (by scleroscope or Brinell machine), and the graph passes through the origin, thus suggesting that a material of zero hardness has no endurance limit. The angle of inclination of the graph to the abscissa is a characteristic of the metal tested.

A. R. POWELL.

Tensile tests on rods and wires of the same iron. J. MUIR (J. Roy. Tech. Coll. Glasgow, 1929, 2, [1], 5—11).—The yield point of iron wire increases by about 20% when the wire is strained at the primary yield point and allowed to recover from overstrain by boiling in water. Further increases in the yield point may be obtained by repeating the procedure two or three times, but finally the wire breaks without yielding. The extension at the primary yield point is considerably increased by annealing at 400—700°, but otherwise the annealing has no effect on the stress values at the yield point.

A. R. POWELL.

Thermal expansion of iron alloys. A. SCHULZE (Z. tech. Physik, 1928, 9, 338—343; Chem. Zentr., 1928, ii, 2056).—The thermal expansion of iron-silicon (0—8.37%), -aluminium (0—10.52%), and -manganese (0—14.41%) alloys up to 500° was determined. In the first two cases the presence of mixed crystals is indicated.

A. A. ELDRIDGE.

Rapid method for dissolving high-chromium steels for determination of sulphur. B. S. EVANS (Analyst, 1929, 54, 286—287).—In order that the sample of steel may be dissolved in hydrochloric acid alone and afterwards oxidised with nitric acid, a flask is used with ground-in hollow stopper carrying a tapped funnel with a stem which reaches to the bottom

of the flask, and a leading tube the short arm of which ends just below the stopper and having the end of the long arm just above the level of the bottom of the flask. 25 c.c. of water are run into the flask containing 5 g. of sample; the outer end of the leading tube is dipped to the bottom of a cylinder containing 35 c.c. of concentrated nitric acid, and 25 c.c. of concentrated hydrochloric acid are run into the flask. When evolution of gas slackens the flask is warmed slightly, and when action again slackens the tap is opened and, finally, the apparatus is cooled. The tap is then closed and the top of the flask held for a few seconds under a stream of hot water, immediately followed by cold, so that the nitric acid is drawn back so quickly that it has not time to react with the ferrous salts before the cylinder is empty. When the reaction ceases the cylinder and tube are rinsed into the flask, 5 c.c. of 20% potassium nitrate solution are added to the liquid, the solution is evaporated to dryness, and the sulphur determined.

D. G. HEWER.

Mechanism of oxidative processes. XVI.

Rusting of iron. H. WIELAND and W. FRANKE (Annalen, 1929, 469, 257—308; cf. A., 1928, 965).—Schönbein's observation (cf. Traube, A., 1882, 795) that hydrogen peroxide is formed when iron amalgam is shaken with water and air is confirmed; the maximum amount is found after about 1 min. In acid solution no hydrogen peroxide results, but in alkaline solution the amount formed increases with increased concentration of hydroxyl ions. The production of peroxide does not depend on occluded hydrogen in the amalgam, and the amalgam has only a slight decomposing action on the peroxide. The autoxidation of iron amalgam in presence of water, 0.01*N*- and *N*-potassium hydroxide solutions at 37.5° has been studied. In the initial stage the absorption of oxygen is more rapid in alkaline solution, but after 4 hrs. a larger amount is taken up in neutral solution. Ferrous oxide (or hydroxide) is first produced, and this is further oxidised more slowly than the metal. Autoxidation is accelerated slightly by hydrocyanic acid. The autoxidation of iron amalgam with 0.01*N*-hydrogen peroxide at 37.5° in absence of oxygen shows that 63% of the peroxide is used for oxidation; the presence of hydrocyanic acid increases this to 95%. In presence of oxygen the consumption of the hydrogen peroxide is more rapid in the initial stages, but at the same time catalytic decomposition of the peroxide is considerably greater. The function of the peroxide appears to consist of directly oxidising the iron and resultant ferrous hydroxide, at the same time undergoing decomposition (by the iron or hydroxide) into oxygen and water. The formation of hydrogen peroxide from iron powder (ferrum reductum) and oxygen occurs only in presence of alkali. The amount formed is only one tenth of that using iron amalgam, and a maximum value is reached after 0.5 min. Decomposition of hydrogen peroxide by iron powder is much more rapid in presence of water than in potassium hydroxide solution, thereby explaining the non-detection of the peroxide by autoxidation of iron in pure water. The authors believe that the peroxide is actually produced in this case and in the rusting of the metal.

Iron powder is oxidised much more rapidly than the amalgam by 0.01*N*-hydrogen peroxide at 37.5°; in 0.01*N*-potassium hydroxide solution the major part of the peroxide is decomposed. This difference in neutral and alkaline solution is not shown by the amalgam. Autoxidation of iron powder by oxygen is accelerated by hydrocyanic acid. This acceleration appears to be specific, and is not due to changes in p_H (limits 6.8 and 6.4). The effect of various acids and acid mixtures on the change shows that the autoxidation of iron is directly opposite to that of ferrous salts (A., 1928, 965), indicating that conversion of ferrous into ferric ions is brought about by hydrogen peroxide, and not by oxygen. Oxidation of leucine by oxygen in presence of iron powder proceeds to the extent of about 0.5% (cf. Handovsky, A., 1928, 718). The activity of the iron depends on the temperature at which the iron oxide is reduced, but cooling in hydrogen or nitrogen has no effect (cf. Handovsky, *loc. cit.*). The velocity of autoxidation of iron in presence of leucine is about ten times as great as in water, but this acceleration depends considerably on the amino-acid concentration. Glycine, alanine, and asparagine have a similar effect. Small amounts of neutral salts (*e.g.*, sodium sulphate) have an accelerating influence, increasing with increased concentration of the salt. A similar effect is shown by copper sulphate. Iron amalgam is practically unaffected by neutral salts, and increasing acidity of the solution causes a retardation in the rate of autoxidation (cf. A., 1928, 965). With added leucine acceleration occurs in each solvent used, and is in the following order: 0.01*N*-sulphuric acid, 0.01*N*-potassium hydroxide, water. The increase with water is, however, not so great as with iron powder. A considerable inhibition of the autoxidation of iron occurs when the metal is rendered passive by treatment with potassium hydroxide, ferricyanide, chromate, or permanganate. The recovered iron from these experiments shows varying but increased oxygen absorption in all cases. Potassium ferricyanide or chromate does not affect iron amalgam, but permanganate has the same action as with the powder. Whilst the oxygen absorption of the powder, in presence of water, is retarded by increasing oxygen concentration, the amalgam shows a reversed effect. When iron powder is oxidised with more concentrated hydrogen peroxide, a smaller amount of oxidation occurs than with a 0.01*N*-solution. Replacement of oxygen by other hydrogen acceptors, namely quinone, methylene-blue, ethyl peroxide, and dithiodiglycollic acid, and working in an atmosphere of nitrogen causes "rusting" of iron in presence of pure water. In the first case added copper or sodium sulphate accelerates the disappearance of the quinone. The above results agree with the prevailing theory that the process of rusting is essentially electrochemical.

H. BURTON.

Nickel-copper alloys. A. KRUPKOWSKI (Rev. Mét., 1929, 26, 131—153, 193—208).—The physical properties of the nickel-copper system have been determined using alloys which have been thoroughly homogenised by annealing for several days. The magnetic transformation point lies on a straight line which cuts the axis of temperature at absolute zero at a composition of 41.5% Ni. The electrical conductivity at 0° falls

rapidly with addition of nickel to copper and with addition of copper to nickel, the curve being almost parallel to the axis of composition for alloys with 30—70% Ni. The temperature coefficient of resistance falls sharply with up to 3% Ni, then more slowly to zero at 40—50% Ni, rises sharply to 75% Ni, where there is a sharp deflexion in the curve, and finally rises rapidly to the value for pure nickel. Temperature-electrical resistance curves for alloys containing 2.1—38.8% Ni show a distinct deflexion corresponding with the change $\alpha\text{Cu} \rightarrow \beta\text{Cu}$ at -110° to -150° . Using the shape of the transformation-composition curves as a basis, the author divides copper-nickel alloys into two classes: those containing up to 41.5% Ni, in which copper plays the part of the solvent, and those containing more than 41.5% Ni, in which nickel is the solvent. The alloys of the first class are non-magnetic at all temperatures, whereas those of the second class can be either magnetic or non-magnetic, according to the temperature. The curves showing the maximum of the temperature coefficient of resistance as a function of the composition indicate that the allotropic transformation from the magnetic to the non-magnetic form begins at a temperature a little below the Curie point. Micrographic examination, dilatometric analysis, and measurements of electrical resistance afforded no indication whatever of the presence of a compound in the system which consists solely of a continuous series of solid solutions.

A. R. POWELL.

Methods of testing the corrosion of light metals and alloys. X. WACHÉ and G. CHAUDRON (Rev. Mét., 1929, 26, 209—213).—As a standard method for determining the rate of corrosion of aluminium and its light alloys a modification of the Mylius oxidising salt solution is recommended. The solution should contain 1% of hydrogen peroxide and 1% of sodium chloride; more concentrated solutions exhibit no greater activity. After immersion of the specimen for a predetermined time it is washed, dried, and weighed, the oxidation products are collected from the solution, washed, dried for 36 hrs. at 50°, and weighed, and from these figures the amount of aluminium oxidised per unit area of surface is calculated. In hydrogen peroxide solutions alone corrosion is very slow, but addition of very little sodium chloride induces rapid corrosion, especially of aluminium alloys; in fact, the rate of corrosion in the above solution affords a useful guide in differentiating between aluminium and duralumin, the latter dissolving much more rapidly.

A. R. POWELL.

Application of the E.M.F. of dissolution to the study of light alloys. AUBERT and PROT (Rev. Mét., 1929, 26, 214—217).—The E.M.F. of aluminium and the usual constituents of aluminium alloys against pure nickel in sea water have been determined, as well as those of couples in which one element is copper, iron, nickel, cadmium, or zinc and the other aluminium or its light alloys. The bearing of the results on the rate of corrosion of the alloys in sea water is briefly discussed.

A. R. POWELL.

Corrosion of aluminium alloys. QUILLARD and BASCOU (Rev. Mét., 1929, 26, 217—220).—The rates of corrosion of aluminium and its light alloys may be

compared by measuring the *E.M.F.* of dissolution before and after immersion of the specimens in mercuric chloride solution. In this way it is shown that the rate of corrosion of aluminium is decreased by the addition of increasing quantities of manganese.

A. R. POWELL.

Properties of cold-drawn wires, with particular reference to repeated torsional stresses. F. C. LEA and R. A. BATEY (Proc. Inst. Mech. Eng., 1928, 865—899).—The resistance of cold-drawn wires to repeated shear stresses is much less than the static properties would indicate. Heavy cold-work producing high static resistance damages and cracks the surface of the material, which fails under bending or torsional tests, of which the Wöhler test is regarded as the most satisfactory. The behaviour of wire under torsional stresses can be improved greatly by low-temperature heat-treatment.

C. A. KING.

Cyanide extraction of gold and silver in arsenical and antimonial ores. E. S. LEAVER and J. A. WOOLF (Chem. Eng. Min. Rev., 1929, 21, 221—223).—To obtain a high extraction of gold and silver by cyaniding, arsenical and antimonial ores must be roasted at 450—500° for a time sufficient to convert the arsenic and antimony into volatile compounds or into insoluble arsenates and antimonates. Addition of coal or pyrites favours volatilisation, and addition of lime favours the formation of insoluble quinquivalent compounds. Too high a roasting temperature leads to incomplete extraction of the values by cyanide owing to the "locking up" of the gold and silver in ferric and lead arsenates and antimonates and to the formation of the silver-silica complex.

A. R. POWELL.

Analysis of chrome ores. T. R. CUNNINGHAM and T. R. MCNEILL (Ind. Eng. Chem. [Anal.], 1929, 1, 70—72).—For general analysis, chromite is decomposed by digesting it with a mixture of sulphuric and perchloric acids or, alternatively, by fusion with a mixture of sodium carbonate and borax. In the latter case erratic results due to the presence of boron are prevented by concentrating the solution in the presence of methyl alcohol. After reduction of chromium with sulphurous acid and separation of silica the solution is oxidised with nitric acid and precipitated twice with ammonia, iron being separated from the mixed hydroxide precipitate by the cupferron method, and titanium determined colorimetrically. The filtrate from the cupferron precipitate after concentration and oxidation with nitric acid and potassium chlorate is precipitated with ammonia, the alumina after reprecipitation being weighed. Chromium is best determined by fusing a fresh sample with sodium peroxide, and oxidising the solution completely with ammonium persulphate and potassium permanganate. Phosphoric acid is then added to the cooled solution, and an excess of standardised ferrous ammonium sulphate is titrated back with permanganate solution. Lime and magnesia together with manganese are precipitated with ammonium monohydrogen phosphate, lime being later separated from alcoholic solution as calcium sulphate, followed by precipitation as the oxalate; manganese can be determined by the bismuthate method after the destruction of all organic matter in the solution.

C. A. KING.

Colloids in the electroplating of metals. W. BRUM (5th Coll. Symp. Mon., 1928, 301—312).—Colloids tend to decrease the growth of existing crystals and increase the formation of new crystals, thus producing a fine-grained deposit; the action is often specific. They may alter the proportions of metals deposited, and tend to produce a uniform deposit on a cathode of irregular shape. In acid copper or zinc solutions they increase the cathode polarisation. The effects produced by colloids in electrodeposition of metals are discussed in general.

CHEMICAL ABSTRACTS.

Rapid copper-plating of steel with a thin intermediate nickel deposit. M. BALLAY (Rev. Mét., 1929, 26, 221—223).—Steel can be plated directly with copper only from cyanide baths from which deposition is slow. By plating it for 1 min. at 35—55° at 15 amp./dm.² in a bath containing 400 g. of nickel sulphate, 22 g. of the chloride, and 22 g. of boric acid per litre, a thin coating of nickel is produced which can be plated directly with copper in an acid sulphate bath at 45° using 10 amp./dm.² In both operations the electrolyte is agitated by means of a current of air. The coatings obtained in 3 min. from this bath surpass those produced in 45 min. from a cyanide bath.

A. R. POWELL.

Chromium plating. R. SCHNEIDEWIND (Univ. Mich. Eng. Res. Bull., 1928, No. 10, 141 pp.).—A survey, whence the optimum conditions for the production of a smooth, bright deposit are defined.

CHEMICAL ABSTRACTS.

Stepped lowering of the α 1 transformation in steels. T. MURAKAMI (Tech. Rep. Tôhoku, 1929, 8, 119—127).—See B., 1928, 300.

See also A., May, 510, **Transformations of β -phase in zinc-copper alloys** (SALDAU). 530, **Determination of molybdenum in steel** (THURNWALD). 534, **Tellurium-bismuth thermo-element** (LEWITSKY and LUKOMSKY). **Platinising glass etc.** (TAYLOR).

Blau gas. DAL PRATO.—See II. **Effect of slags on firebrick.** GRIGSBY.—See VIII. **Chromium plating baths.** PINNER and BAKER.—See XI.

PATENTS.

[Tilting] furnaces for metals. T. HILL, and BRIT. COPPER MANUFRS., LTD. (B.P. 308,637, 23.12.27).—A furnace body which is charged from the top is mounted on trunnions for side-pouring. The top of the furnace is provided with a fume-collecting casing with fume exhaust which may be common to a series of such furnaces.

C. A. KING.

Metallurgical apparatus and process [for reducing iron ore]. F. D. S. ROBERTSON, Assr. to F. G. CLARK (U.S.P. 1,702,575, 19.2.29. Appl., 21.4.21).—Iron ore is allowed to fall through a vertical cylindrical furnace in which are suspended a number of vertical electric resistors and which is provided with a conical discharge outlet into an air-tight receiver through which the reducing gas (hydrogen or carbon monoxide) passes in countercurrent to the ore stream.

A. R. POWELL.

Annealing furnace. A.-G. BROWN, BOVERI & Co. (B.P. 282,799, 28.12.27. Ger., 27.12.26).—In a continuous furnace for bright annealing, the charging chamber communicates with the annealing chamber through a

gas drum, both chambers having bottom openings in the same plane. The action of charging or discharging by means of a charging table pumps protective gas into the drum.
C. A. KING.

Annealing furnace. W. J. DIEDERICH, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,707,300, 2.4.29. Appl., 7.11.27).—Means are provided for stacking material to be annealed, of substantially annular form, around an electrical resistor in a gas-tight container, so that the inner parts of the material are heated by direct radiation and the outer parts by convection.
J. S. G. THOMAS.

Heat-treating furnaces. SURFACE COMBUSTION CO., Asses. of R. M. HEAMES, H. STARK, and B. W. LINDQUIST (B.P. 291,446, 22.5.28. U.S., 2.6.27).—Small metal articles are heat-treated in a furnace heated by liquid fuel burning below the hearth. The articles are carried through the furnace on a chromium-steel ribbon conveyor, a travelling hood confining the objects in a more or less closed channel.
C. A. KING.

Heat-resisting metallic material. W. E. RUDER, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,706,130, 19.3.29. Appl., 14.9.25).—The surface layer of a metallic article consisting principally of iron is alloyed with aluminium to form a heat-resisting coating, whilst the interior portion, which is free from aluminium, contains a material preventing the penetration of aluminium thereinto at high temperatures.
A. B. MANNING.

Heat treatment of articles. A. LE BOUTILLIER, Assr. to WESTERN ELECTRIC CO. (U.S.P. 1,706,725, 26.3.29. Appl., 19.2.27).—Metallic sheets to be annealed at a high temperature are prevented from becoming welded together by being interleaved with metal plates having an oxide scale before being subjected to heat treatment.
H. ROYAL-DAWSON.

Imparting a shearing-resistant hardness to rust-proof knives and scissors. G. HAMMESFAHR (B.P. 294,573, 12.7.28. Ger., 26.7.27).—The articles are finished from the forged state by grinding and then hardened by heating in a salt-bath furnace; they are subsequently polished.
M. E. NOTTAGE.

Reduction of ores and manufacture of metals [iron] and alloys. W. A. LOKE (B.P. 309,458, 28.12.27).—Iron sands are cleaned by magnetic separation, mixed with a carbonaceous reducing agent, and passed downwards through a rotating kiln provided with internal vanes to keep the charge thoroughly mixed during its reduction by means of a countercurrent of reducing gases. The discharge from the kiln passes directly into an arc furnace through the arc itself to obtain molten iron ready for refining and carburising to steel.
A. R. POWELL.

Manufacture of steel in electric furnaces. FONDERIA MILANESE DI ACCIAIO (B.P. 283,489, 10.1.28. Italy, 10.1.27).—Steel scrap is melted and carburised in a cupola, from which it is tapped into one of two electric refining furnaces which are used alternately to make the process continuous and to ensure a constant load on the electric supply.
A. R. POWELL.

Manufacture of steels and castings. E. H. DOPFÉE (B.P. 293,764, 10.7.28. Fr., 11.7.27).—Metals and/or

metalloids are introduced into baths of steel and cast iron in the form of alloys with aluminium, such alloys containing more than 9% Al and being free from carbide, iron, carbon, sulphur, and phosphorus.

M. E. NOTTAGE.
Manufacture of steel and alloy steels. F. C. LANGENBERG and M. A. GROSSMANN (B.P. 309,971, 18.1.28).—Iron containing less than 0.05% C is treated with 0.15–1% of manganese to reduce the oxygen content below 0.03%. Addition of 0.15–0.6% of copper and 0.03–0.25% of molybdenum produces a steel free from red-shortness and suitable for the manufacture of boiler tubes.
A. R. POWELL.

Manufacture of [high-speed] steel. STAHLWERK BECKER A.-G. (B.P. 282,744 and 305,105, 15.12.27. Ger., 30.12.26).—In steels containing (A) vanadium and/or cobalt, the carbon content is increased above 0.6% by 0.16% for each 1% V and by 0.04% for each 1% Co, or (B) cobalt, addition of 0.4% C is similarly made for each 1% Co.
F. G. CROSSE.

[Manganese-chromium steel] alloys. H. ETHELLE, A. POPPLEWELL, and L. CAMERON & SON, LTD. (B.P. 309,841, 15.10.27).—The steel contains 12–40% (Mn + Cr), of which the manganese is 5–10%. The preferred composition is 9.8% Mn, 18.88% Cr, 0.7% C, and the remainder iron, silicon, and up to 1% of one or more of the following: nickel, cobalt, vanadium, titanium, molybdenum, aluminium, copper, or tungsten.
A. R. POWELL.

Treatment of complex copper ores. W. G. PERKINS (B.P. 309,940, 18.1.28).—Copper ores containing the metal in the oxidised and in the sulphide form are leached, with or without a preliminary low-temperature roast, with a solvent which dissolves the copper not present as sulphide, and the residue is subjected to froth flotation to recover the sulphide.
A. R. POWELL.

Melting and refining of copper. H. H. ALEXANDER (B.P. 309,848, 16.1.28).—Crude copper is melted on a sloping hearth in a reverberatory furnace so that the molten metal runs down into a bath in front of the hearth; melting is effected by means of hot combustion gases of such composition that the impurities in the copper are oxidised as it melts without at the same time oxidising the copper.
A. R. POWELL.

Production of crystalline cuprous oxide upon copper surfaces. A. K. CROAD. From HANOVIA CHEM. & MANUF. CO. (B.P. 309,966, 14.12.27).—Clean polished copper is heated slowly up to 1025° in an atmosphere in which the partial pressure of oxygen does not exceed 15 mm.; the oxygen pressure is then increased to 200 mm. for 5–15 min. until a predetermined thickness of the cuprous oxide coating is produced, and finally the article is cooled in an atmosphere in which the partial pressure of oxygen does not exceed 15 mm.
A. R. POWELL.

Smelting of zinc ores. R. L. LLOYD, Assr. to DWIGHT & LLOYD METALLURG. CO. (U.S.P. 1,709,135, 16.4.29. Appl., 2.11.26).—A receptacle chiefly composed of zinc oxide bonded with zinc sulphate is suitable for the process.
H. ROYAL-DAWSON.

Manufacture of [antimonial] lead alloys [containing manganese]. J. STONE & Co., LTD., and W. LAMBERT (B.P. 309,399, 9.1.28).—An alloy containing 82.9—71% Pb, 12—18% Sb, 5—10% Sn, and 0.1—1% Mn is made by adding the requisite amounts of 5% manganese-tin and 50 : 50 antimony-lead alloy to molten lead.

A. R. POWELL.

Manufacture of [antimonial lead] alloys. W. E. BEATTY. From WESTERN ELECTRIC CO., INC. (B.P. 309,629, 14.1.28).—Antimonial lead alloys containing up to 4% Sb are melted with up to 1% of arsenic, cadmium, sulphur, tellurium, thallium, or bismuth, the alloy is cast, annealed at 240°, quenched, and aged at 20—100° until maximum hardness is obtained.

A. R. POWELL.

Production of amalgams to be used in dentistry. E. W. FISCHER and E. W. J. VIRGIN (B.P. 283,488, 10.1.28. Ger., 10.1.27).—Powdered silver-tin alloy is coated with mercury by heating it with acid mercuric chloride or with finely-divided mercury, from mercuric formate, and dilute hydrochloric acid. A. R. POWELL.

Refining and mixing of metals and alloys. T. D. KELLY (B.P. 310,119, 13.2.28).—The metal or alloy is melted by means of a direct-current arc between a graphite anode and the metal as cathode, the molten metal being stirred by eddy currents induced by an alternating current passing through a conductor encircling the bath. The impurities tend to migrate below the anode, whence they rapidly oxidise or volatilise. The process is useful in purifying iron-nickel-aluminium alloys, ferrochromium, and copper-tungsten alloys from carbon, sulphur, and other deleterious impurities.

A. R. POWELL.

Coating metals [with silicon]. GEN. ELECTRIC CO., LTD., and W. SINGLETON (B.P. 309,393, 9.1.28).—The metal is heated in an inert gas containing silicon tetrachloride, and then annealed in hydrogen or other non-oxidising atmosphere. The resulting coating is resistant to oxidation at high temperatures and to attack by acids.

A. R. POWELL.

Manufacture of [metallic] catalysts. HOWARDS & SONS, LTD., J. W. BLAGDEN, and G. C. H. CLARK (B.P. 309,743, 20.4.28).—An alloy of copper, nickel, and/or iron with magnesium, zinc, and/or calcium is oxidised by heating in the air or by pouring it in thin streams through heated air, and the resulting mass of oxides is crushed and used as a catalyst, with or without partial reduction in hydrogen.

A. R. POWELL.

Working-down metal residues. M. and L. MEYER (HÜTTENWERKE TEMPELHOF A. MEYER) (B.P. 285,462—3, 16.2.28. Ger., 17.2.27).—(A) From the raw material (metal residues containing tin and at least two of the metals antimony, lead, and copper) in the form of oxides, the copper is extracted as sulphate by treatment with sulphuric acid, the lead sulphate thereby produced being separated as lead chloride by treatment with boiling chloride solution; the residue, on fusion with caustic alkali and alkali chloride, yields a mixture of alkali stannate, antimonate, and plumbate, which on lixiviation with water yields insoluble alkali antimonate, which is collected, and a solution from which the lead

may be precipitated as sulphide. The three components may then be treated by known methods. (B) Residues consisting of a mixture of the metals and oxides are fused with caustic alkali and alkali chloride at the outset, the molten mass is separated from the unchanged metal and extracted with water, and any copper oxide separated as copper sulphate from the sodium antimonate is removed by treatment with sulphuric acid.

M. E. NOTTAGE.

Production of aluminium. J. WEBER and H. HAUSER, Assrs. to ALUMINIUM-IND. A.-G. (U.S.P. 1,709,759, 16.4.29. Appl., 3.6.26. Ger., 27.1.26).—See B.P. 265,170; B., 1928, 412.

Production of mercury. W. GLAESER, Assr. to GLAESER RES. CORP. (Re-issue 17,276, 23.4.29, of U.S.P. 1,637,481, 2.8.27).—See B., 1927, 819.

Electrodeposition of metals. T. W. S. HUTCHINS (U.S.P. 1,709,268, 16.4.29. Appl., 30.6.25. U.K., 24.7.24).—See B.P. 239,977; B., 1925, 926.

Production of foundry moulds. MORRIS MOTORS (1926), LTD., and A. SMITH (B.P. 310,187, 27.4.28).

[Mould for] producing [wheel] castings. W. E. EVANS. From GES. F. FÖRDERANLAGEN E. HECKEL M.B.H. (B.P. 310,663, 3.5.28).

[Torch for autogenous] welding. AIR REDUCTION CO., INC. (B.P. 303,896, 8.1.28. U.S., 13.1.28).

[Apparatus for solvent] removal of grease and oil from metal articles. E. C. R. MARKS. From E. HARBECK (B.P. 310,683, 25.5.28).

Linings for furnaces (B.P. 303,574).—See I.

XI.—ELECTROTECHNICS.

“Bent cathode test” for determining the optimum ratio of chromic acid to sulphate in chromium plating baths. W. L. PINNER and E. M. BAKER (Amer. Electrochem. Soc., May, 1929. Advance copy. 11 pp.).—In order to be able to determine rapidly the necessary change to be made in the sulphate content of a chromium plating bath so as to remedy unsatisfactory working, a sample of the solution is removed and electrolysed in a beaker provided with a lead anode and a cathode consisting of a strip of copper sheet bent into an L-shape with the horizontal portion projecting towards the anode. From the character and distribution of the deposit on the bent cathode, the required change in the ratio of chromic acid to sulphate can be approximately estimated, and, since the chromic acid content of the bath is readily determined, the change to be made in its sulphate content is known. The method has the advantage of avoiding the lengthy sulphate determinations otherwise required, and also of indicating the true optimum sulphate ratio for an actual bath which is no longer of the standard composition in respect of other substances. Test experiments with the bent cathode apparatus indicate that increasing the chromic acid concentration from *M* to 6*M* lowers the optimum sulphate ratio (molar concentration of chromic acid : normality of sulphate) from about 75 to 30, the value 50 corresponding with 2.5*M*-chromic acid, but the permissible range of variation of the sulphate

ratio increases as the chromic acid concentration is increased to 3*M*. and then decreases again. Increasing amounts of tervalent chromium increase the optimum sulphate ratio, but the effect is small until the concentration reaches about 0.3*M*. Increasing amounts of iron in the solution first raise the optimum sulphate ratio, but a maximum is reached with about 6 g./litre of iron; higher concentrations of iron decrease the ratio again. Also the range of permissible variation of the sulphate ratio is greatest in solutions containing about 6 g./litre of iron, the optimum ratio then being about 70. This concentration of iron does not appreciably affect the throwing power of the bath, but higher concentrations decrease it, and also have the disadvantage of increasing the specific resistance. Increasing amounts of tervalent chromium decrease the throwing power considerably.

H. J. T. ELLINGHAM.

See also A. May, 496. **New superconductors** (VAN AUBEL and others). 521. **Anodic behaviour of diphenylacetic acid** (KATAGISHI and others). **Electrolytic reduction of aldehydes** (SHIMA). 531. **Analysis by electrolysis with mercury cathode** (MOLDENHAUER and others). 533. **Gauge for measurement of high vacua** (STANLEY). 535. **X-Ray photographs** (EBERT).

Conduction in textiles. MURPHY.—See V. **Electroplating of metals.** BLUM. **Copper-plating of steel.** BALLAY. **Chromium plating.** SCHNEIDEWIND.—See X.

PATENTS.

Electrolytic rectifier. G. D. BAGLEY and F. T. BOWDITCH. ASSIG. to NAT. CARBON CO., INC. (U.S.P. 1,706,950, 26.3.29. Appl., 28.6.24).—Electrodes comprising, respectively, aluminium and a conducting, solid metal oxide are immersed in an electrolyte containing a citrate radical.

J. S. G. THOMAS.

Electrolytic apparatus. R. C. BENNER. ASSIG. to NAT. CARBON CO., INC. (U.S.P. 1,706,951, 26.3.29. Appl., 28.6.24).—A tantalum electrode and one composed of graphite coated with lead peroxide are immersed in an electrolyte.

J. S. G. THOMAS.

Electrode material. W. B. GERO, ASSIG. to WESTINGHOUSE LAMP CO. (U.S.P. 1,707,002, 26.3.29. Appl., 26.7.26).—A gaseous conduction device of the point-to-point discharge type comprises an extended cathode and a small anode composed, at least in part, of porous mixed metal.

J. S. G. THOMAS.

Manufacture of electron-emitting bodies. WESTINGHOUSE LAMP CO., ASSIG. to C. V. IREDELL (B.P. 285,884, 24.2.28. U.S., 24.2.27).—A tungsten filament is coated with a mixture of thorium and a small proportion (1–3% of its thorium content) of ceria and the mixture is reduced. [Stat. ref.]

J. S. G. THOMAS.

Ionisation chamber. SIEMENS-REINIGER VERFA. GES. F. MED. TECHN. (B.P. 296,769, 23.3.28. Ger., 8.9.27).—The outer wall of an ionisation chamber consists of a hollow, pressed, and sintered graphite electrode protected by a thin sheet of elastic material, preferably celluloid.

J. S. G. THOMAS.

Insulating materials [for under-water cables]. W. E. BEATTY. FROM BELL TELEPHONE LABS., INC.

(B.P. 309,851, 15.2.27. Cf. B.P. 307,966: B., 1929, 401).—Water-soluble substances and nitrogenous protein matter are removed from crude rubber or latex by treatment with water in a pressure tank at about 150°; one or more substances such as montan wax, montan pitch, balata, and gutta-percha may be added to give the product strength and plasticity.

J. S. G. THOMAS.

Separation of materials of different physical qualities. B. GRANIG (B.P. 254,307, 29.9.27. Austr., 28.1.27).—The particles of materials having different permeabilities or dielectric constants are separated by being passed singly through an electro-magnetic or electrostatic field (preferably alternating) which is one of a pair that are exactly equal when no foreign bodies are present. The current induced in a circuit coupled to both fields caused by the presence of such materials is utilised to operate deflecting or collecting devices to remove the bodies into various paths according to their physical properties. The induced currents may be amplified by known means.

B. M. VENABLES.

Pasted accumulator grid or plate. W. HADDOX and J. McD. BURNETT (U.S.P. 1,710,617, 23.4.29. Appl., 14.4.27. U.K., 19.4.26).—See B.P. 274,208: B., 1927, 705.

Precipitation of hafnium and zirconium on an incandescent body [electric lamp filament]. J. H. DE BOER and A. E. VAN ARKEL, ASSIG. to N. V. PHILIPS' GLOEI-LAMPENFABR. (U.S.P. 1,709,781, 16.4.29. Appl., 6.10.25. Holl., 25.7.25).—See F.P. 604,391; B., 1926, 886.

Galvanic cell [for pocket lamp batteries]. C. GAISER (B.P. 284,626, 21.1.28).

[Depolarising bobbin for] dry cells. E. C. R. MARKS. FROM NAT. CARBON CO., INC. (B.P. 309,769, 25.5.28).

Luminous electrical discharge tube. R. W. JAMES. FROM MANHATTAN ELECTRICAL SUPPLY CO., INC. (B.P. 310,056, 22.10.27).

Electronic discharge bulbs. ETABL. GAUFFE-GALLOT ET PILON (B.P. 301,855, 9.2.28. Fr., 7.12.27).

Manufacture of electrical porcelain insulators. C. H. THOMPSON (B.P. 310,201, 8.5.28).

Removal of insulation from insulated wire. F. M. VAN GELDEREN (B.P. 299,866, 6.9.28. U.S., 4.11.27).

Linings for furnaces (B.P. 303,574). **Drying and impregnating insulating materials** (B.P. 308,641).—See I. **Refining metals and alloys** (B.P. 310,119).—See X. **Electrodeposition of rubber** (U.S.P. 1,702,705).—See XIV. **Röntgen-ray photography** (B.P. 286,283 and 309,529).—See XXI.

XII.—FATS; OILS; WAXES.

Colloidal calcium soaps. E. SAUER (Chem. Umschau, 1929, 36, 129–132).—The conditions governing the formation of colloidal calcium soap in the reaction between alkali soaps and hard waters has been studied. The proportion of colloidal soap formed is increased by the presence of excess of alkali soap, but varies with the fatty acid and particular calcium soap employed. The presence of filter-paper fibres or textile fibres

induces precipitation of the bulk of the colloidal calcium soap, which is most effectively avoided by the use of suitable protective colloids, *e.g.*, gelatin, gum arabic.

E. LEWKOWITSCH.

Tung oil. III. D. HOLDE, W. BLEYBERG, and M. A. AZIZ (Farben-Ztg., 1929, 34, 1725—1726; cf. B., 1928, 612, 825).—The apparent tendency for the six-membered conjugated linking system of elæostearic acid to close on elimination of halogen hydride from the halogenated acid, giving rise to aromatic derivatives (*loc. cit.*), was apparently confirmed by the detection, among the oxidation products of elæostearic acid treated in this manner, of phthalic acid by the usual fluorescein reaction. Further investigation, however, showed that a number of other oxidation products feasibly present, *e.g.*, mono- and di-basic aliphatic acids, could give rise to similar fluorescence. The fluorescein test was therefore modified by omission of the sulphuric acid and by raising the temperature of fusion to 205—210°, under which conditions phthalic acid alone gave a fluorescing solution. On repeating the halogenation *etc.* of elæostearic acid, no phthalic acid was now detected.

S. S. WOOLF.

Bromo-derivatives of linolenic acid. W. KIMURA (Chem. Umschau, 1929, 36, 126—128).—Linolenic acid, prepared by the reduction of hexabromostearic acid (m.p. 183°), on treatment with the theoretical amount of bromine yielded a crystalline hexabromostearic acid and another oily hexabromide: the latter appears to be a mixture of isomeric liquid hexabromostearic acids, which darkens in air or on heating to a yellow or red-brown colour, but is stable at 100°. No partially saturated tetrabromide could be found, so that the formation of β -linolenic acid by debromination of the hexabromide is doubted. On repeated debromination and bromination of the liquid hexabromide a gradual decrease in the yield of solid and liquid hexabromides was observed, accompanied by a decrease in the iodine value, and an increase in the mol. wt. of the linolenic acid, probably due to the formation of oxidised and polymerised acids.

E. LEWKOWITSCH.

American safflower-seed oil. G. S. JAMIESON and S. I. GERTLER (Oil & Fat Ind., 1929, 6, [4], 11—13).—Hot-pressed oil from safflower seed grown in Montana had d_{20}^{25} 0.9243, n_D^{25} 1.4744, acid value 5.56, iodine value 149.3 (Hanus), 149.1 (Wijs), saponif. value 190.5, acetyl value 12.5, Reichert-Meissl value 0.2, Polenske value 0.1, hexabromide 0.4%, unsaponifiable matter 0.59%. The oil contained 87.72% of unsaturated acids (iodine value 156) and 5.93% of saturated acids; from the detailed examination of these the composition of the oil was determined as: glycerides of acids—oleic 25.7%, linoleic 65.8%, linolenic 0.15%, myristic 0.04%, palmitic 4.1%, stearic 1.6%, arachidic 0.4%, lignoceric 0.06%; unsaponifiable 0.59%. Safflower-seed oil has better drying properties than sunflower-seed or soya-bean oils (probably due to the larger proportion of linoleic acid), and is suitable for use in the paint and varnish industries.

E. LEWKOWITSCH.

Staphylea oil. A. FERENCZ and G. CSERESZNYÉS (Magyar Gyóg. Társ. Értes., 1928, 4, 24—29).—The oil has d_{15}^{15} 0.9355, n_D^{25} 1.47165, acid value 2.00, saponif. value 190.28, iodine value 108.34, Hehner value 95.51,

Reichert-Meissl value 2.64, Polenske value 0.50. The unsaturated acids (90.30% of the total) consist of oleic (94%), linoleic (5.50%), and linolenic (0.50%) acids.

CHEMICAL ABSTRACTS.

Determination of iodine value of fatty drying oils. VON REIBNITZ (Farben-Ztg., 1929, 34, 1782—1784).—The iodine values of raw and cooked drying oils were determined by the Hübl-Waller and Hanus methods, the time and temperature of reaction being varied. The results show the marked influence of these variables on the iodine value of tung oil and, to a smaller extent, linseed oil. The Wijs method is less prone to these uncertainties, and is therefore recommended, although it gives higher values than the other two methods, which give concordant results when standard conditions obtain.

S. S. WOOLF.

Accuracy of the determination of viscosity of oils and varnishes by the [rising] bubble method. RUCHTI (Farben-Ztg., 1929, 34, 1899—1901).—A close relationship was observed between the Engler viscosities of stand oil-mineral oil solutions and the times of rise of an air bubble in a narrow tube filled with these solutions. The times observed in the latter method may be converted directly by factor into the Engler values.

E. LEWKOWITSCH.

Thermal analysis of beeswax. J. STITZ (Magyar Chem. Fol., 1928, 34, 18—22).—The f.p. of beeswax is markedly altered by adulteration. The f.p. of three fractions, obtained by means of cotton filters, are compared with tabulated values.

CHEMICAL ABSTRACTS.

See also A., May, 539, **Synthetic glycerides** (AVERILL and others). 540, **Degradation of saturated fatty acids** (NAEGELI and others). 541, β -**Dihydroxy-palmitic acid** (VOTOČEK and PRELOG). 568, **Saponin of sugar beet** (REHORST). 607, **Decomposition of fats by bacteria** (HAAG). 609, **Chemical nature of vitamin-A** (DRUMMOND and BAKER). 610, **Determination of vitamin-A** (JONES and others).

Report of Government Laboratory, Amsterdam. STRAUB.—See XIX.

PATENTS.

Manufacture of margarine. A. K. EPSTEIN (U.S.P., 1,707,800, 2.4.29. Appl., 8.3.26).—The warm liquid emulsion of fats and milk is stabilised by contact with cold water, the temperature of which is sufficient to solidify it into discrete particles. These are then conveyed in thin layers through relatively warmer water at below the m.p. of the solidified emulsion in order to bring about a rapid tempering process; the whole is finally worked into a plastic mass.

F. R. ENNOS.

Neutralising free fatty acids by glycerin. METALLGES. A.-G. (METALLBANK & METALLURGISCHE GES. A.-G.) (B.P. 291,767, 8.6.28, Ger., 8.6.27).—Neutralisation is effected under a high vacuum produced economically by the interposition of an ejector (preferably steam-operated) between the reaction vessel and the condenser, which is fitted with a vacuum pump.

E. LEWKOWITSCH.

Heating stills particularly for steam-distillation of fatty acids. METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 291,093, 10.5.28. Ger., 27.5.27).

—Preliminary heating is effected by ordinary boiler steam, and the increasing steam temperature subsequently required is automatically produced by a steam compressor interposed between the boiler and the heating system of the still.
E. LEWKOWITSCH.

Treating castor oil to produce substances miscible with mineral oils. M. MELAMID (B.P. 308,502, 27.4.28).—Castor oil is heated with metallic catalysts (e.g., tin, bismuth, nickel, or their alloys) at about 280° under usual pressure or *in vacuo*.
E. LEWKOWITSCH.

Cooling of margarine emulsions. A. GERSTENBERG and H. J. J. BIGUM (B.P. 309,744, 23.4.28).

Substances for cleansing purposes, more especially for removing [theatrical] make-up. (SIR) G. C. MARKS. FROM CHESAVON-FABR. CHEM.-TECHN. PHARM. PRÄPARATE GES.M.B.H. (B.P. 279,454, 18.10.27).

Viscosity of oils (B.P. 307,602).—See I. **Oxidation products of waxes etc.** (B.P. 309,875).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Determination of oil absorption of pigments. C. P. VAN HOEK (Farben-Ztg., 1929, 34, 1784—1785).—Agreement is obtained between oil absorption values as obtained by laboratory methods (due, e.g., to Gardner or Wolff) and those obtained under working conditions in paint manufacture, if in laboratory determinations the pigment is added to the oil instead of *vice versa*. This is considered a logical procedure since a pigment-in-oil dispersion is sought. Oil absorption cannot be regarded as a criterion of a pigment since so many factors influence the results obtained, e.g., size and shape of particles, adsorbed air and moisture, electric charges on the pigment, interfacial tension and chemical relationships between pigment and vehicle, acid value and viscosity of vehicle, time and method of mixing, etc.

S. S. WOOLF.

Determination of oil absorption of pigments. H. WOLFF, G. ZEIDLER, and W. TOELDT (Farben-Ztg., 1929, 34, 1901).—In answer to the criticism of van Hoek (cf. preceding abstract), it is re-asserted that the method described previously (Farben-Ztg., 1928, 33, 2730) gives the most reproducible characteristics and the lowest values so far obtained in practice, although the figures may not be identical with the theoretical minimum oil absorptions.
E. LEWKOWITSCH.

Crystallising lacquers and toluol resins. H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., April, 1929, Circ. No. 348, 321—331).—Crystallising or frosting lacquers may be produced by incorporating powdered synthetic resins, which are initially soluble in the combined solvents, but crystallise out as the solvents become concentrated by evaporation. Resins produced by condensation of toluene derivatives with formaldehyde are highly satisfactory; they are obtainable in various grades of hardness, are retained in colloidal solution with cellulose acetate, are soluble in the usual lacquer solvents but insoluble in linseed oil, are transparent to all wave-lengths contained in sunlight, and show little tendency to become yellow on exposure.
E. LEWKOWITSCH.

Determination of acid value of resins. E. STOCK (Farben-Ztg., 1929, 34, 1727).—The acid value of a number of Albertol and other resins were determined by the normal direct-titration method and by Albert's back-titration method (full details of which are quoted). The results obtained by the two methods are in substantial agreement. It is pointed out, however, that the Albert method shows the advantage of a sharp colour change even in the cases of dark-coloured resins where the normal method gives an indefinite end-point.
S. S. WOOLF.

See also A., May, 572, **Resin acid of Manila copals** (RUZICKA and HOSKING). 573, **Acid from Finnish pine resin** (NORDSTRÖM).

Acetone-soluble acetylcellulose. WERNER and ENGELMANN.—See V. **Colour-lake formation.** BAUR and SCHNYDER.—See VI. **Safflower-seed oil.** JAMIESON and GERTLER. **Viscosity of varnishes.** RUCHTI.—See XII.

PATENTS.

Nitrocellulose solutions and the like. NOBEL INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 308,230, 14.10.27).—A homogeneous nitrocellulose solution, suitable for use in the production of artificial leather, contains a vegetable oil which causes stickiness and is a softener but a non-solvent for nitrocellulose, e.g., blown castor oil, cottonseed oil, rape oil, together with 2—5% (on the total oil content) of a non-volatile paraffin or naphthene hydrocarbon oil in order to overcome the stickiness due to the vegetable oil.
S. S. WOOLF.

Nitrocellulose solutions or compositions. W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,062, 18.10.27).—Wet pigments and wet nitrocellulose containing 5—60% of water are incorporated, together with other ingredients, with a solvent volatile in the air and having a b.p. not below about 120°, e.g., butyl acetate, ethylene glycol ethyl ether. Another solvent of higher b.p., e.g., methylcyclohexanone, may be added when the water content of the composition is above 6%.
L. A. COLES.

[Production of coloured] nitrocellulose products and the like. W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,964, 11.10.27).—Pigmented celluloid, lacquers, etc. are produced by mixing cellulose esters with pigments while either (or both) of these is in a wet condition, with or without the addition of plasticising agents, softening agents, etc., and subsequently removing the water by passing the mixture between rollers at a suitable temperature, the dry product being dissolved in solvents when lacquers etc. are desired. [Stat. ref.]
L. A. COLES.

Coating composition. M. BLANCATO, ASST. to KENT-BLANCATO Co., INC. (U.S.P. 1,709,056, 16.4.29. Appl. 2.5.27).—Celluloid is dissolved in a mixture of acetic acid and acetone containing ethyl sulphate.

H. ROYAL-DAWSON.

Application of materials or bodies for the purpose of coating or otherwise covering surfaces. A. C. HYDE (B.P. 310,080, 19.1.28).—Material, e.g., finely-divided strontium sulphide, and an agglutinant,

e.g., sodium silicate solution, are sprayed on to a surface through separate nozzles adjusted so that the sprays meet before reaching the surface. L. A. COLES.

Production of oil-soluble phenol-aldehyde condensation products. BAKELITE GES.M.B.H. (B.P. 283,124, 3.1.28. Ger., 3.1.27. Addn. to B.P. 282,414; B., 1929, 333).—Natural resins, *e.g.*, colophony, copal, shellac, or solutions of these in suitable diluents or in oils or varnishes, are mixed with the condensation products obtained with or without the use of catalysts as described in the prior patent. L. A. COLES.

Manufacture of resinous condensation products of aldehydes with phenol and urea, or of aldehydes and urea. J. W. CRUMP, and BAKELITE, LTD. (B.P. 309,849, 16.1.28).—A solution of urea in part of the aldehyde solution used for the condensation is added gradually to the remainder of the aldehyde solution which is kept hot or, preferably, at the b.p., and contains in solution the phenol, when this forms one of the constituents. Water and uncondensed material are removed from the initial condensation product, while this is still in a soluble form, by distillation *in vacuo*, preferably in the presence of a neutral dehydrating agent capable of forming a constant-boiling mixture with water, *e.g.*, butyl alcohol, followed by open-pan boiling. An alkaline condensing agent may be added towards the end of the condensation, and an acid agent, *e.g.*, oxalic acid, during the vacuum distillation. The ratio of phenol to urea may vary from $\frac{1}{2}$:1 to 5:1, the lower and higher ratios giving products suitable for the manufacture of moulded articles etc. and of lacquers etc., respectively. L. A. COLES.

[Synthetic] resinous compounds. BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, R. NEWBOUND, and A. T. WARD (B.P. 308,671, 23.12.27).—A "glyptal" or "plasticised glyptal" resin is heated under ordinary or raised pressure with a natural or another artificial resin for some hours until a transparent resinous product which will dissolve readily in fatty oils and is then soluble in benzol, turpentine, white (petroleum) spirit, etc. is obtained. S. S. WOOLF.

[Roller] grinding mills [for paint etc.]. A. E. G. MACCALLUM (B.P. 307,312—3, 5.9.27).

Testing the effect of light etc. (B.P. 309,726).—See I.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Chart for the estimation of equivalent cures [conditions of vulcanisation of rubber]. C. L. BRITAIN (Ind. Eng. Chem., 1929, 21, 362—364).—An "area diagram" is constructed with a horizontal axis graduated with a uniform "time" scale; the vertical "temperature" scale is so constructed that the distances of the "temperature" lines above the "time" axis are proportional to the corresponding rates of vulcanisation. The conditions of any vulcanisation, whether at constant or various temperatures, are plotted on this chart; the area enclosed between the "vulcanisation curve" and the time axis gives a mathematical expression of the intensity of the vulcanisation effect. D. F. TWISS.

Accelerator consumption during vulcanisation. L. V. WISTINGHAUSEN (Kautschuk, 1929, 5, 57—61,

75—77).—Experiments were made as to the proportion of mercaptobenzthiazole and diphenylguanidine, respectively, recoverable by means of extraction with acetone at various stages of vulcanisation. Diphenylguanidine was determined as the picrate, and mercaptobenzthiazole by dissolving the acetone extract in benzene and adding a benzene solution of copper oleate; the initially-formed cupric salt changes overnight into dibenzthiazole disulphide and the insoluble cuprous salt, the weight of mercaptobenzthiazole being obtained from the latter by using the factor 1.4556. With sprayed rubber the rate of consumption of either accelerator was greater than with crêpe rubber, Peruvian rubber occupying an intermediate position except for mercaptobenzthiazole with zinc oxide, when accelerator consumption was least for crêpe and greatest for Peruvian rubber, although the mixture containing the latter was less vulcanised. Increase of ratio of sulphur to rubber caused a marked increase in the rate of vulcanisation as judged by tensile characteristics. In the absence of zinc oxide, however, increased sulphur had little influence on the rate of disappearance of accelerator, but with zinc oxide present it induced increase in rate of vulcanisation and of accelerator consumption. In all cases the disappearance of mercaptobenzthiazole appeared to approach a more definite limit than that of diphenylguanidine. With neither accelerator is the action purely catalytic; in the presence of zinc oxide a zinc compound is formed which then reacts with sulphur. Certain forms of amorphous carbon, probably by adsorptive influence, reduce the percentage of extractable accelerator. D. F. TWISS.

Electrokinetic potential of rubber. B. N. GHOSH and P. STAMBERGER (Kautschuk, 1929, 5, 99—100).—Measurements of electro-osmotic flow provide the most convenient method for investigation of electrokinetic potential with rubber latex. Glass capillaries (approx. 0.5 mm. diam., and 5 cm. long) were lined with a film of rubber which had been applied by means of rubber solution or purified latex and, after drying, fixed in position by heating for 3 hrs. at 120—130°. Experiments were made with one or more such capillaries in parallel, and a potential of 200 volts, using water and solutions of sodium hydroxide, ammonia, hydrochloric and acetic acids, potassium and barium chlorides, and thorium nitrate. The relation of the results to the electrokinetic potential of natural latex, and the influence of composition of this on its potential and stability, are being investigated. D. F. TWISS.

PATENTS.

Production of rubber from rubber latex. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 309,245, 23. and 27.1.28).—Latex is coagulated with acids or acid-producing substances in the presence of an organic substance which possesses soap-like properties and reduces the surface tension of the latex but is not decomposed by acid with formation of insoluble products. Suitable substances of this character are aromatic or hydro-aromatic sulphonic acids containing substituent alkyl, cycloalkyl, or aryl groups, or sulphonic acids derived from aliphatic tar oils or mineral oils. If the latex is quickly acidified to $pH < 2$ before or after the addition of such a substance, the whole mass sets to an irreversible

jelly which can easily be washed with water, sheeted, and dried. D. F. TWISS.

Manufacture of synthetic rubber. I. G. FARBER and A.-G. (B.P. 283,840—1, 16.1.28. Ger., [A] 14. and [B] 15.1.27).—In the conversion of butadiene hydrocarbons into rubber the process of polymerisation is expedited and a superior product obtained by (A) emulsifying the butadiene compound in an aqueous medium and effecting polymerisation in the presence of oxygen and/or of a substance, *e.g.*, a perborate or percarbonate, capable of yielding oxygen, or (B) polymerising in an atmosphere of hydrogen, nitrogen, or carbon dioxide with the aid of an alkali metal in the presence of an organic hydroxy-compound or ether, *e.g.*, starch, cellulose, a cellulose ether, or superficially oxidised rubber, and, if desired, with the additional presence of an inorganic hydroxide, *e.g.*, sodium hydroxide, and an inert solvent or swelling agent such as aromatic or aliphatic hydrocarbons. D. F. TWISS.

Electrodeposition of rubber. J. PORTER (U.S.P. 1,702,705, 19.2.29. Appl., 24.2.28. U.K., 13.2.28).—The rubber is deposited in sheet form on a rotating bipolar electrode of readily oxidisable material, *e.g.*, lead, separating the latex and the ammonia solution. R. BRIGHTMAN.

Vulcanisation of rubber. NAUGATUCK CHEM. Co., Assees. of S. M. CADWELL (B.P. 302,176, 5.10.27. U.S., 1.11.26).—A vulcanisation accelerator is produced by boiling the condensation product of an amine with an aldehyde containing a chain of carbon atoms, *e.g.*, heptaldehyde-aniline, with acid, *e.g.*, hydrochloric acid; the reaction product is precipitated from aqueous solution by sodium hydroxide. D. F. TWISS.

Manufacture of rubber articles. NAUGATUCK CHEM. Co., Assees. of E. HAZELL (B.P. 295,700, 23.12.27. U.S., 19.8.27).—In making rubber articles from latex, the rate of deposition of the rubber on a porous former is accelerated by increasing the filterability of the latex. This is effected by the presence of suitable substances, *e.g.*, colloidal sulphides or polysulphides, which may be formed *in situ*, *e.g.*, by the addition of a metallic oxide and a soluble sulphide such as zinc oxide and sodium polysulphide. The rate of deposition may be further increased by dilution, agitation, and heating. D. F. TWISS.

Production of articles made of fabric coated, proofed, or impregnated with organic materials, and of sheetings of organic materials. DUNLOP RUBBER Co., LTD., and G. W. TROBRIDGE (B.P. 309,391, 9.1.28).—Fabric or woven material is impregnated or covered with organic material, *e.g.*, rubber, in aqueous dispersion, by dipping; deposition of the organic material is facilitated by coating or impregnating the material, or the porous former on which it may be fitted, with solutions of substances, *e.g.*, alum, acetic acid, or calcium chloride, capable of effecting agglomeration of the dispersed organic material. When a porous former is used, deposition may be aided by imparting a pressure difference between the inside and outside. D. F. TWISS.

Manufacture of products of or containing rubber

and/or rubber-like substances. ANODE RUBBER Co. (ENGLAND), LTD. From P. KLEIN (B.P. 309,630, 14.1.28. Cf. B.P. 283,871; B., 1929, 295).—Articles of rubber, gutta-percha, etc. are manufactured from natural or artificial dispersions by building up the products from parts containing unmasticated rubber and different proportions of masticated rubber or similar material, plastic or tacky in character. D. F. TWISS.

Manufacture of liquefied rubber composition. H. P. BUTLER (U.S.P. 1,710,470, 23.4.29. Appl., 8.4.27).—See B.P. 274,742; B., 1927, 757.

Manufacture of rubber conversion products. B. F. GOODRICH Co., Assees. of H. L. FISHER (B.P. 282,778, 23.12.27. U.K., 28.12.26).—See U.S.P. 1,642,018; B., 1927, 917.

Manufacture of rubber goods. P. KLEIN and A. SZEGVÁRI, Assrs. to ANODE RUBBER Co., LTD. (U.S.P. 1,708,181, 9.4.29. Appl., 5.6.25. U.K., 13.2.25).—See B.P. 254,765; B., 1926, 797.

Manufacture of rubber or the like [hollow] articles. C. MACINTOSH & Co., LTD., S. A. BRAZIER, and G. F. THOMPSON (B.P. 310,602, 22.2.28).

Manufacture of goods of india-rubber or the like, *e.g.*, hot-water bottles. CAMPBELL, ACHNACH, & Co., LTD., J. GILLAN, J. MCP. SWAN, and T. GARSTANG (B.P. 310,629, 3.4.28).

Heat-insulating material (B.P. 309,662). **Concentration or drying of liquids** (B.P. 307,315).—See I. **Insulating materials** (B.P. 309,851).—See XI.

XV.—LEATHER; GLUE.

Analyses of some English bookbinding leathers. R. W. FREY, L. R. LEINBACH, and E. O. REED (J. Amer. Leather Chem. Assoc., 1929, 24, 190—196).—Of 23 samples of goatskin, sheepskin, calfskin, cowhide, and pigskin leathers, only 5 had a breaking strength below 2000 lb./in.²; the free mineral acid content of four of these was more than 0.5%. No relationship was observed between strength and the direction of the skin. On analysis the leathers gave (%) total ash 0.2—0.8, insoluble ash 0.2—0.9, petroleum spirit extract 0.9—11.1, water-soluble matter 2.4—14.6, combined tannin 24.0—39.4, hide substance 48.0—62.9, uncombined tannin 0.8—7.1, and had acidity (Procter-Searle) 0.0—2.2, p_H of water extract 2.4—4.7. Six leathers were tanned with mixtures of pyrocatechol and pyrogallol tanning materials; the rest with pyrogallol tannins alone. Of 13 leathers specified as "free from injurious acids," 9 contained no acid, and the rest only 0.2—0.3%. Most of the leathers were overtanned, and their grease content was low; the majority, however, complied with the chemical requirements of durable bookbinding leathers. D. WOODROFFE.

Modified shaking method for analysing tannins and the Darmstadt apparatus. G. BALDRACCO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1929, 7, 82—94).—The advantages of this method (B., 1920, 123, 460, 496, 523), in conjunction with Stiasny's apparatus (Collegium, 1928, 383; cf. Parker, B., 1929, 140), are confirmed, and

the following procedure is recommended: 6.5 g. of dry hide powder, previously chromed with chrome alum, are introduced, together with 75 c.c. of the tannin solution to be examined, into the Darmstadt apparatus, which is closed with its two covers and shaken for 15–20 min. The liquid is then withdrawn by suction and the residue shaken for 15 min. with a further quantity of 75 c.c. of the tannin solution. The liquid is transferred to a clean, dry dish, and filtered through filter-paper, 50 c.c. of the clear liquid being used for the determination of the non-tannins. T. H. POPE.

Analysis of [tannery] beam-house liquors. J. H. HIGHBERGER and E. K. MOORE (*J. Amer. Leather Chem. Assoc.*, 1929, 24, 68–76).—(a) *Total nitrogen*. The Kjeldahl–Gunning method is used. (b) *Total protein-nitrogen*. A sample (50 c.c.) is acidified to phenolphthalein with 2*N*-hydrochloric acid, mixed with 150 c.c. of a sodium chloride-chloroacetic acid reagent, left overnight, the precipitate washed with diluted reagent, and the nitrogen determined as above. (c) *Non-protein-nitrogen*. This value is given by $a - b$. (d) *Heat-coagulable protein-nitrogen*. 100 c.c. of the sample are acidified with acetic acid, weighed, heated to boiling for 5 min., cooled, made up to original weight with distilled water, shaken, and filtered. By making a Kjeldahl determination for total nitrogen on an aliquot portion of the filtrate and deducting the result from the corresponding value for (a) the value of d is obtained. (e) *Volatile bases*. A measured volume of the liquor is distilled under reduced pressure with excess of lime, the distillate collected in a known quantity of 0.1*N*-sulphuric acid, using the Van Slyke apparatus, and the excess acid is determined by titration with standard alkali. (f) *Ammonia and amines*. The titrated distillate from (e) is transferred to a 500 c.c. graduated flask, diluted to 480 c.c., 5 c.c. of 20% sodium hydroxide solution and 5 c.c. of 30% sodium carbonate solution are added, the mixture is made up to 500 c.c., poured into a brown glass bottle, and 0.1 g. of yellow mercuric oxide is added for each c.c. of 0.1*N*-acid equivalent to the total volatile nitrogen present; the bottle is then stoppered, covered with a black cloth, shaken for 1 hr., allowed to remain overnight, and the supernatant liquor forced through a cotton filter. 400 c.c. of the filtrate are distilled into 0.1*N*-sulphuric acid and the acid neutralised represents the volatile, aliphatic amino-nitrogen. The free ammonia-nitrogen is the difference between this value and that of the total volatile nitrogen. (g) *Chlorides*. 20 c.c. of the filtered soak or lime liquor are made up to 100 c.c. with distilled water, and to 15 c.c. of the mixed liquids 25 c.c. of 0.1*N*-silver nitrate and 10 c.c. of nitric acid are added; after being heated to just below boiling for 2–3 hrs., the mixture is cooled, diluted to 100 c.c. with distilled water, and the excess silver nitrate determined by titration with 0.1*N*-potassium thiocyanate. The presence of sulphides in the lime liquor does not interfere with this method.

D. WOODROFFE.

See also A., May, 533, **Shaker for Clark hydrogen electrode vessel** (CAMERON and SHEARER). 574, **Non-tans in extract of badan root** (TSCHITSCHIBABIN and others).

PATENTS.

Manufacture of imitation doe-skin [from rubber-coated fabrics]. C. E. SIMPSON (B.P. 309,675, 18.2.28).

Nitrocellulose solutions (B.P. 308,230).—See XIII.

XVI.—AGRICULTURE.

“Single value” soil properties: significance of certain soil constants. II. **Natal soils.** J. R. H. COUTTS (*J. Agric. Sci.*, 1929, 19, 325–341; cf. Keen and Coutts, *B.*, 1928, 905).—Determinations have been made of the sticky point, loss on ignition, clay content, and water content in equilibrium with an atmosphere of 50% R.H., of a number of Natal soils, and it is shown that the first and second, and the third and fourth pairs of quantities exhibit close correlation. With reference to the Keen–Raczkowski box data, correlation exists between pore space and clay content, and between volume swelling and water retained, and hence with sticky point. E. A. LUNT.

Formation and composition of humus matter [in soil]. K. LIESCHE (*Landw. Jahrb.*, 1928, 68, 435–488; *Bied. Zentr.*, 1929, 58, 156–158).—Comparison is made of humus from various sources, both natural and artificial. Humus from glycine, tyrosine, cystine, and dextrose contains more carbon and less nitrogen than the natural product from green-manuring. In nitrification experiments the behaviour of artificial humus was generally similar to that of the natural material, although tyrosine- and cystine-humus were slightly inferior. Examination of the carbon dioxide production from humus in soil showed no marked difference between the natural and artificial materials. The permanganate consumption of humus in acid and alkaline solutions is not directly proportional to the carbon content. Natural humus favoured nitrogen fixation by azotobacter, but with the exception of cystine-humus this was not the case with artificial samples. During humus formation the production of yellow and brown substances from dextrose and nitrogen compounds takes place earlier in the presence of soil organisms than in their absence. The slow nitrification of natural humus indicates that its nitrogen constituents are neither adsorbed nor combined as ammonium compounds. The existence of a mixture of nitrogen-free and nitrogen-containing substances in natural humus is suggested.

A. G. POLLARD.

Unsaturated soils. N. P. REMESOV (*Z. Pflanz. Düng.*, 1929, 13A, 228–242).—In the examination of unsaturated soils the adsorbed hydrogen ions are not completely removed from the clay complex when the reaction of the soil suspension is adjusted to p_H 7. No essential difference exists between exchange and hydrolytic acidity, the more complete displacement of hydrogen ions by treatment of the soil with salts of weak acids being due entirely to the more alkaline reaction of their solutions. To determine the lime requirement of soil a series of samples (10 g.) are treated with 25 c.c. of *N*-calcium chloride, and 0.004*N*-calcium hydroxide is added in increasing proportions through the series. The samples are shaken and after 24 hrs. the p_H values are determined by the quinhydrone electrode. The

lime requirement is calculated from the amount of lime necessary to bring the final p_H of the soil suspension to 7.0. Parallel experiments using chlorides and hydroxides of bases other than calcium yielded slightly different results, but the substitution of calcium acetate for the chloride did not alter the figures. A. G. POLLARD.

Determination of the saturation condition of soils. H. KAPPEN (Fortsch. Landw., 1928, 3, 1009; Bied. Zentr., 1929, 58, 147—148).—Exchangeable bases in soil are determined by treating 50 g. of soil with 250 c.c. of 0.1N-hydrochloric acid and shaking for 1 hr. After 24 hrs. the mixture is filtered and the filtrate titrated with 0.1N-alkali. From the acid consumption is calculated the mg.-equivalent of dissolved bases per 100 g. of soil. The base requirement for the complete saturation of the soil is determined electrometrically (Jensen) to p_H 8.5. A. G. POLLARD.

Determination of the nutrient content of arable soils by Mitscherlich's method. OPTIZ and RATHSACK (Landw. Jahrb., 1928, 68, 321—348; Bied. Zentr., 1929, 58, 145—147).—Considerable lack of agreement exists between the soil nutrient content as determined by pot experiments and those obtained in field trials, the latter showing greater values in the majority of cases. Nitrogen added to the soil by green manuring and by the growth of legumes is not registered in pot experiments by Mitscherlich's method. The "effect factor" for nitrogen was smaller in reference to grain yields than straw yields. In sand cultures the effect factor for nitrogen in ammonium nitrate was greater than that for ammonium sulphate, and fluctuated with the nature of the phosphatic manure used. It is also smaller after heavy green manuring. A. G. POLLARD.

Effects of carbon disulphide treatment of soil for the Japanese beetle on the abundance of micro-organisms and on the ammonia and nitrate content. W. E. FLEMING (Soil Sci., 1929, 27, 153—158).—Treatment of soil with carbon disulphide at the rate of 1 lb. per cub. yd. stimulated the growth of fungi without affecting the numbers of bacteria present. Accumulation of ammonia occurred. In poorly-drained land plant injury resulting from the use of carbon disulphide emulsions is considered to be largely the outcome of detrimental effects on micro-organisms, the decrease in nitrate production, and the accumulation of ammonia. In well-drained soils the effects of carbon disulphide emulsions are similar to those following fumigation. The importance of aerating soils after fumigation is noted. A. G. POLLARD.

Comparison between the culture methods of Mitscherlich and Wiessmann [for determining nutrient values of soils]. H. WIESSMANN (Z. Pflanz. Düng., 1929, 13A, 205—208).—A reply to Behrens (B., 1929, 183). A. G. POLLARD.

Determination of organic carbon in soils. G. W. ROBINSON, W. MCLEAN, and R. WILLIAMS (J. Agric. Sci., 1929, 19, 315—324).—The method used consists in determining the amount of sulphur dioxide produced in the ordinary Kjeldahl digestion by absorption in standard iodine solution. The results for British soils average $89.6 \pm 1.03\%$ of the figures obtained by combustion.

The advantages claimed for this method are its applicability to carbonate soils without the necessity for any correction for inorganic carbon, and its technical simplicity. E. A. LUNT.

Availability of potash in a typical Mauritius soil. N. CRAIG and R. LINCOLN (J. Agric. Sci., 1929, 19, 397—403).—The application of potassium salts to Mauritius lateritic soil effects a gradual increase in the amount of non-available potassium oxide, potassium sulphate being more potent in this respect than potassium nitrate. Conversely, the application of molasses to the soil produces a decrease in the amount of non-available potassium. E. A. LUNT.

Nutritive value of pasture. IV. Influence of intensity of grazing on yield, composition, and nutritive value of pasture herbage. II. H. E. WOODMAN, D. B. NORMAN, and J. W. BEE (J. Agric. Sci., 1929, 19, 236—265; cf. B., 1928, 381).—Pasture grass obtained under a system of 3-weekly cuts is slightly less rich in digestible protein than grass cut fortnightly, but equal in respect of total digestible organic matter and of starch. The total yield over the whole season obtained by cutting at 3-weekly intervals is 62.3% greater than that obtained by weekly cutting. E. A. LUNT.

Influence of feeding on the composition of milk. H. T. CRANFIELD (J. Agric. Sci., 1929, 19, 302—310).—Dried sugar-beet pulp feeding produces a higher yield of milk and of milk solids than mangel feeding. The average composition of the milk is the same on both diets. E. A. LUNT.

Factors affecting yield and quality of milk. I. Age of the cow. R. R. KAY and A. C. MCANDLISH (J. Agric. Sci., 1929, 19, 342—372).—Milk and butter fat production in cows reaches a maximum at 7 years of age, whilst the percentage of fat reaches its maximum at 3 years of age and then remains constant until advanced ages. E. A. LUNT.

Action of carbon monoxide on green plants. M. PADOA and N. VITA (Annali Chim. Appl., 1929, 19, 141—148).—Carbon monoxide is absorbed by alcoholic solutions containing either carotin and chlorophyll or pure chlorophyll (A and B) alone and is expelled from such solutions only by a large excess of carbon dioxide. In presence of the xanthophyll or other ingredients of the alcoholic extracts, carbon monoxide is apparently oxidised to the dioxide. The assimilative power of *Plantago major*, *Lemna minor*, and *Elodea Canadensis* is inhibited by a large excess or sensibly diminished by a small amount of the monoxide, but, even when highly concentrated, the gas does not impede the respiratory processes of these plants. Unlike other anaesthetics, carbon monoxide does not demolish the cell-walls separating enzymes from glucosides, e.g., emulsin from amygdalin in cherry-laurel leaves, and hence does not give rise to formation of hydrocyanic acid. The monoxide inhibits alcoholic fermentation and greatly retards the inversion of sucrose. T. H. POPE.

Soil reaction and the growth of meadow plants. K. E. LANDGRAF (Z. Pflanz. Düng., 1929, 13A, 213—228).—Changes in the reaction and in physical and chemical properties of soil produced by treatment with small quantities of sulphuric acid and sodium hydroxide were

examined. The citric solubility of the soil nitrogen increased with both acid and alkaline treatments; that of phosphorus increased with alkali and decreased with acid; that of potash decreased with both, and that of calcium increased slightly with acid and was not appreciably affected by alkaline treatment. From within the reaction range p_H 4.4—4.7 the water capacity and hygroscopicity of soils increased with both acid and alkali treatment up to maximum points, beyond which the values again decreased. The growth curve in treated soils of *Festuca pratensis* showed maximum points in both the acid and alkaline regions. This is almost entirely the outcome of changes in the solubility of nutrients due to the soil treatments. In respect of soil reaction *Festuca pratensis* exhibits a region of optimum growth over the range p_H 4.0—7.0, within which growth is governed, not by soil reaction, but by the extent to which "growth factors" are effective. The optimum reaction range of the cell sap is p_H 5.95—6.25. Changes of reaction towards neutrality induced by alkaline soil treatment result in an increased calcium content, and *vice versa*. A change of 0.3 p_H unit affected the growth of the plant. No relationship exists between the reaction of the cell sap and that of the soil. The former is governed by the presence of definite ions in the nutrient medium. A. G. POLLARD.

Crop variation. VI. Response of the potato to potash and nitrogen. T. EDEN and R. A. FISHER (J. Agric. Sci., 1929, 19, 201—213).—Experiments are recorded with reference to the effect on crop in the potato of potash manuring in the form of sulphate, chloride, and low-grade salt, in the presence and absence of nitrogenous manures. E. A. LUNT.

Influence of potassium on the structure of fibre cell-walls in fibre plants. F. TOBLER (Z. Pflanz. Düng., 1929, 13A, 208—213).—Potassium sulphate increased the yield of fibre from flax to a greater extent than did "potash salts." Differences in the length of straw were not marked. With potash salts the individual fibre bundles were round in outline, but the sulphate produced more compact, angular, and narrower bundles. Regarding the individual cells, potash salts induced rounder and fuller cells, larger cavities, and thinner walls, whereas potassium sulphate produced larger cells, more angular in section, thicker walled, and more closely united with neighbouring cells. With hemp similar results were obtained, but the effects of the different fertilisers were less accentuated. Following the potash fertilisation of ramie, the loosely arranged fibres in the tissue tend to concentrate into bundles. Potassium sulphate produces thicker walls than potash salts. In general, potash fertilisers induce the formation of smoother fibres, with consequent decreased loss in separating and improved spinning qualities. A. G. POLLARD.

Factors influencing the growth and sugar contents of cane. K. K. RAO (Agric. J. India, 1929, 24, 91—101).—A review of published information.

A. G. POLLARD.

Treatment of beet seed with naphthalene. V. STEHLÍK (Z. Zuckerind. Czechoslov., 1929, 53, 333—339).—Naphthalene mixed with the seed before planting

with the object of checking pest infestation gave negative results. The application had no effect on the stand, yield, or sugar content. J. P. OGILVIE.

Adventitious roots and the supply of nitrogenous and mineral substances to oats. T. TSCHIRIKOV (Z. Pflanz. Düng., 1929, 13A, 201—204).—Comparison of the chemical analyses of oat plants and the development of adventitious roots indicates that the number of the latter present controls the quantity of mineral nutrients absorbed by the plants. A. G. POLLARD.

Effects of hydrogen-ion concentration on rice cultures. S. K. MITRA and L. N. PHUKAN (Agric. J. India, 1929, 24, 109—116).—Rice plants were grown in water cultures the p_H values of which varied from 3.0 to 8.4, and the total length of root produced was taken as a measure of the plant growth. Maximum growth was obtained at p_H 7.9. At p_H 6 growth was below normal, at p_H 3.9 the culture solution was strongly toxic, and at p_H 3.3 growth was inhibited. Evidence is presented that toxicity was due directly to the hydrogen-ion concentration and not to the titratable acidity or alkalinity. A. G. POLLARD.

Leaching-out of autumnal dressings of nitrogenous fertilisers. H. H. NICHOLSON and B. PANTIN (J. Agric. Sci., 1929, 19, 297—301).—Over 50% of a normal autumnal dressing of ammonium sulphate, calcium cyanamide, and rape dust is lost by leaching-out during the winter, the loss being greatest in the case of ammonium sulphate and least with rape dust. The rate of the loss by leaching is dependent on rainfall and temperature. E. A. LUNT.

Pyrethrin I and II; their insecticidal value and determination in pyrethrum (*Chrysanthemum cinerariæfolium*). I. F. TATTERSFIELD and R. P. HOBSON (J. Agric. Sci., 1929, 19, 266—296).—Experiments are described in agreement with those of Staudinger and Ruzicka (B., 1924, 398, 573) showing that pyrethrin I is more highly toxic to *Aphis rumicis* than is pyrethrin II. Two micro-methods for determining pyrethrin are described: (a) from the acids liberated on hydrolysis, (b) by means of the semicarbazone.

E. A. LUNT.

See also A., May, 523, **Development of chlorophyll in seedlings in light of different wave-lengths** (SAYRE). 611, **Soil respiration and activity of forest soils** (FEHÉR). 612, **Root-forming substance** (WENT).

PATENT.

Calcium nitrate-ammonium nitrate (B.P. 310,276). See VII.

XVII.—SUGARS; STARCHES; GUMS.

Colloid chemistry in relation to sugar manufacture. J. BERGÉ (Bull. Soc. chim. Belg., 1929, 38, 31—46).—It has not yet been found possible to extract the sugar from beets without accompanying colloidal matters. Although liming of the diffusion juice at the rate of 100 g. CaO per 100 kg. of beets suffices to produce flocculation of colloids, difficulties of filtration necessitate the addition of very much more lime,

followed by carbonatation. Some of the most promising attempts to reduce the consumption of lime are those in which the diffusion juice, before liming, is mixed with a proportion of unfiltered carbonatation juice and filtered as a means of preliminary purification. The Raffinerie Tirlemontoise has succeeded in drying carbonatation scums to a powdery condition by means of centrifuges of very high speed. Heating carbouated juices to 90–95° has been found to facilitate filtration considerably. The thick juice from the evaporators contains an appreciable amount of precipitated colloids; some factories filter with kieselguhr at this stage, but decolorising carbon is more effective. The effect of colloids in retarding the crystallisation of sugar is discussed, and also their tendency to be obstinately retained by sugar crystals, influencing the colour of the latter. The purity of centrifuged sugars can be much improved by the use of centrifuges of very high centrifugal force, e.g., 1200–7000 times the force of gravity instead of 500 times as usually employed.

J. H. LANE.

p_H Curves [of sugar solutions] following phenolphthalein titrations. V. MAJER (Z. Zuckerind. Czechoslov., 1929, 53, 301–305).—Ten c.c. of liquid (e.g., alkaline beet juice) were titrated with 0.1N-hydrochloric acid, using phenolphthalein as indicator, and continuing the addition to the point at which the pink shade just disappeared. The p_H of the liquid was then determined colorimetrically, using bromothymol-blue or bromocresol-purple; a few more drops of the standard acid were added, and the p_H was again determined. Curves having the additions of 0.1N-acid as abscissæ and the p_H values found as ordinates gave "titration curves" which were characteristic. In sugar solutions containing lime the phenolphthalein titration finished at p_H 7.0, and gave curves which fell sharply before a further addition of 0.1 c.c. of 0.1N-acid had been made. On the other hand, in well-buffered solutions the titration finished at about p_H 8.0, whilst a further addition of 0.4 c.c. of acid could be made before the p_H fell to 6.0.

J. P. OGILVIE.

[Determination of sugar with] alkaline copper solutions [Fehling's solution]. J. DE VILMORIN and E. CAZAUBON (Bull. Assoc. Chim. Sucr., 1929, 46, 54–61).—A discussion of the nature of the reactions involved in the determination of reducing sugars, with special reference to Fehling's original method, the gravimetric method, and the methods of Bertrand, Saillard, and Possoz.

J. H. LANE.

Methyl and ethyl alcohols in sugar beet pulp. M. JEDLIČKA (Z. Zuckerind. Czechoslov., 1929, 53, 293–299).—By boiling beet pulp with water containing lime, methyl and ethyl alcohols were liberated in approximately equal amount, equivalent to a total of about 38.8 c.c. of absolute alcohol per 100 kg. Small amounts were also identified in the condensed waters from the second body of the evaporator.

J. P. OGILVIE.

Determination of the starch value of frozen potatoes. W. EKHard (Z. Spiritusind., 1929, 52, 77–78).—Before the starch value of potatoes which are completely or almost completely frozen can be deter-

mined, thawing and washing in luke-warm water are necessary. On account of their softness, careful handling is necessary, and it is not advisable to attempt to wipe them before weighing. Experience has shown that a deduction of 1% should be made from the starch value of frozen potatoes to allow for the unavoidable loss of sap from the tubers in their thawing and cleaning. The indirect determination of the starch value of frozen potatoes from their content of dry substance, as found by actual drying, is tedious and not more accurate than the use of the potato balance.

J. H. LANE.

Determination of starch by the diastase method. H. COLIN (Bull. Assoc. Chim. Sucr., 1929, 46, 177–179).—Since ordinary hydrolysis by diastase is never complete, it is best to liquefy first by diastase and complete the hydrolysis by hydrochloric acid. Commercial preparations of diastase may be freed from reducing sugars by shaking with methyl alcohol. "Rapidase," prepared from *Tyrophrix*, is very suitable; it has high liquefying and low saccharifying powers. By the use of yeast autolysate in addition to diastase, dextrose is formed by the action of yeast maltase. Preparations such as taka-diastase and "biolase," from *B. subtilis* or a similar organism, also hydrolyse starch to dextrose. The varying proportions of amylose and amylopectin in starches of different origins prevent the employment of physical methods for their determination.

F. E. DAY.

See also A., May, 568, Saponin of sugar beet (REHORST). 612, Sugar from pine wood (HÄGGLUND).

Adhesion etc. of glazes. FOSTER.—See VIII. **Sugar contents of cane.** RAO. **Treatment of beet seed.** STEHLÍK.—See XVI.

PATENTS.

Saturation of sugar juices in the manufacture of beetroot and cane sugars. F. BLANKE (B.P. 283,202, 15.12.27. Ger., 8.1.27).—In an apparatus for the continuous, automatically-controlled saturation of sugar juices with carbon dioxide or sulphur dioxide, the outlet pipe from the bottom of a raw juice container communicates with the lower end of a tall vertical pipe into which, near the lower end, the gas is also injected. In passing up this pipe the juice becomes saturated, and near the upper end it flows through two cross-pipes fitted with an electrometric indicator into a descending pipe which delivers it to another container. Float valves automatically stop the flow of juice in the event of either container becoming too full. If the electrometric indicator shows too low an alkalinity in the saturated juice, a proportion of air is automatically mixed with the injected gas until the correct alkalinity is again indicated.

J. H. LANE.

Desaccharification of syrups and molasses. J. BERGÉ (B.P. 308,867, 14.2.28).—To promote the further crystallisation of sugar from massecuites, or from centrifuged syrups or molasses, these are mixed with pure sugar crystals so that the liquid is distributed as a thin layer over a large area of crystal surface, and the mixtures may be kept under conditions favourable to crystallisation. The final separation of the exhausted syrups from the crystals may be effected by means of

centrifuges of specially high centrifugal force, and the separated crystals may be used again for a similar purpose.

J. H. LANE.

Recovery of sugar from the exhausted sludges or scums of [beet] sugar manufacture. H. CLAASSEN (B.P. 296,985, 24.8.28. Ger., 10.9.27).—The greater part of the sugar at present lost in carbonatation scum cakes, which is considerably more than the usual methods of analysis indicate, may be recovered by mashing the cakes with water in a mixing apparatus, heating to about 80°, and separating the watery liquor from the solid by sedimentation. The watery liquor, containing $\frac{1}{2}$ —1% or more of sugar, may be used for washing later scums in the presses. A saving of at least 0.1% of sugar, calculated on beets, is claimed.

J. H. LANE.

Purification of saccharine liquids. H. WIESE, and ANGLO-SCOTTISH BEET SUGAR CORP., LTD. (B.P. 308,529 21.6.28).—The dark affination syrups obtained by the washing of raw sugars prior to refining are mixed with a proportion of carbonatation or sulphitation cake obtained in the purification of affined sugars in accordance with B.P. 12,642 of 1911 (B., 1912, 653), and heated to about 82° and filtered. This neutralises any acidity and improves the quality of the raw sugars and molasses obtained from the affination syrups. By applying similar treatment to solutions of the affined sugars their subsequent purification (*loc. cit.*) can be simplified by the omission of sulphitation, or of one of the carbonatation stages.

J. H. LANE.

Production of dextrose. R. E. DALY, ASSR. to AMER. MAIZE PRODUCTS CO. (U.S.P. 1,708,940, 16.4.29. Appl., 15.8.24).—Sugar liquor derived from converted starch is brought to $d\ 1.34$ — 1.41 , cooled to 49°, seeded with dry crystals, cooled to 38°, introduced into a crystalliser containing wet seed crystals in amount equal to half of the introduced liquor, agitated slowly while the temperature is reduced to 32—35°, and kept at this temperature till permeated with crystals. The temperature is raised to 43—49° to cause the finer crystals to dissolve, again lowered to 32—35° to allow the unmelted crystals to grow, and kept at this temperature for 12 hrs. with slow agitation of the liquid. Finally the crystals are centrifuged from the mother-liquor.

W. J. BOYD.

Alcohol (U.S.P. 1,709,610).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

[Starch] liquefying and saccharifying powers of [German] malts of the 1928 harvest. H. LÜERS and F. WIENINGER (Woch. Brau., 1929, 46, 173—174).—The amylases of two malts, I and II, from different barleys but of similar appearance and growth, were followed during flooring and kilning. In both the saccharifying amylase developed normally, but though I showed a moderate development of liquefying amylase, II was decidedly lacking in this respect. The saccharifying and liquefying powers are evidently independent. Since I, of colour 0.55 (c.c. of 0.1N-iodine) and diastatic power 96.5 (Windisch-Kolbach), saccharified in 35 min., whilst II, of colour 0.35—0.40 and diastatic power 103.5, required 40 min., it is considered probable that

the slow saccharification characterising these malts is due to low liquefying power. No difference could be detected between the starches of the malts in question.

F. E. DAY.

Alteration of hemicellulose-splitting enzymes during flooring and kilning [of malt]. H. LÜERS and L. MALSCH (Woch. Brau., 1929, 46, 163—164).—Contrary to the finding of Lüers and Volkamer (B., 1928, 281) for high enzyme concentrations, at low concentrations and short durations of action the degree of hemicellulose hydrolysis is proportional to the amount of cytase. Taking the original cytase content of a barley, measured by its action on elder-pith xylan, as unity, the activity during malting decreased to 0.63 on the second day on the floor, increased steadily to 2.59 at withering, and decreased to 0.59 on the kiln.

F. E. DAY.

Improvement of brewing waters containing much [calcium] carbonate by lactic acid. V. KOUDELKA (Woch. Brau., 1929, 46, 164—170, 174—178).—Laboratory experiments show that though reduction of temporary hardness by the action of lime, with addition of calcium sulphate or chloride, effects some improvement, much better results are obtained by the addition of lactic acid. A slight excess is not objectionable since, owing to greater protease and phosphatase action, buffering is increased, though too great an excess may restrict diastatic action. The worts from hard waters treated with lactic acid "break" better on boiling and contain more proteins and phosphates. The yeast settles better, and the resulting beers are paler and have a finer though less bitter flavour. The conclusions are supported by the results of two comparative technical brewings.

F. E. DAY.

Detection of coal-tar dyes in red wines. D. MAROTTA (Annali Chim. Appl., 1929, 19, 148—164).—Certain of the methods published for the detection of artificial colouring matters in wines give false results, but the following two methods are sufficient to obtain exact indications:—(1) 100 c.c. of the wine are boiled down to 60 c.c., treated with 2 c.c. of 10% hydrochloric acid, and boiled for 5 min. with about 0.5 g. of defatted embroidery wool. The liquid is decanted off and the wool rinsed in water and boiled for 5 min. with 100 c.c. of water containing 1 c.c. of 10% hydrochloric acid, this treatment being repeated with fresh quantities of the dilute acid until extraction of colour from the wool ceases. The wool is then freed from acid by repeated washing with water and is subsequently boiled with 50 c.c. of water and 10—15 drops of 10% ammonia solution. The alkaline liquid is decanted into another beaker, rendered distinctly acid to litmus with hydrochloric acid, and boiled for a few minutes with a woollen thread about 50 cm. long. A red or pink coloration of the wool shows the presence of an artificial acid dyestuff in the wine. If the colour is weak or uncertain, the wool is boiled with the dilute ammonia and the colour fixed on fresh woollen yarn. (2) 50 c.c. of the wine are mixed with 5 c.c. of 20% ammonia solution and 10 c.c. of perfectly colourless amyl alcohol and shaken gently. The two liquids are separated and the alcohol is washed with water until it loses its alkaline reaction

and is then filtered through a dry paper. If the filtrate is coloured more or less intensely red or yellow, or if it becomes coloured on acidification with acetic acid, the wine contains artificial colouring matters.

T. H. POPE.

Determination of formic acid in vinegar. W. SCHUT (Chem. Weekblad, 1929, 26, 228—229).—One c.c. of the sample is treated with concentrated sulphuric acid, stirred, and heated to 105°, and the volume of carbon monoxide driven off is measured.

S. I. LEVY.

Alcohol from apples. M. L. LÉVY (Bull. Assoc. Chim. Sucr., 1929, 46, 148—155).—Extraction of the juice by hydraulic and continuous presses and by other methods is discussed, the first method being preferred. Details are given of the author's methods of building up a yeast culture and of carrying out the fermentation. The yield is often only 50 litres, at most 57 litres, per 100 kg. of sugar. The residues from the distillation are most difficult of disposal. Types of plant suitable for distillation and rectification are mentioned. The product is used in the preparation of certain liqueurs.

F. E. DAY.

Lime treatment for the preparation of anhydrous alcohol. O. VON KEUSSLER (Z. Spiritusind., 1929, 52, 147—148).—Rectified spirit (about 94%) is stirred with 20—25 kg. of quicklime per hectolitre for about 1½ hrs. in an autoclave at 4—5 atm. at 125—130°. It is distilled from the same vessel, with continued stirring, finishing under vacuum to remove the last traces of alcohol. The powdery residue of slaked lime is either blown out by steam or mixed with water to a sludge. Part of the alcohol distilled from lime contains colloidal lime, which is removed, before re-distillation, by partial (30—50%) neutralisation with sulphuric acid, when the precipitated gypsum carries down the excess of lime. The process occupies only 10 hrs. as against 48 hrs. by the usual method, and the loss is about 2% instead of the usual 5—8% (cf. B.P. 270,612; B., 1927, 541).

F. E. DAY.

Ropiness in wine. G. MEZZADROLI and E. VARETON (Zymologica, 1929, 4, 6—20).—See B., 1929, 373.

Acetone-butyl alcohol fermentation and its application to molasses. G. MEZZADROLI and G. MAGNO (Zymologica, 1929, 4, 21—30).—See B., 1929, 145.

See also A., May, 603, Preparation of bone phosphatase (MARTLAND and ROBISON). 604—5, Specific action of enzymes, and fission of polypeptides (ABDERHALDEN and others). 604—6, Influence of erepsin and trypsin-kinase on polypeptides etc. (ABDERHALDEN and others). 607, Phosphoric esters in alcoholic fermentation (BOYLAND). Equation of alcoholic fermentation (HARDEN and HENLEY). Alcoholic fermentation (KOSTYTSHEV and JEGOROVA). Top yeast (VON EULER and NILSSON). Ergosterol content of yeast (HEIDUSCHKA and LINDNER). Decomposition of fats by bacteria (HAAG). 608, Rennin production (WAHLIN).

PATENTS.

Malt and maltose preparation. A. E. NIENSTADT, Assr. to A. F. STREGER, INC. (U.S.P. 1,209,366, 16.4.29.

Appl., 20.9.23).—An aqueous solution of edible albuminous matter, which is coagulable at moderate temperature, is added to a viscous extract of malt of about *d* 1.38 and the mixture heated to approx. 70° and filtered.

C. RANKEN.

Production of alcohol. J. J. DE LA ROZA, Assr. to BAGASSE PRODUCTS CORP. (U.S.P. 1,709,610, 16.4.29. Appl., 22.3.27).—Burned green sugar cane is allowed to remain under ordinary atmospheric conditions until the contained sugar is inverted. The liquid content is extracted and distilled after fermentation.

C. RANKEN.

Treatment of air for cooling and drying [brewing] casks and such like. CHARRINGTON & Co., LTD., and A. C. REAVENALL (B.P. 307,371, 2.12.27).

Dietetic preparations (B.P. 309,097).—See XIX.

XIX.—FOODS.

Report of the Government Laboratory at Amsterdam for 1928. J. STRAUB (Chem. Weekblad, 1929, 26, 212—215).—Work done in the various departments of the laboratory is summarised. In milk examination comparison was made of the Ringeling and "pegallac" methods for detection of *B. coli*, and many cases were observed in which one method gave positive, the other negative, results. The plate method and the reductase method of counting bacteria were not found to give parallel results in examination of some 200 samples. Experiments were in hand with silver chromate paper to distinguish milk from inflamed udders. In examination of edible oils and fats, data are being collected for the preparation of m.-p. and setting-point diagrams so that curves prepared with the aid of standard apparatus may be used in detecting adulteration. Work has been published on fluorescence of oils and fats. The Meihuizen apparatus for determination of water is not so suitable for milk products as for starch and flour. An earlier investigation into calorific value, albumin content, and price of the average diet for different sections of the population has been resumed. Work has been published on analysis of mixtures of starch and sugar syrups.

S. I. LEVY.

Influence of cooking on the nutritive value of food. N. JARUSSOVA (Biochem. Z., 1929, 207, 395—404).—Feeding experiments with rats show that heating food for several hours at 100° does not noticeably affect its nutritive value.

P. W. CLUTTERBUCK.

Correlation studies with diverse strains of spring and winter wheats: inheritance of quality. H. K. HAYES, F. R. IMMER, and C. H. BAILEY (Cereal Chem., 1929, 6, 85—96).—Data from milling and baking trials of wheats have been studied by means of correlation coefficients in order to determine the practical value of selecting new strains by means of determinations of kernel texture and protein content. There is no indication that such determinations are valuable as a means of obtaining high-quality wheat strains.

W. J. BOYD.

Relation of hydrogen-ion concentration and buffer value to the baking quality of flour. II.

E. A. FISHER and P. HALTON (Cereal Chem., 1929, 6, 97—114; cf. B., 1929, 262).—It is confirmed by baking tests that the p_H value is not a factor of great importance in determining dough or loaf quality even when the variations in p_H are much greater than those produced by prolonged over-fermentation. Increased hydrogen-ion concentration may produce increased dough toughness, more pronounced flavour, and improved colour of crumb. W. J. BOYD.

Determination of starch in cereals and mill products. C. VON SCHEELE and G. SVENSSON (Tekn. Tidschr., 1928, 58, Kemi, 57—60, 65—72; Chem. Zentr., 1928, ii, 2202).—Comparative experiments show that the method of Lüers and Wieninger is the most accurate; in control work the method of polarisation after treatment with diastase, or Evers' method employing corrections, is recommended. The methods are described. A. A. ELDRIDGE.

Germination, food value, and vitamin-B of rice preserved for 4 years in carbon dioxide and air-tight containers. M. KONDÔ, S. MATSUSHIMA, and T. OKAMURA (Proc. Imp. Acad., Tokyo, 1929, 5, 159—160).—Rice so preserved showed no loss in germination, food value, and vitamin-B content when compared with freshly harvested rice, whilst rice kept in straw sacks over the same period showed a total loss of vitality. C. C. N. VASS.

Bacterial content and keeping quality of milk. H. BARKWORTH, L. S. MEANWELL, and M. G. D. TAYLOR (J. Min. Agric., 1929, 36, 170—173).—Tables compiled from examination of a further 2400 samples in the course of 8 further competitions confirm the deleterious effect of the presence of *B. coli*, and the falling off in keeping quality as bacterial content increases. S. I. LEVY.

F.p. of milk. A. VAN RAALTE (Analyst, 1929, 54, 266—268).—The normal f.p. of unaltered milk with an acidity not exceeding 9 (c.c. of 0.25N-alkali/100 c.c. of milk) is taken as -0.54° to -0.57° . Milk from cows with diseased udders can have f.p. below -0.57° owing to formation of lactic acid (not shown in the acidity). In Holland about 1 sample of milk is analysed yearly per 30 inhabitants, and the f.p. is taken of any milk with solids-not-fat of 8.2 or less in winter or 8.0 in summer. D. G. HEWER.

Analysis of egg paste. M. SETTIMI (Annali Chim. Appl., 1929, 19, 182—188).—The egg albumin in this material is best detected by Leone's method (B., 1925, 687). To determine the presence of yolk, tests should be applied for lecithin by extracting with absolute alcohol and precipitating with cadmium chloride; for cholesterol by extracting with chloroform, saponifying, and using the colour reaction with concentrated sulphuric acid; and for lipochromes by taking advantage of their resistance to decolorisation. The tests are described in detail. T. H. POPE.

PATENTS.

Treatment of grain. H. J. WHITE, Assr. to BETHLEHEM MILLING Co. (U.S.P. 1,706,760, 26.3.29. Appl., 25.2.27).—Grain particles are mixed with potassium carbonate while heating. H. ROYAL-DAWSON.

Manufacture of food or dietetic preparations. G. M. CLARK. From MATRO GES.M.B.H. (B.P. 309,097, 28.12.27).—The rootlets of malted grain or flours made therefrom are mixed with dry or diluted alkalis, alkaline-reacting salts, or lime, and the mixture is added to corn meal; the bitter taste of the material is thereby removed. If it is unnecessary to retain the vitamins the mixture of rootlets and alkali may be roasted for a short time at above 100° . The rootlets may be freed from dirt, sifted, soaked in sufficient alkaline solution, dried at a low temperature, sifted, and ground to a flour suitable for adding to the corn meal. They may also be steamed before treatment. The finished product contains 27—30% of nitrogenous material. W. J. BOYD.

Food product. B. DASS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,708,914, 9.4.29. Appl., 4.10.21).—Dried yeast is incorporated with peanut butter in the absence of sufficient moisture to permit fermentation. H. ROYAL-DAWSON.

Manufacture of food products. CALIFORNIA FRUIT GROWERS' EXCHANGE (B.P. 283,581 and 284,278, 3.1.28. U.S., [A] 15.1.27, [B] 27.1.27).—(A) A mixture of fruit juice, especially of citrus fruits, and milk is agitated to ensure fine subdivision of the curd, passed through a homogeniser, and dried in powder or flake form. (B) A protective colloid, e.g., $\frac{1}{8}$ —2% of pectin, may also be added. W. J. BOYD.

[Non-hygroscopic] food product. CALIFORNIA FRUIT GROWERS' EXCHANGE (B.P. 283,579, 3.1.28. U.S., 15.1.27).—A solution of lactose in fruit juice is dehydrated rapidly to a powder at low temperature. The lactose added must be equal to 10% or more of the weight of fruit juice solids present. Other sugars may be added. W. J. BOYD.

Concentration of fruit juices and other liquids containing aromatic constituents. M. K. SERAILIAN (B.P. 303,956, 8.11.27).—The liquid is evaporated while flowing in a continuous stream, first in a smaller evaporator the aromatic vapours from which are recovered by condensation, and then in a larger evaporator until the required density is obtained. The second evaporator is not necessarily provided with a condenser, but should have a connexion to a vacuum pump. The evaporators differ only in size, and preferably comprise a cylindrical lower part divided by radial walls into a number of segments through which the liquid flows in succession, leaving at the segment next to the point of entry, while evaporation and local circulation are effected by a (steam) heater at the centre. The vapour dome is common to all segments. B. M. VENABLES.

Heat treatment of cereal substances. C. W. CHITTY and D. W. KENT-JONES, Assrs. to WOODLANDS, LTD. (U.S.P. 1,710,472, 23.4.29. Appl., 28.6.26. U.K., 24.7.25).—See B.P. 263,897; B., 1927, 203.

Preparation of mild aromatic yoghurt-curdled milk or sweet yoghurt junket. G. WINKLER, Assr. to A. ZIERER (U.S.P. 1,710,133, 23.4.29. Appl., 18.5.27. Austr., 9.6.26).—See B.P. 272,468; B., 1928, 909.

Cooking retorts. R. A. SINDALL (B.P. 310,643, 17.4.28).

Drying of cereals (B.P. 308,548).—See I. Margarine (U.S.P. 1,707,800).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Mechanism of the [biological] action of colloidal sulphur. R. AGNOLI (Zymologica, 1929, 4, 31—34).—The effects produced in the animal organism by injection of colloidal sulphur appear to be due, not merely to the poisonous action of the resulting hydrogen sulphide, but also to a colloidoclastic influence, which is due to the colloidal nature of the sulphur and is capable of producing a fatal shock.

T. H. POPE.

Oil containing geraniol. B. RUTOVSKI and N. MAKAROVA-SEMLJANSKAJA [with I. V. VINOGRADOVA]. (Riechstoffind., 1928, 3, 140—161; Chem. Zentr., 1928, ii, 2197).—Oil of *Laserpitium hispidum*, M.B., during blossoming contains little alcohol, whilst in the fruit the alcohol seems to accumulate; it is chiefly contained in the fractions having d_{20}^{20} 0.8544—0.8884, $\alpha_D + 8.6^\circ$ to $\pm 0^\circ$, n_D^{20} 1.4720—1.5665 (89.35% of the total). Oil from umbels of ripe or partly ripe fruit yielded geraniol, which is present in the essential oil to the extent of about 40%.

A. A. ELDRIDGE.

Essential oil of flowers of *Polianthes tuberosa*. L. F. ELZE (Riechstoffind., 1928, 3, 154; Chem. Zentr., 1928, ii, 2198).—Eugenol, geraniol, nerol, methyl anthranilate, and methyl and benzyl benzoates are present.

A. A. ELDRIDGE.

Determination of citronellal in Java citronella oil. SCHIMMEL & Co. (Ber. Schimmel, 1928, 20—21; Chem. Zentr., 1928, ii, 2199).—The various methods have been critically examined. In most of the Java citronella oils examined the values obtained by the methods of Dupont and Labaune and of Reclaire and Spoelstra were in accord. Holtappel's modifications of Bennett and Salamon's method give good results if the final result of the titration is not recorded until the following day.

A. A. ELDRIDGE.

Non-heptane constituents of Jeffrey pine. P. A. FOOTE (J. Amer. Pharm. Assoc., 1929, 18, 350—353).—*n*-Octoic, *n*-nonoic, and *n*-decoic aldehydes have been identified in Jeffrey pine oil from California.

E. H. SHARPLES.

Bromometric and thiocyanometric examination of essential oils. II. II. P. KAUFMANN [with H. BARICH] (Arch. Pharm., 1929, 267, 249—267).—The bromometric and thiocyanometric iodine values of many constituents of essential oils very frequently differ, and use has been made of this fact to determine successfully the composition of several arbitrary mixtures. The method was then applied to various essential oils with the following results, the two sets of numbers in each case referring to the bromometric and thiocyanometric iodine values (the latter in parentheses), respectively: aniseed oil 188.8—177.5 (162.6—161.7), caryophyllin oil 335.9—307 (110—127.4), cinnamon oil 185.2—207 (10.07—30.3), juniper oil 352—364 (241.7—217.7), lavender oil 240—266 (141—145), rosemary oil 149.9—180.8 (107—110.8). Calamus oil, peppermint oil, thyme oil, caraway oil, and fennel oil afforded variable values, but in the case of the last-

mentioned oil the thiocyanometric method differentiated between old and fresh samples. *iso*Butylene reacts quantitatively with both bromine and thiocyanogen, but stilbene reacts only with bromine. Winkler's statement (B., 1927, 669) that the bromometric iodine value of an essential oil is characteristic is not strictly true, but in certain cases it is of value.

S. COFFEY.

See also A., May, 528, **Ultra-violet light in quantitative chemical examination** (EISENBRAND). 553, **6-Amino-*m*-cresol and derivatives** (BOGERT and CONNITT). 576, **γ -Pyrrolidino- and γ -pyrrolino-propyl benzoates** (ANDREWS and McELVAIN). 577, **Piperidine derivatives** (BOLYARD and McELVAIN). **Local anæsthetics** (MARVEL and SHELTON). 583, **Test for ephedrine and related compounds** (CHEN). 584, **Microchemical reactions of cinchonine, cinchonidine, quinine, and quinidine** (WAGENAAR). **Reactions of morphine** (EKKERT). 600, **Basic titanium salicylate** (PICHON). 613, **Corn-silk** (NOGUCHI). **Determination of nitrate content of tobacco** (ANDREADIS).

PATENTS.

Manufacture of a therapeutically active iron preparation having yeast as its basic substance. C. MASSATSCH, Assr. to MATRO GES.M.B.H. (U.S.P., 1,710,584, 23.4.29. Appl., 21.12.27. Ger., 12.11.25).—See B.P. 304,895; B., 1929, 303.

Manufacture of arylazodiaminopyridines useful as bactericides. I. OSTROMISLENSKY, Assr. to PYRIDIUM CORP. (Re-issue 17,281, 23.4.29, of U.S.P. 1,680,108, 7.8.28).—See B., 1928, 837.

Viscosity of oils (B.P. 307,602).—See I. **Concentration of aromatic liquids** (B.P. 303,956).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Substances activating or determining the sensitivity of silver salts embedded in gelatin. E. CALZAVARA (Bull. Soc. Franç. Phot., 1929, 16, 56—59).—Doubt is expressed as to the conclusiveness of the proofs put forward for the pre-existence of compounds such as thiosinamine, cystine and its decomposition products, in naturally active gelatins. Up to the present no specific microchemical tests of sufficient sensitivity have been devised for the detection of either of these types of compounds *in situ* in the gelatin. Sullivan's test for cystine, using the sodium salt of 1:2-naphthaquinone-4-sulphonic acid, although specific, is too insensitive since it will only detect 1 pt. of cystine in 2500 pts. of water. A study of the variations in the sensitometric characteristics (gamma, inertia, etc.) of a standard emulsion, produced by the addition of these compounds during the ripening period, may afford a possible method for their identification.

J. W. GLASSETT.

Anti-fogging and anti-sensitising effects. S. E. SHEPPARD (Phot. J., 1929, 69, 206).—A reply to criticisms by Hamer and Price of the author's paper (B., 1929, 151).

J. W. GLASSETT.

See also A., May, 522, **Coloration of thin layers of silver iodide in light** (JENCKEL). **Theory of the**

photographic process (KIESER). Photographic effects with silver bromide emulsions (TOLLERT). Coloration produced by dyes and silver halides (STEIGMANN). 535, X-Ray photographs (EBERT).

PATENTS.

Röntgen-ray photography. Röntgen diaphragm. K. MAYER (B.P. 286,283 and 309,529, [A, B] 11.10.27).—Two diaphragms for the stopping of secondary radiation from the object or scatter from the fluorescent screen are described, of which (A) consists of finely-divided silver particles embedded in gelatin, prepared by development of ordinary sensitive plates or films which have been exposed to a parallel beam of X-rays; and (B) comprises a thin apertured screen of material opaque to X-rays, which may be in the form of a suitably slotted, endless band arranged to move over rollers past the plate. J. W. GLASSETT.

Photographic printing. R. FRITSCH (B.P. 309,747, 27.4.28).—The ordinary blue-print image of Turnbull's blue is converted into a more stable green-coloured uranium compound by treatment after washing with a mixture of the following solutions: (a) uranium nitrate, 10 g., water, 1 litre; (b) potassium ferri-cyanide, 10 g., water, 1 litre; (c) 100 c.c. of acetic acid. J. W. GLASSETT.

Production of photographs in natural colours. W. LANGGUTH and C. HUMMEL (U.S.P. 1,710,455, 23.4.29. Appl., 29.8.27. Ger., 9.7.26).—See B.P. 274,129; B., 1928, 503.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Rendering explosives non - deliquescent. L. J. W. JONES (U.S.P. 1,706,668, 26.3.29. Appl., 21.3.27).—The ingredients, including commercial impurities such as magnesium and calcium chlorides, are mixed with dehydrated sodium carbonate and ground. After drying, the mixture is coated with a mixture of liquid nitrotoluenes. S. BINNING.

Recovery of explosives from shells. J. H. ROBERTS, Assr. to F. W. OLIN (U.S.P. 1,706,938, 26.3.29. Appl., 13.11.26).—Amatol (80 : 20) is recovered from shells by dissolution in 100% of hot 95% alcohol. On cooling the solution the resultant explosive crystals may be recovered in a non-compact form. S. BINNING.

Manufacture of fuse compositions. B. W. FOSTER, A. G. WHITE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,625, 13.1.28).—Pitch or a mixture of pitch and sulphur is heated and cooled, after which the salt-petre and other ingredients (if any) are added without further heating. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Water treatment to prevent embrittlement [in steam boilers]. F. G. STRAUB (J. Amer. Water Works' Assoc., 1929, 21, 511—523).—Embrittlement in high-pressure steam boilers, manifesting itself as cracks in highly stressed areas, e.g., seams and joints, is thought to be due to the presence of sodium hydroxide in the

boiler water as a result of the decomposition of sodium carbonate under the conditions attained, the carbon dioxide being driven off along with the steam. When a proper sodium sulphate to sodium carbonate alkalinity ratio is maintained embrittlement is prevented, and it is suggested that the ratio, suitable for the particular steam pressure of the boiler recommended by the Amer. Soc. Mech. Eng. boiler code should be adhered to, the proportion of sodium sulphate being adjusted by any convenient means. Addition of tannin and phosphate will also inhibit embrittlement, but careful control is necessary to ensure that the proper conditions are maintained within the boiler. C. JEPSON.

Bacterium coli in iced and uniced samples of water. G. ELLISON, H. W. HACKLER, and W. A. BUICE (J. Amer. Water Works' Assoc., 1929, 21, 528—530).—Samples of water sent for examination showed after 20—72 hrs. very little variation in *B. coli* content whether they had been dispatched in iced containers or not. On arrival, the ice was always melted, but the samples had a definitely lower temperature in each case. C. JEPSON.

Chlorophenol tastes in waters of high organic content. L. B. HARRISON (J. Amer. Water Works' Assoc., 1929, 21, 542—549).—Superchlorination and subsequent dechlorination with sulphur dioxide may only be successfully employed in preventing chlorophenol tastes when the water is of high organic purity, otherwise side tastes are produced which are considered to be worse. The addition of ammonia prior to chlorination fails to prevent the production of chlorophenol tastes, but is of value in preventing chlorinous odours. It has been shown on a practical scale that with waters of both high and low organic content a chlorinated water free from objectionable tastes can be obtained by the addition of potassium permanganate prior to coagulation and filtration; the amount used must be small enough to be entirely precipitated in the coagulation basins or a musty taste will be caused (cf. B., 1927, 381). C. JEPSON.

Pyrethrin insecticides, TATTERSFIELD and HOBSON. —See XVI. **Brewing waters.** KOUDELKA.—See XVIII.

PATENTS.

Sterilisation of air. J. F. SCHILLER and W. W. WESCOTT (U.S.P. 1,706,594, 26.3.29. Appl., 14.5.24).—Air is compressed to approx. 3 atm. and then expanded by several successive stages and in minute streams to approx. 2½ atm. H. ROYAL-DAWSON.

Sewage disposal system. R. M. BIRNBACH (U.S.P. 1,708,864, 9.4.29. Appl., 8.6.25).—Sewage is passed through a number of closed cells in series, under strictly anaerobic conditions, each cell being fitted with a vent through which any air entrained with the sewage may be withdrawn. C. JEPSON.

Chambers for the fermentation of offal, garbage, and like manurial matter. M. VALTANCOLI (B.P. 310,402, 25.1.28).

Respirators. R. E. LANE, and CHLORIDE ELECTRICAL STORAGE Co., LTD. (B.P. 309,770, 26.5.28).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 5, 1929.

I.—GENERAL; PLANT; MACHINERY.

Nomography. XIV. O. LIESCHE (Chem. Fabr., 1929, 170—171).—This chart, constructed exactly as No. 13 (B., 1929, 305), permits of immediate reading of English weight units (troy and avoirdupois) into metric units, and *vice versa*.

S. I. LEVY.

Heat transfer in recuperators. TERRES and BESECKE.—See II. **Synthetic resins as construction materials.** KALMAN.—See XIII.

PATENTS.

Furnace for treatment of materials. E. B. THORNHILL, Assr. to THORNHILL-ANDERSON Co. (U.S.P. 1,705,039, 12.3.29. Appl., 1.11.26).—A furnace has an outer roof and inner arches under the roof which support heating elements.

B. M. VENABLES.

Regulating the firing of furnaces. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 299,885, 31.10.28. Ger., 4.11.27).—Oxygen is added to the combustion air in a proportion which increases with increase of load. The rate of fuel firing is also increased, but the introduction of oxygen abolishes the time lag between the addition of fuel and consequent increase of heat. The quantity of air admitted may either remain constant at all loads or be regulated inversely to the oxygen.

B. M. VENABLES.

Recuperators for furnaces. W. H. FITCH (B.P. 282,443, 17.12.27. U.S., 18.1.26).—The air receiving heat flows in one line through several banks of tubes, expanding between each bank into chambers the width of which is alternately larger and smaller than the length of the tubes, the wider chambers being used for withdrawal of tubes when replacement is necessary. The waste gas giving up heat flows in zig-zag fashion transversely to and outside the tubes in as many passes as there are banks of tubes, the general result being that the gases and air enter (and leave) at opposite ends of the recuperator to each other. The tubes may have metallic rods threaded through them so that the streams of air are annular. The tubes may be made of silicon carbide.

B. M. VENABLES.

Mercury boilers. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. J. NERAD (B.P. 293,756, 5.7.28. U.S., 11.7.27).—To avoid the high cost of pure mercury in a boiler in sufficient quantity to allow a good circulation, an amalgam is used, *e.g.*, of lead, which is liquid at the temperature at which the boiler operates, only mercury being vaporised. The main heating surface of the boiler preferably consists of double concentric tubes, the inner one being partly jacketed with heat-insulating medium, alternating, if desired, with heat-conducting blocks to permit enough heat to pass into

it to keep the amalgam liquid but to avoid excessive heat-transfer, which would interfere with the circulation.

B. M. VENABLES.

Heat interchanger of the plate type. G. M. G. DE BEAUVAIS, Assr. to G. J. PRAT (U.S.P. 1,705,471, 19.3.29. Appl., 12.6.24. Belg., 13.6.23).—Channel iron is placed between the plates alternately at the horizontal and vertical edges, in each case with the flanges outwards. The plates and flanges are held together with elastic riders or U-shaped elements, no other clamping or welding being used.

B. M. VENABLES.

Heat exchangers and manufacture of tubes provided with gills. CARRIER ENGINEERING Co., LTD., and W. H. OWEN (B.P. 309,845, 16.1.28).—Gilled tubes are constructed from thick-walled tubing by cutting the gills out of the solid, the ends of the tubes being left of slightly larger diameter than the gills so that the tubes may be withdrawn through the tube plate. If it is desired to secure the tubes in the plate by expanding them, the thickness of the ends is reduced on the inside.

B. M. VENABLES.

Effecting heat exchange. LA MONT CORP., Assees. of W. D. LA MONT and A. F. ERNST (B.P. 278,704, 1.10.27. U.S., 7.10.26).—In a heat exchanger, a fluid stream is confined in passages (with heat-transferring surfaces) of which the cross-section is varied to maintain the fluid above the critical velocity and give it a substantial "ranging" motion. The walls confining the fluid converge in the direction of the cooler zone. To avoid excessive draught losses, spacing of the surfaces is not reduced proportionately to the reduction of volume of the gases, but is reduced sufficiently to maintain at least the critical velocity.

B. M. VENABLES.

Apparatus for carrying out exothermic gas reactions, regenerating heat, and cooling the walls of the said apparatus. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 306,054, 15.9.27).—Two forms of apparatus are described in which the incoming gas for the reaction keeps the outer pressure-retaining wall cool and is preheated by heat conducted from the reaction chamber or chambers. The gas is caused to follow a sinuous course while in the preheating stage.

B. M. VENABLES.

Absorption apparatus [for exothermic reactions]. G. B. TAYLOR, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,708,685, 9.4.29. Appl., 8.4.27).—A tower constructed to withstand raised pressures comprises a bottom section having a liquid outlet and a gas inlet above it, a top section having a gas outlet and a liquid inlet, and a series of alternating absorption sections fitted with bubbler-cap trays etc. and cooling sections

comprising vertical tubes in contact with a cooling medium.
L. A. COLES.

Carrying out exothermic catalytic chemical reactions under pressure. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE & L'EXPLOIT. DES PROC. G. CLAUDE, ASSEES. of A. T. LARSON (B.P. 288,577, 11.4.28. U.S., 12.4.27).—The apparatus comprises a double-walled vessel having an outer pressure-resisting wall and a lighter inner one containing the catalyst. Incoming fresh gases are passed through the space between the walls, then mixed with gases that contain products of the reaction, and the whole mixture is allowed to pass through the catalyst.
B. M. VENABLES.

Apparatus for chemical reaction on heated solid material. T. GRISWOLD, Assr. to DOW CHEM. CO. (U.S.P. 1,705,614, 19.3.29. Appl., 27.1.27).—An apparatus in which to conduct reactions between gases or vapours and solids comprises a vertical retort below which is a chamber divided by a horizontal partition into two parts, the lower of which serves as a vaporiser and the upper as a receptacle from which ash or the treated solid material may be removed. The apparatus is particularly adapted to the manufacture of carbon disulphide.
A. R. POWELL.

Thermostats. R. MACLAREN (B.P. 307,135, 21.12.27).—A tube of metal which expands by heat surrounds a rod of inexpandible metal; the latter operates the short arm of a lever, the long arm of which operates electrical contacts.
B. M. VENABLES.

Treatment of discrete materials with gaseous media, particularly applicable to drying. R. V. FARNHAM (B.P. 308,074, 10.3.28).—Powdered material falls in a zig-zag course through a vertical casing counter-current to a drying gas. The material is guided by cellular rotary retarders (shaped like the spokes of a wheel in cross-section) which make substantially gas-tight joints with the casing alternately at either side, a free space being left for the current of gas at the other side of any retarder, which is also the discharge side for the powdered material.
B. M. VENABLES.

Dryer. M. D. JONES, Assr. to FULLER-LEHIGH CO. (U.S.P. 1,709,351, 16.4.29. Appl., 22.11.26).—The material under treatment passes down a casing into a hopper below fitted with a discharge spout at the lower end. The casing contains means for heating the material and a distributing device co-axial with the casing, the axis of the hopper being parallel to but at one side of that of the casing.
L. A. COLES.

Drying processes and plant. J. G. OLSSON and F. I. E. STENFORS (B.P. 309,331, 18.5.28).—In drying plants where the material is heated directly or indirectly from hot surfaces and the evaporated moisture condensed on cold surfaces, a substantial amount of the latent heat of the moisture is recovered by using the same medium as a heating agent for the hot, and cooling agent for the cold, surfaces, each stage being graduated. The material may be caused to travel longitudinally through the dryer by such means as trucks, and air or gas is caused to circulate transversely over the material and over longitudinal heating and cooling pipes. The medium in the pipes is heated, introduced to the hottest

zone, and travels longitudinally first through the heating pipe and becomes cooler, then (after supplementary cooling if necessary) back through the cooling pipe and becomes hotter.
B. M. VENABLES.

Drying apparatus. N. TESTRUP, T. GRAM, O. SÖDERLUND, and TECHNOCHEMICAL LABS., LTD. (B.P. 306,200, 21.11.27).—In apparatus where material is dried, while borne in a moving stream of gas, by heat that is transmitted through the walls of the conduit, every part of such conduit, which includes any bends, separators, etc., as well as the actual drying zone is maintained at a temperature above that of the interior, or at any rate above the dew point of the gas stream, the object being to prevent blockages by moist material.
B. M. VENABLES.

Vacuum drying apparatus. A. E. JONSSON (B.P. 292,105, 11.6.28. Swed., 13.6.27).—The apparatus described in B.P. 232,962 (B., 1925, 655) is modified; e.g., the cylinder preferably oscillates only, and the inlet and outlet for heating medium and outlet to vacuum pump are made by flexible tubes which can twist.
B. M. VENABLES.

Spray-drying plants. J. A. REAVELL (B.P. 306,009, 14.10.27).—The disc used for spraying the liquid has its surface roughened to prevent formation of local dry spots. The roughening may be effected by sand-blasting before hardening the metal, or by turning ribs or serrations, or by building up the disc from a number of nested cups the edges of which form the rough working surface.
B. M. VENABLES.

Drying and grading plants for granular water-containing materials. L. HONIGMANN and F. BARTLING (U.S.P. 1,705,617, 19.3.29. Appl., 2.6.26. Ger., 3.6.25).—An apparatus for the drying of, e.g., moist coal dust comprises a revolving ring drying oven into which the material is charged in a thin layer and through which is drawn by suction air preheated by the hot, moist, waste gases from the dryer. The dust-laden air from the dryer passes through settling devices in which partial classification of the material is effected prior to passing into the preheater.
A. R. POWELL.

Means for simultaneous drying and grinding. E. BARTHELMUSS (U.S.P. 1,702,333, 19.2.29. Appl., 15.5.26. Ger., 15.5.25).—Coal or similar material is subjected, in a cycle of operations, to the action of a hot drying fluid and sifted; the undersize constitutes finished material, and the oversize is ground, mixed with fresh material, and re-subjected to drying as above.
B. M. VENABLES.

Pulverising apparatus. W. R. WOOD (B.P. 292,175, 16.6.28. U.S., 17.6.27).—In a system of air-borne pulverisation working in substantially closed circuit, if the air is also used as a drying medium it is necessary to allow some of the air to go to waste. In this invention there is inserted in the return air main, between the cyclone collector and the pulveriser, a device by which the residual very fine dust is temporarily removed from the air stream, and, after removal of the necessary amount of clean waste air, the dust is returned to the main stream. One device described comprises a drum with tangential inlet at one end, tangential outlet for

the bulk of return air and dust at the other end, and an outlet for clean waste air axially.

B. M. VENABLES.

Pulverising mill. J. CRITES, Assr. to RAYMOND BROS. IMPACT PULVERIZER CO. (U.S.P. 1,702,248, 19.2.29. Appl., 31.12.26).—In the passage between the pulverising chamber and the air classifier above are a number of transverse baffles, of which the under surfaces are concave and which are in staggered rows; the baffles are intended to prevent large particles being flung by the pulveriser into the classifier.

B. M. VENABLES.

Pulverising mill. J. MEAD, JUN. (U.S.P. 1,704,757, 12.3.29. Appl., 29.9.26).—The "screens" of an impact pulveriser are supported in annular grooves in opposite ends of the casing. The "screens" comprise a complete circle of arcuate elements, all of which have pyramidal interior surfaces, and one or more have perforations through the points of the pyramids.

B. M. VENABLES.

[Impact] pulverisers. T. BROADBENT & SONS, LTD., and W. HALLITT (B.P. 308,095, 4.4.28).—The apparatus comprises two horizontal discs rotating co-axially in opposite directions at centrifugal speed. The material is fed through the centre of the upper disc to the space between the discs, and leaves at the periphery. The working faces of the discs are provided with intercalating annular teeth of buttress form, the steep faces being towards the axis. Fillets of the material under treatment are caught in the angles between the steep sides of the teeth and the main part of the discs, so that the impact of the material flung off from the preceding tooth is always upon a mass of the material itself, thus avoiding abrasion of the metal.

B. M. VENABLES.

Crushing machine. J. J. DENNY (U.S.P. 1,704,823, 12.3.29. Appl., 1.10.26).—A number of crusher plates with flat backs are linked together to form two rounds of chain-conveyor or crusher belts. The conveyors are supported at their ends by rollers (four) with sprocket wheels (eight) at the ends of the rollers. Two runs of the conveyors approach each other, forming the crushing nip, the crusher plates being supported by the above-mentioned rollers and by others with unyielding axes under and above, respectively, the straight approaching runs of the crushing belts.

B. M. VENABLES.

Crushing machines having vibrating jaws. F. M. VALE (B.P. 306,609, 28.11.27).—In a jaw-crusher preferably of the granulator type the corrugations on the jaws are longitudinal, of comparatively fine pitch, but formed in more than one plane; the same effect may be produced by superposing fine corrugations upon much coarser ones. Transversely, each jaw exactly mates with the other, but longitudinally one jaw may be convex to the other.

B. M. VENABLES.

Grinding or crushing mill. C. J. COOPER and A. M. MASON (C. J. COOPER & Co.) (B.P. 306,630, 3.12.27).—The mill, which is intended for paints and the like, is similar in construction to a multi-plate friction clutch.

B. M. VENABLES.

Crushing and grinding machine. A. C. HAMEY

and J. STONEHAM (U.S.P. 1,706,290, 19.3.29. Appl., 10.2.27. Austral., 19.2.26).—The material is fed through a hollow vertical shaft which rotates a muller above another fixed muller. The shaft and muller are rotated by a power-driven sleeve, and are also given a vertical reciprocating movement.

B. M. VENABLES.

Colloid-treating apparatus. H. W. A. DIXON (U.S.P. 1,702,380, 19.2.29. Appl., 23.3.27).—A colloid mill is contained in a casing, and the material is supplied under positive pressure and withdrawn under negative pressure by means of a pair of gear-wheel pumps operated by a common motor. Two containers are used, filled and emptied alternately by the same material until it is sufficiently dispersed, but the material always flows through the mill in the same direction, the change-over of flows being effected by a rotary valve. Spaces are provided in the body of the valve and the mill for a heating (or cooling) fluid which flows through pipes adjacent to those for material, the pairs being surrounded by insulation so that the material is always subjected to the heating (or cooling) influence.

B. M. VENABLES.

[Rotary-drum] comminuting mills. ALLIS-CHALMERS MANUF. CO., Assees. of [A] E. C. GREISEN, [B] R. C. NEWHOUSE (U.S.P. 1,710,659 and 1,710,666, 23.4.29. Appl., [A] 12.3.28, [B] 8.3.28).—(A) The mill is provided with scoops for feeding the material through the circumference of the drum; detachment of the scoops leaves openings for direct removal of material from the drum. (B) The mill has a grinding compartment surrounded by a cylindrical screen attached to it. The oversize travels longitudinally within the screen to a stationary pocket, whence it is picked up by scoops attached to the mill, and returned within the screen and back into the mill through the same openings, now at the top, through which it had emerged at the bottom. The undersize after passing through the screen is conveyed longitudinally in the reverse direction by worms attached to the outside of the screen, and is picked up by other scoops and delivered to another compartment of the mill for finer grinding, or disposed of as finished product.

B. M. VENABLES.

Homogenising mill. W. EPPENBACH (B.P. 306,502, 22.11.27).—The apparatus comprises two relatively rotatable "grinding" elements within a casing. The inlet for material is to the outside of the grinding elements, and outlet for emulsion from the space between the grinding elements through the hollow shaft of one of them. The casing may be jacketed to control the temperature and may be split in the same plane as the operating surface between the two rotating elements.

B. M. VENABLES.

Mixer. R. L. COOK (U.S.P. 1,702,931, 19.2.29. Appl., 17.3.28).—A rotating drum is provided with internal guides to drive material, received at one end of the drum, in one longitudinal direction when the rotation is in one direction and back again when the rotation is changed. One end of the mixer is provided with a double series of scoops—one series engaging and charging the material into the mixer and effective when rotated the first way, the other being effective for discharge on reversal.

B. M. VENABLES.

Mixing apparatus. P. LENART (U.S.P. 1,706,176, 19.3.29. Appl., 17.12.27. Ger., 28.12.26).—Two containers are arranged one within the other, the inner one having openings in the bottom and sides. The material is drawn through the bottom of the inner vessel by a pump below it, but within the outer vessel, and delivered up the annular space and into the inner vessel again.

B. M. VENABLES.

Producing an intimate mixture of several media by means of centrifugal force. P. JANES (B.P. 294,871 and 305,877, [A] 21. and [B] 23.4.28. Addns. to B.P. 283,975; B., 1929, 191).—(A) In the apparatus described in the original patent the mixture is cooled by partial evaporation by forming the deflecting or collecting shield of porous material. The shield is preferably stationary and inclined to the issuing mixture. (B) Mechanical additions and improvements are made to the original apparatus.

B. M. VENABLES.

Apparatus for mixing or emulsifying especially viscous or adhering liquids. O. J. BUDTZ (B.P. 307,283, 24.10.28).—A vessel with hemispherical bottom is provided with a number of concentric hoops alternately fixed and rotating. The fixed hoops are attached to a fixed bush at one side of the vessel, and the moving ones to a bush at the other side of the vessel rotated by a shaft which extends across the vessel and turns freely in the fixed bush.

B. M. VENABLES.

Machines for kneading or mixing plastic masses, liquids, or powdery material. R. RIEDL (B.P. 302,152, 14.11.28. Austr., 10.12.27).—A disc rotates in the bottom of a fixed bowl or container, and by means of blades on its upper surface presses the material against the wall of the bowl. Air may be drawn or blown into the material from an opening in the casing below the disc.

B. M. VENABLES.

Screening of materials. L. S. DEITZ, JUN. (U.S.P. 1,710,208, 23.4.29. Appl., 29.9.26).—A stream of the materials is caused to flow perpendicularly through a screen, and the screen is moved transversely to the stream of materials, but the latter is substantially prevented from following the motion of the screen.

B. M. VENABLES.

Treatment of solids with liquids. K. KOMERS (B.P. 288,999, 1.11.27. Czechoslov., 19.4.27).—Comminuted solid material is subjected to countercurrent leaching in an apparatus comprising a trough-like container of large diameter compared with its length. The solids are dragged round the circumference of the trough by comb-like arms attached to a drum rotating about the axis of the trough. The solids are delivered over the side of the large trough by fixed comb-like teeth alternating with the moving teeth into a smaller and longer trough provided with a permeable bottom and spiral conveyor blades, from which horizontal conveyor the material is elevated by a vertical presser worm and delivered in a nearly dry state to a final conveyor for removal to any point desired. Any leaching fluid that drains through the permeable bottom, plus a supply of fresh fluid, is forced by a pump round the trough of large diameter in the opposite direction to the solids, and overflows at a point near the entry for solid.

B. M. VENABLES.

Apparatus for treating semi-solids and liquids. E. E. LINDSEY (U.S.P. 1,705,822, 19.3.29. Appl., 14.11.27).—The material is circulated by means of a centrifugal pump through a conical tank, in which it meets a stream of steam or hot air, as desired, to cause the material to gelatinise or the excess moisture to evaporate. The pump withdraws the liquid from the bottom, and returns it at the top of the tank through a spray device, which causes it to run down the walls as a thin film.

A. R. POWELL.

Separation of solids from liquids. A. L. BLUMFIELD, L. S. HARNER, and H. S. COE, ASSTS. to CYCLE CO. (U.S.P. 1,702,192—3, 12.2.29. Appl., [A] 12.5.24, [B] 2.3.26).—A pulp is thickened and clarified by admitting it above a column of filtering material, part of the clear liquid being overflowed some distance above the level of entry, and part simultaneously withdrawn as filtrate downwards through the filtering column. Settled thick pulp with thin successive layers of the filtering medium are scraped off and removed through the centre of the bottom of the tank.

B. M. VENABLES.

Centrifugal separators. AKTIEBOLAGET SEPARATOR (B.P. 298,949, 16.10.28. Swed., 17.10.27).—In a separator for two liquids means are described for keeping the outlet for heavy liquid comparatively close to the axis without any risk of contaminating the lighter liquid.

B. M. VENABLES.

Centrifugal separators. E. B. MÖLBACH (B.P. 299,703, 19.10.28. Norw., 29.10.27).—In a separator for two liquids the main discs are stacked alternately with discs of smaller diameter. The smaller discs are provided with ports so that the feed passes from a common central passage above each of the smaller discs, and the lighter fluid passes under the smaller discs to a number of outlet passages parallel to, and not far removed from, the axis. The heavier fraction is passed out into a volute pump-casing, in which the loss of kinetic energy produces a certain back-pressure, which in turn helps to force out the lighter fraction. Supplementary to this, another pump, complete with rotor, may be provided for the light fraction.

B. M. VENABLES.

Centrifugal separators and coolers. B. C. CARTER (B.P. 310,550, 28.10.27).—The separator part comprises a bowl to which the mixed fluids are supplied under pressure through a hollow pivot, and which is rotated only by the action of a separated fluid issuing through tangential jets. In separating oil-water emulsions, the lighter fluid (oil) will issue through the jets and be cooled by impingement on a stationary outer shell provided with fins or otherwise kept cool.

B. M. VENABLES.

Centrifuge. NAT. ACME CO., ASSEES. of D. S. PATERSON and G. B. PETSCH (B.P. 294,525, 9.5.28. U.S., 25.7.27).—In a centrifuge of the bowl type the bowl is supported on a door of the casing and swung out for cleaning. Other mechanical improvements are also described.

B. M. VENABLES.

Liquid separator. H. S. COE, ASSR. to DORR CO. (U.S.P. 1,709,971, 23.4.29. Appl., 20.5.25).—For the continuous separation of liquids of different sp. gr. by settling, a number of (conical) diaphragms are

assembled in a vertical (cylindrical) casing. The feed of mixed liquids is through a common supply pipe with branches leading to the spaces between each diaphragm, at the circumference; the outlets for heavy liquid are similarly situated, but well spaced (say 180°) circumferentially from the inlets. The outlet for lighter liquids is axially upwards through the points of the cones.

B. M. VENABLES.

Hydraulic separator. H. H. CARL and H. E. MUEHLHOF (U.S.P. 1,710,568, 23.4.29. Appl., 26.11.27).—The separator comprises a tank having a feed at one end and discharges at the other end for heavy liquid at the bottom and for light at the top; the latter discharge is subdivided by an adjustable skimmer, the uppermost layer of liquid removed being sent to a pump which delivers it back to the tank in such a manner as to cause upward and forward currents.

B. M. VENABLES.

Conservation of centrifuged liquids. H. O. LINDGREN, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,705,934, 19.3.29. Appl., 27.7.26. Swed., 29.7.25).—The separated liquids, which are likely to be delivered in finely-divided form, are collected in vessels under slight vacuum and provided with means to separate finely-divided liquid from any gas in which it is entrained.

B. M. VENABLES.

Means for separating air, vapour, and volatile liquids from liquids. E. DODSON (B.P. 308,010, 11.1.28).—The liquid, *e.g.*, lubricating oil containing water and/or petrol, is passed through a Venturi tube, the drop in pressure in the throat of which causes the volatile fluids to form bubbles. After the stream of liquid has expanded to the normal bore of the conduit, but before the bubbles have had time to redissolve, the latter are allowed to rise out of the stream into a vent chamber. The action is much increased by permitting air to be drawn into the throat of the Venturi through a side passage.

B. M. VENABLES.

Apparatus for pasteurising liquids. A. JENSEN (U.S.P. 1,701,777, 12.2.29. Appl., 21.6.26. Cf. U.S.P. 1,693,034; B., 1929, 192).—A chamber is heated by a fluid under pressure in a jacket. The inlet for the liquid to be treated is centrally at the bottom and the outlet peripherally at the top; a rotor is provided to force the liquid centrifugally through the vessel. The shaft of the rotor is hollow and perforated within the upper part of the chamber, and is utilised to afford connexion to an exhaust pump.

B. M. VENABLES.

Conveyance of viscous substances. C. P. BRASINGTON, Assr. to INTERNAT. PRINTING INK CORP. (U.S.P. 1,710,144, 23.4.29. Appl., 23.3.28).—A moving endless wire dips into the fluid at the starting point and adhering fluid is removed therefrom at the delivery point.

B. M. VENABLES.

Tubular rotary crystallisers or crystalliser-boilers. F. LAFEUILLE (B.P. 301,453, 4.10.28. Fr., 30.11.27).—The tubes for heating or cooling agent are not expanded into tube plates, but are freely supported only by stay-plates, the junctions with the return bends and to the supply and discharge headers being all welded. The massecuite or other fluid under treatment occupies the whole space within the cylinder right up to the dished

ends, which are pressure-resisting, and one of which is provided with concentric inlet and outlet for heating or cooling fluid, the other with concentric inlet being for fluid to be crystallised and for outlet of vapour. In operation the cylinder and heating or cooling tubes rotate and with them a worm in the inlet pipe for massecuite; the actual inlet and outlet pipes remain stationary.

B. M. VENABLES.

Separating out the mother-liquor from crystals or crystalline bodies. RAFFINERIE TIRLEMONTOISE Soc. ANON. (B.P. 286,599, 24.2.28. Ger., 5.3.27).—In a centrifugal process for draining crystals, the basket is first charged, closed by an air-tight cover, and then run up to speed. This prevents circulation of air which would have an evaporating effect on the mother-liquor adhering to the crystals, and cause surface-discoloration in the case of materials like sugar with syrup.

B. M. VENABLES.

Precipitation apparatus. R. W. SHAFOR, A. R. NEES, and R. J. BROWN (U.S.P. 1,708,332, 9.4.29. Appl., 19.7.24).—A vessel provided with an outlet to maintain a constant liquid level inside it is divided into mixing and precipitation zones communicating with one another in the lower part of the vessel. The material under treatment is fed into the mixing zone in which means are provided to produce a current flowing downwards and into the precipitation zone, the lower part of which contains means for bringing the contents to a temperature suitable for precipitation.

L. A. COLES.

Combined decanting and filtering apparatus. C. PICCARDO (B.P. 305,976, 22.8.28. Ger., 13.2.28).—A sleeve of fabric is suspended within a container and spaced from the wall thereof by means of a permeable material such as spiral wire mesh, and from the annular space a draw-off for clear liquid is provided. The top of the sleeve may be sealed to an angle-iron ring within the container, and the bottom may slip into an internal channel and be sealed with sand. The mud is discharged through a hopper bottom.

B. M. VENABLES.

Filtering or like devices. H. A. THOMPSON (B.P. 307,267 and 308,166, [A] 31.7.28, [B] 26.10.28).—(A) The filter comprises a pack of annular laminations interleaved with scraping laminae capable of rotation or rocking by means of suitable shafts on which they are threaded. In (B) the same shafts that rotate the scrapers also carry pinions gearing with teeth formed on the edge of the main laminations, so that the latter as well as the scrapers rotate.

B. M. VENABLES.

Distilling and like apparatus. H. GRIFFITHS (B.P. 309,976, 19.11.28).—Condensate from a still is collected in a high-vacuum receiver which is connected to a low-vacuum receiver, some distance below it so as to create a hydrostatic head of condensate, by a pipe having a valve which is closed when it is desired to discharge the condensate from the low-vacuum receiver. The receivers may be connected to the respective stages of a single, two-stage, vacuum pump, and the low-vacuum receiver is cut off from the vacuum pump when air is admitted to discharge condensate; these operations may be effected automatically by floats in the low-vacuum receiver.

B. M. VENABLES.

Still. E. H. RECORDS (U.S.P. 1,710,070, 23.4.29. Appl., 24.2.25).—A removable charging bucket having a perforated bottom, perforated side walls, and a central pipe open at both ends and leading to the outside of the bucket, supported therein, forms an annular steam passage within a still-cylinder having a steam inlet and an outlet for volatile gases. J. S. G. THOMAS.

Apparatus for separating grit, dust, etc. from smoke and gases. A. PARKER (B.P. 307,168, 27.1.28).—The gases are admitted upwardly through an annular space between an inverted, conical, outer casing and a similarly shaped collecting surface. On reaching the top (base) of the cone the gases are caused to whirl inwards and downwards by means of fixed vanes; the solid matter separates out on the interior of the catcher cone, and the cleaned gases have their motion changed from whirling downwards to rectilinear upward motion along the axis of the cones by means of another set of fixed vanes, and are discharged through the top of the apparatus. B. M. VENABLES.

Removal or separation from gaseous fluid of material suspended therein. R. S. PORTHAM, and TANGENTIAL DRYERS, LTD. (B.P. 306,697, 15.2.28. Addn. to B.P. 271,545; B., 1927, 544).—The many-pointed body on which the gas impinges with change of direction is so arranged that the dust can pass away between the points in the original direction of the gas. The gas may afterwards pass through a filter bed. B. M. VENABLES.

Rotatable air or gas filter of the plate type. A. SCHIRP (B.P. 303,740, 5.10.28. Ger., 7.1.28).—The filter comprises a number of closely spaced plates kept moist with a liquid such as oil and secured at one end to a chain which slowly travels round two small sprockets vertically over each other. The free ends of the plates are guided only. When the plates pass over the sprockets they open out, affording opportunity for cleansing them and renewing the liquid film by means of a vessel of liquid at the bottom. Several of these chain-and-plate elements are arranged in horizontal series, and the gas passes through them all, entering through the free ends of a set of plates which is, say, rising, passing between the plates, through both runs of the chain, between a set of plates which is falling, and so on. The driving is effected at the lower sprockets by gearing outside the washing chamber. B. M. VENABLES.

Filter for straining and clarifying such portions of the gases, from furnace flues etc., as may be desired for analysis etc. A. and L. LUMB (B.P. 305,874, 10.4.28).—The apparatus comprises a casing with closed top and water-sealed bottom containing a filter medium (e.g., coke and asbestos fibre) between two grids. B. M. VENABLES.

Filter medium for air or gas filters. A. JORDAHL (B.P. 306,348, 15.6.28).—The medium comprises a number of layers of knitted fabric made from a flat ribbon, preferably metallic. B. M. VENABLES.

Separation of mixtures of gases and gases with vapours. H. BLAU (B.P. 278,712, 4.10.27. Ger., 5.10.26).—The mixture of gases of different solubility is passed, preferably under increased pressure, up a tower where it meets a descending current of cooled

absorbing liquid, with the result that the practically pure constituent of lower solubility (A) leaves from the top of the tower. Below the absorbing tower is a rectifying tower, heated slightly at the bottom, in which the absorbing liquid is gradually heated so that all of gas A (and some of the constituent of higher solubility [B]) is expelled from solution; the evolved mixture mingles with the fresh mixture in the absorbing tower above. The liquid from the bottom of the rectifier, having only gas B dissolved in it, passes through a heat exchanger to an expelling tower, where it is more strongly heated, the vapour of pure gas B being cooled with or without condensation. The barren absorbing liquid passes through the heat interchanger and a cooler and is re-used. If there are several constituents of moderate solubility they may be further separated by the use of several expelling towers in series. The towers each have a cooled top and heated bottom, the point of entry for the liquid with dissolved constituents being intermediate. B. M. VENABLES.

Air- and gas-washing apparatus. PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (B.P. 306,058, 14.11.27).—In the apparatus described in B.P. 277,112 (B., 1927, 832), the roof is rigid but the baffles are loosely mounted. A hopper with suitable valves is also provided for collection of sludge. B. M. VENABLES.

Rectification of mixed gases. S. G. ALLEN, Assee. of W. L. DE BAUFRE (B.P. 283,101, 285,468, and 294,994, Appl., [A—C] 11.10.27. U.S., [A, C] 3.1.27, [B] 18.2.27).—These patents refer to an elaborate system of heat exchangers, bubbling columns, and tubular rectifiers, for the purpose of separating a mixed gas (e.g., air) into its pure constituents. B. M. VENABLES.

Recovery of gases and vapours from gas mixtures. V. PANTENBURG (U.S.P. 1,702,311, 19.2.29. Appl., 28.1.26. Ger., 7.2.25).—A number of absorption chambers are connected to a single regeneration chamber to which the absorption material is transferred in turn. B. M. VENABLES.

Drying of gases and vapours. HOLZVERKOHLUNGS-IND. A.-G. (F.P. 630,612, 9.3.27. Ger., 28.5.26).—The gases etc. are passed up towers containing sodium bisulphate, the aqueous solution formed of this being evaporated to dryness for re-use. L. A. COLES.

Apparatus for preparing compressed gases. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 279,041, 3.9.27. Ger., 18.10.26).—A pressure vessel, in which gases are stored in their liquid condition and from which compressed gases of different controllable pressures may be drawn off, is provided (a) with a thin-walled inner vessel, spaced from the pressure-resisting vessel, gas only being allowed to fill the space between the two, and (b) with a draw-off coil leading from below the level in the liquid and round the pressure-resisting vessel in coils which gradually get further away from that vessel and in which evaporation takes place. The walls of both vessels are made of a metal that does not change its coefficient of expansion at low temperatures. A rectifying column may be placed within the inner vessel, in which an additional gas may be liquefied

(*e.g.*, oxygen-rich air) partially to take the place of that drawn off (*e.g.*, oxygen). The rectifier is used in conjunction with a double-walled inlet and outlet tube, which serves as a heat exchanger. B. M. VENABLES.

Liquefying and supplying gases. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 282,813 and 307,083, 29.12.27. Ger., [A] 29.12.26, [B] 14.1.27).—(A) In transferring liquid air or other gas from an expansion machine to vessels for storage or transport, gas formed by evaporation is led back by a pipe to the expansion machine, which is operated at a higher pressure than usual (250 atm. instead of 200 in the case of air), the resulting excess cold being sufficient to re-liquefy the returned gas. (B) Since large storage vessels as described in B.P. 279,041 (p. 500) cannot produce gas at a pressure much above 40 atm., a small quantity of liquid is evaporated in a small strong vessel to give gas at a much higher pressure, say 150 atm., which is utilised to work an injector drawing medium-pressure (40 atm.) gas from one storage bottle and delivering it into other bottles at normal storage pressure, the medium-pressure bottle being thereby emptied ready to receive gas from the main liquid storage again. B. M. VENABLES.

Accumulating, conveying, and consuming liquefied gases of low b.p. without loss. GES. F. INDUSTRIEGASVERWERTUNG M.B.H., and C. W. P. HEYLANDT (B.P. 280,588, 12.11.27).—Means are described for reducing time and opportunities for evaporation when transferring liquefied gas from a transport vessel to measuring vessels, and thence to a storage vessel. The inner transport vessel is suspended on chains which are provided either with insulating links or with metallic links having line-contact only, and any gas (oxygen) which does escape may be used in the motor of the vehicle. B. M. VENABLES.

Re-gasification of liquefied gases. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 287,909, 28.3.28. Ger., 29.3.27).—The re-gasification is accelerated by admitting a reserve supply of compressed gas to the gasifier, the pressure, number of molecules, and the rate of heat transmission from the exterior of the gasifier to the liquid being thereby increased, thus quickening (but not dangerously so) the evolution of gas from the liquid. Suitable valves are described. B. M. VENABLES.

Continuous absorption refrigerating apparatus. R. F. BOSSINI and G. MAIURI (B.P. 307,236, 11.4.28).—The inert gas is of approximately the same density as the vaporised refrigerant, and is either a single gas or a mixture of gases. When ammonia is the refrigerant, the inert gas may be methane or a mixture of nitrogen and hydrogen. B. M. VENABLES.

Antifreeze composition. J. R. PAUL (U.S.P. 1,689,153, 23.10.28. Appl., 17.2.27).—Sodium thio-sulphate and calcium chloride in approximately equal amounts and a relatively small amount of tartaric acid are added to water. R. BRIGHTMAN.

F.p. depressant. S. ISERMANN and W. VERNET (U.S.P. 1,687,094, 9.10.28. Appl., 9.7.25).—Formamide, acetamide, diacetone alcohol, acetylacetone, diacetoneglycerol, or mixtures of these, are used with or without glycerol to depress the f.p. of water etc. R. BRIGHTMAN.

Disintegrating or mixing apparatus. C. W. BOISE and W. R. DEGENHARDT (U.S.P. 1,711,259, 30.4.29. Appl., 2.8.27. U.K., 10.8.26).—See B.P. 280,276; B., 1928, 72.

Separating and purifying apparatus for liquids. J. SCHÄFER (U.S.P. 1,711,428, 30.4.29. Appl., 31.5.27. Fr., 15.6.26).—See B.P. 272,927; B., 1928, 216.

[Means for excluding air to interior of] rotary furnaces and kilns. METALS PRODUCTION, LTD. From T. J. TAPLIN (B.P. 310,780, 30.1.28).

Heating by air and apparatus therefor. F. LEBRE (B.P. 282,371, 1.12.27. Fr., 17.12.26).

Refrigerating apparatus of the absorption type. N. V. KODOWA REFRIGERATOR COMP., and W. A. SLATER (B.P. 310,901, 2.1.28).

Absorption refrigerating apparatus. H. D. FITZPATRICK. From N. V. KODOWA REFRIGERATOR Co. (B.P. 296,792 and 311,496, [A] 16.2.28, [B] 9.3.28).

Refrigerating apparatus. J. HUDSON (B.P. 311,595, 11.7.28).

[Automatic] means for controlling the supply of materials to pulveriser and like mills. STIRLING BOILER Co., LTD., and E. E. NOBLE (B.P. 311,606, 30.7.28).

[Shaking table for] separating or cleaning inter-mixed divided materials. K. DAVIS (B.P. 307,978, 13.10.27).

[Bag] filters. A.C. SPARK PLUG Co. (B.P. 284,982, 6.2.28. U.S., 5.2.27).

Treating material with gases (B.P. 305,883).—See X. **Sorting articles by light** (B.P. 292,474).—See XI. **Distillation apparatus** (B.P. 310,400 and 310,403).—See XVIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Mechanism of the carbonisation of coal. E. AUDIBERT (Fuel, 1929, 8, 225—243; cf. B., 1927, 383).—The factors controlling the physical phenomena accompanying the carbonisation of coal and determining the strength of the coke formed are divided into two classes: (a) the initial characteristics of the network of interspaces, *i.e.*, the particle size and the density of packing, and (b) the factors determining the maximum fluidity of the coal in the plastic state, *i.e.*, the nature of the coal, its degree of oxidation, and the rate of heating. The strength of a small cylinder of coke prepared under standard carbonising conditions increases rapidly as the particle size of the coal used decreases below about $\frac{1}{2}$ mm. diam.; above that size the strength is not greatly affected by this factor. The strengths of the cokes produced from different mixtures of coals of three different particle sizes are conveniently represented on a trilinear diagram. The mechanism of the agglomeration of the coal particles in a coke oven is discussed; neither pyrolysis of the vapours passing through the coke nor the deposition of pitch on the coal in the region within the plastic layer plays any part in this agglomeration. The maximum fluidity of a coal is measured by the "agglutination index," *i.e.*, the amount of an infusible substance that must be added

to 100 pts. of the coal in order that, at a rate of heating of $1^{\circ}/\text{min.}$, the mixture may be just at the limit of intumescence. This index is independent of the fineness of the particles used, the apparent density of the mass, and also, very nearly, of the nature of the infusible substance. As the rate of heating is increased the agglutination index increases until it reaches a limiting value. Consideration of the factors responsible for the fissuring of coke has led to an empirical test for distinguishing between coals giving a lump coke and those giving a "fingery" coke. The finely-divided coal is carbonised within a cast-steel cylinder under carefully-controlled conditions which give results, with respect of the type of coke, comparable with large-scale practice. A "fingery" coke can be converted into a lump coke by adding to the coal a suitable proportion of coke, semi-coke, or a very slightly fusible coal. To make a dense coke the diluent substance should contain 10–15% of volatile matter. The practical application of these principles to determine the most suitable ternary mixtures for carbonisation in the oven is described in detail. The binary mixtures of zero agglutination index are first found; the line joining the points corresponding to these on the triangular diagram divides the latter into regions of positive and negative agglutination index respectively. It is then easy to determine by a few trials the curve in the former region which separates the zones of "fingery" coke from that of lump coke. A. B. MANNING.

Rectangular graphs as applied to the proximate analyses of Chinese coals. H. S. WANG (Bull. Geol. Soc. China, 1928, 7, 175; Fuel, 1929, 8, 244–248).—The smoothness of the curves obtained by plotting moisture and volatile matter against fixed carbon (on the ash-free basis) for 40 Chinese coals indicates that there is no break in the series from low-rank bituminous coal to anthracite in Palæozoic, Mesozoic, and Tertiary coals. The importance of geological age in relation to coal classification is emphasised. A. B. MANNING.

Heat transfer in recuperators. E. TERRES and W. BESECKE (Gas- u. Wasserfach, 1929, 72, 417–421, 447–449, 466–469, 476–477).—Experiments have been carried out on a vertical-chamber oven, heated by an internal producer, with particular reference to the efficiency of the recuperator system. Temperatures of flue gases and air were measured by rare-metal thermocouples protected by silica and by iron sheaths, and the volumes of the gases were obtained by calculation from hourly analyses, and from the coke consumption; the coke used in 24 hrs. was weighed, sampled, and analysed. Producer gas was sampled through a special pipe fitted in one of the cleaning ports, and flue gases were withdrawn from four different points in the setting; slow withdrawal of the gases was found to give unreliable results owing to change in composition, and intermittent samples were taken more rapidly. The volume of producer gas was calculated by assuming 97% of carbon in the dry, ash-free coke; the calorific value of the latter was 7950 kg.-cal./kg. The carbon content of the flue gases at different points was compared with that of the producer gas, so that the amount of secondary air drawn in through leaks could be determined. The

methods employed in interpretation of the results are explained in full, and calculations are included for gas volumes, mean temperature differences, heat balances, sp. heats, efficiency of recuperation, and coefficient of heat conductivity. The 18 tests recorded in detail gave the following mean results: coke used in 24 hrs., 2822 kg.; volume of producer gas, flue gas, and air, 470, 937, and 539 m.³/hr., respectively. The flue gases gave up 93,100 kg.-cal./hr., and the efficiency of recuperation was 33.8%; the conductivity through the bricks of the recuperator was 7.93 kg.-cal./m.²/hr. $^{\circ}\text{C.}$ for a mean temperature of 500° . The air drawn in through leaks varied widely in volume, and accounted for irregular results in the heat balance, so that the heat absorbed by the incoming air sometimes appeared to be more than that lost by the waste gases. Further calculations are included, showing the relations between gas velocity, temperatures, and the rate of heat transfer, and it is found that the coefficients of conductivity for the bricks are considerably larger than those derived from existing information on the subject. Curves are included which show the change of rate of heat transfer with temperature and with gas velocities between 0.87 and 1.22 m./sec., and the causes of the low general efficiency of recuperation are discussed and compared with those concerned in regenerative systems.

R. H. GRIFFITH.

Distillation of peat. V. E. RAKOVSKI (J. Chem. Ind. Moscow, 1928, 5, 915–919).—The rôle of water vapour injected into the retort during the distillation of peat, whereby the tar contains a smaller quantity of asphaltenes, is considered to be the impeding of condensation processes normally occurring with formation of water.

CHEMICAL ABSTRACTS.

Determination of the elementary oxidisable carbon in solid fossil fuels. J. BLUM (Bul. Chim. pura aplic. Bukarest, 1927, 30, 43–49; Chem. Zentr., 1928, ii, 2314).—From the carbon dioxide produced on combustion must be subtracted that absorbed in the coal, that arising from mineral substances, and that produced by decomposition of organic compounds.

A. A. ELDRIDGE.

Modern illuminating gas production consequent on the Krummhübl calorific value agreement. R. GEIPERT (Gas- u. Wasserfach, 1929, 72, 169–174, 205–210).—The Krummhübl agreement advocates the mixing of coal gas with water-gas to give a mixture of calorific value 4200 kg.-cal. This practice results in a saving of gas coal, a reduction in the number of retorts needed, and a greater flexibility in dealing with varying loads. Flue gases may be mixed with coal gas, instead of water-gas, but to less advantage. Working results are compared in the case of two plants with vertical retorts, the carbonising times being 12 and 16 hrs. respectively. In the former less water-gas is obtained and the deficiency has to be drawn from producers. The retorts carbonising in 16 hrs. are decidedly more efficient thermally, and avoid the necessity of transporting and grading the coke for the producers. The ash content of the lump coke is not increased by steaming, the superficial mineral matter rubbing off as dust; steaming in this retort does not appear to produce a greater proportion of coke fines. The quantity of

ammonia liquor is increased, but its strength is not diminished, owing to the greater yield of ammonia from vertical retorts. A portion of the hot flue gases may be by-passed to a steam boiler, the remainder passing through the recuperators. Fixed and revolving grates for water-gas producers are discussed, together with slag prevention by correct choice of coke, and prevention of explosions. Reducing the depth of the fuel bed from 1.9 m. to 1.2 m. had little effect on the working of the producer. The water-gas should be added in measured proportion to the coal gas after the latter has passed the tar extractor and scrubbers. The water-gas or mixed gas may be carburetted with crude benzol instead of with gas oil, although the high naphthalene content of the former might be a drawback. The working results of four gasworks prove the great flexibility of vertical-retort practice.

W. T. K. BRAUNHOLTZ.

Production of blue and carburetted water-gas in continuous vertical retorts. T. F. E. RHEAD (Gas J., Special No., Feb. 14, 1928, 59—65).—Experiments are recorded on (1) the production of blue water-gas by passing steam through graded coke in a continuous vertical retort, and (2) the cracking of light tar and gas oil under similar conditions. Effects of steam, rate of coke extraction, coke quality and size, temperature, exhaustor "pull," tightness of retort, etc. were investigated. An average hourly make of 4600 cub. ft. (290 B.Th.U. gross) was obtained from 190 lb. of steam and a coke extraction of 126 lb./hr. Thermal efficiency tests showed that 58% of the total heat in coke "consumed" in the retorts plus gross coke to producers appeared as potential heat in gas, and that waste gases could generate more than sufficient steam to be self-supporting. Scuffing of retorts was shown to be possible. The extent to which the steamed coke became deteriorated was demonstrated. Gas oil was cracked in the retort and gave gases of 375—447 B.Th.U. when using 0.97—1.86 gals. per 1000 cub. ft. of carburetted water-gas. Extensive "crude tar" cracking tests proved disappointing and impracticable. Either the seal pots, offtakes, etc. became blocked with pitch, or the retort itself with pitch-coke. Gases of 361—387 B.Th.U. were obtained using 1.73—3 gals. per 1000 cub. ft. Several suggestions of the value of the above water-gas process for emergencies are outlined.

Grozni oil gases. P. I. BOGARJEVSKI (Neff. Choz., 1928, 15, 636—638).—The amount of gasoline obtained from natural gas was 9.42 gals. (d 0.700), 5 gals. (d 0.640), and 1.69—3.93 gals. (d 0.635—0.658) per 1000 cub. ft. from three sources, respectively.

CHEMICAL ABSTRACTS.

[Production of] high-value anti-knock fuels by cracking low-temperature coal tar. G. EGLOFF (Petroleum, 1929, 25, 573—577).—Tars from the low-temperature carbonisation of West Virginian, Ohio-Indiana, and Utah coals are cracked at 452° and 7 atm., 427° and 7 atm., and 435° and 7.7 atm., respectively, with the production of (1) 22—24% of acid-free benzine of more than 50% benzene equivalent, (2) fuel or Diesel oil, (3) tar acids of low b.p. which are suitable for making phenol condensation

products or as insecticides or wood preservatives, (4) cracked gases of calorific value 1300 B.Th.U. per 1000 cub. ft., and (5) coke of low ash content and calorific value 16,000 B.Th.U. per lb. Neutral oil distillate (freed from tar acids and pitch) from the low-temperature carbonisation of bituminous West Virginian coal, when cracked at 454° and 17 atm., gives more than 50% of petrol of more than 50% benzene equivalent and, per brl. of oil, 85 lb. of practically ash-free coke of calorific value 16,000 B.Th.U. per lb., and 840 cub. ft. of gas of calorific value nearly 1300 B.Th.U. per 1000 cub. ft.

W. S. NORRIS.

Theory of cracking petroleum. H. A. WILSON (Proc. Roy. Soc., 1929, A, 124, 16—45).—The cracking of hypothetical oils consisting only of paraffins and unsaturated hydrocarbons is considered. Assuming that the oil has been in the reaction chamber long enough for equilibrium to have been established between all the substances present, and that no coke is deposited in the reaction chamber, and applying the results obtained in previous investigations of the chemical equilibrium in mixtures of paraffins and unsaturated hydrocarbons (cf. A., 1927, 1139; 1928, 1190), the fractions of the oil coming out of the reaction chamber as liquid, vapour, gas, gasoline, and unsaturated hydrocarbons are calculated. The values of these fractions (in weight percentages) for four hypothetical oils having the assumed compositions $\text{CH}_{2.1}$, $\text{CH}_{2.15}$, $\text{CH}_{2.2}$, and $\text{CH}_{2.25}$ (corresponding, roughly, to fuel oil, crude oil, gas oil, and kerosene), at temperatures ranging from 400° to 760° and pressures from 7.5 to 60 atm. are tabulated. It is found that there is good general agreement between the calculated results for the hypothetical oils and the results obtained with actual oils, and it is concluded that the hypothetical oils resemble actual oils sufficiently closely for the theory of the cracking of the former to represent the main features of actual cracking, thus confirming Berthelot's suggestion (Ann. Chim. Physique, 1866, 9, 445) that approximate equilibrium must be actually attained in actual cracking operations. As a question of practical importance in liquid-phase cracking, the amount of a given oil which can be cracked in unit time at any temperature and pressure with a reaction chamber of given volume is considered at some length. In practice, the time of passing through the reaction chamber is usually considerably longer than necessary. It is found that the production of gasoline increases with the amount of oil pumped through, at first rapidly and then more and more slowly. When the amount pumped through is small, the gasoline is nearly proportional to it. It follows as a practical rule from the theoretical results that the amount of oil which should be pumped into the reaction chamber per day is inversely proportional to the gasoline fraction. At a given temperature and pressure the gasoline production for a given reaction chamber should be about the same for any kind of oil, provided that the gasoline fraction is not less than about 10%. In any cracking process the theoretically possible maximum yield of gasoline would be obtained if the oil were converted into gasoline and carbon only, and the possibilities of obtaining such a yield by "recycling" all the products formed except the gasoline are indicated.

Further, a considerable increase in the percentage yield of gasoline should be theoretically obtainable by adding to the oil a small quantity of a mixture of hydrocarbons rich in hydrogen, but no experimental results are yet available for comparison with the theoretical calculations on the effect of adding gas to the oil. The factors affecting the quality of the gasoline produced are discussed, and the theoretical compositions of the gasoline obtained from the oil $\text{CH}_{2.25}$ are calculated for various temperatures and pressures. L. L. BIRCUMSHAW.

Insulating oils. G. STADNIKOV and Z. VOSSCHINSKAJA (Petroleum, 1929, 25, 651—659).—The presence of a sulphonic acid or a salt thereof in a transformer oil may cause the results of an oxidation test to be fallacious. The action of oxygen gives rise to acids, alcohols, and carbonyl compounds. In the presence of a sulphonic acid and of resinous materials, *e.g.*, in an insufficiently refined oil, the products of oxidation condense with the resin or gum (this reaction being accelerated by a sulphonic acid or its salts) and the condensation products remain in suspension, the excess of resin acting as a protective colloid. The acid value of the oil will remain somewhat low, and the oil, according to the oxidation-acid value test, may appear satisfactory although, if it is used for some time in a transformer, sludge will be formed and the acid value will rise. If, however, the oil contains a sulphonic acid, but has been freed from resin by energetic refining, the products of oxidation will be condensed under the action of the sulphonic acid and the products of condensation, in the absence of gum, will be precipitated. Thus a well-refined oil will appear less stable according to the oxidation test than one that contains an appreciable quantity of resin. These conclusions are based on experiments in which fatty acids are heated at 100° in gas-oil solution, with and without the addition of a sulphonic acid, and in the presence of alcohols, aldehydes, or ketones; besides synthetic acids, volatile acids obtained by the oxidation of a transformer oil at 160° are employed. Since sulphonic acids or salts thereof are very difficult to wash out of a transformer oil, their presence in acid-refined oils may account for discrepancies in the results of previous workers. W. S. NORRIS.

Treating heavy oils by acid sludge. N. GRAMENETZKI (Nef. Choz., 1928, 15, 212—216).—Heavy oils are treated with acid sludge at 70 – 80° without preliminary dilution or subsequent distillation.

CHEMICAL ABSTRACTS.

Paraffin wax in Apsheron crude oils. A. N. SACHANOV and L. G. ZHERDEVA (Nef. Choz., 1928, 15, 639—642).—For the determination of paraffin wax the oil (10 g.) was mixed with light gasoline (500 c.c.) and treated at the ordinary temperature with fuller's earth (60—70 g.). The fuller's earth was extracted with gasoline in a Soxhlet extractor for 2 days, the extract evaporated to 10—20 c.c., and the wax was precipitated by Holde's method with alcohol and ether, the solid being redissolved in ether and reprecipitated. The paraffin wax contained in various crude oils was determined and examined. CHEMICAL ABSTRACTS.

Ultra-violet spectroscopy of flames of motor fuels. IV. Practical utilisation of a small quartz-

prism spectrograph for the determination of lead tetraethyl in gasoline. G. L. CLARK and H. A. SMITH (J. Physical Chem., 1929, 33, 659—675. Cf. B., 1926, 522; A., 1927, 810).—A small quartz-prism spectrograph provides a simple and accurate means for determining lead tetraethyl in gasoline and its effect on a detonation flame. Details are also given of a simple type of mercury arc, and a combined still and burner for liquid fuels suitable for analyses of this kind. By the method used, 5 g. of lead in 10^6 c.c. of gasoline have been detected, and by the use of standard spectra larger quantities can be accurately determined. Seventeen lines in the flame spectrum of lead have been identified, including a hitherto undiscovered line at $\lambda = 2167 \text{ \AA}$. The band positions, structures, and intensities have been compared, under similar conditions, for flames of gasoline alone and containing small amounts of ethylene bromide, lead tetraethyl, and aniline, and of benzene, alcohol, acetone, ether, and carburetted water-gas. Further evidence has been obtained that the anti-knock action of lead tetraethyl is due not to the compound *per se* but to the process of its decomposition and to its products of disintegration. L. S. THEOBALD.

Use of the Parr calorimeter for determining the calorific value of solid fuels. H. WINKELMANN (Z. tech. Physik, 1928, 9, 422—425; Chem. Zentr., 1928, ii, 2612—2613).

PATENTS.

Retort oven. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,707,734, 2.4.29. Appl., 31.8.21. Renewed 17.8.28. Ger., 7.12.18).—A combustion chamber containing a group of retorts is divided by partitions transverse to the retorts in such a way that vertical-flame flues are formed alongside each of the retorts and over the tops thereof. Regenerators for preheating the gas and air are arranged below the retort chamber and communicate through distributing channels with corresponding ports at the bottoms of the flues. Means are provided for periodically reversing the direction of gas flow through the flues.

A. B. MANNING.

Apparatus for coking solid fuel. E. RAFFLOER (U.S.P. 1,708,152, 9.4.29. Appl., 1.4.25).—A horizontal, rotary drum has longitudinal partitions forming a number of channel-shaped chambers extending along the inner side of the wall of the drum. A rotary, tubular roller is arranged longitudinally within the drum in such a position as to cover the open inner side of the lowermost chamber. As the drum rotates, the chambers are brought in succession below the rotating roller. Means are provided for passing the fuel to be coked first through the roller and then through the chambers of the outer drum, which is externally heated. A. B. MANNING.

Destructive distillation [of solid fuels]. P. GIRARD, F. PETIT, and A. CHARBONNEAU (B.P. 290,606, 16.5.28. Fr., 16.5.27).—Solid carbonaceous fuels are distilled at low temperatures in the presence of halogens, halogen acids, or compounds, *e.g.*, bleaching powder, which yield halogens under the experimental conditions. The materials are preferably subjected at the same time to the action of a high-frequency electric discharge.

High yields of light hydrocarbon oils are thereby obtained.

A. B. MANNING.

Apparatus for distillation [of solids]. E. PIRON and V. Z. CARACRISTI, Assr. to PIRON COAL DISTILLATION SYSTEMS, INC. (U.S.P. 1,709,370—1, 16.4.29. Appl., [A] 4.4.22, [B] 14.2.23).—(A) The solid material is conveyed over and adjacent to the surface of a bath of molten material within a distillation chamber. The molten material is heated by means of flues passing through it. (B) The solid material is conveyed over the molten bath on an endless conveyor, from which it falls on to a second endless conveyor which carries it in contact with the return run of the first conveyor to the discharge opening of the chamber (cf. B.P. 207,460; B., 1924, 122).

A. B. MANNING.

Apparatus for distilling materials. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,706,421, 26.3.29. Appl., 20.1.21).—Within a cylindrical casing to which are attached a number of hollow conical, annular hearths projecting radially inwards is a revolving column from which similar hearths project outwards. The two sets of hearths fit into one another to form a confined, inclined, sinuous passageway down which the material to be distilled is passed. Hollow conical rabblers project downwards from the inclined lower surfaces of the hollow hearths almost to the upper surfaces of the next adjacent lower hearths. Hot gases are circulated in the interior of the hearths. Means are provided for charging the material into the apparatus and for discharging the solid product therefrom, as well as for collecting separately the vapours evolved from the material at progressive intervals during its passage through the retort.

A. B. MANNING.

Distillation of solid carbonaceous materials. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,706,420, 26.3.29. Appl., 11.1.23).—The material is distilled in a retort provided with a number of heating mandrels round which the fuel is packed so that on removing the carbonised mass from the retort a series of openings is formed therein.

A. B. MANNING.

Low-temperature distillation of bituminous coal. G. E. ROHMER, Assr. to NAT. COAL DISTILLATION CORP. (U.S.P. 1,708,740, 9.4.29. Appl., 23.4.27).—A horizontal rotary retort has external and internal heating members with an annular distillation chamber between them. The outer wall of the chamber is provided with lifting baffles and the inner wall with retarding baffles, the coal being alternately advanced and retarded in its passage through the distillation chamber, and subjected at the same time to different distillation temperatures from opposite sides of the chamber.

A. B. MANNING.

Low-temperature carbonisation of coal. W. M. CRANSTON (B.P. 309,231, 13.8.28).—Ground or slack coal is mixed with a mineral oil and distilled, first in a retort maintained at or below 180°, then in a second retort at a higher temperature, preferably 300–500°. The pressure in the first retort is maintained slightly below atmospheric, and that in the second at about 20 lb./in.² above atmospheric. Steam may be supplied to the second retort, the contents of which are agitated by stirrers. This retort may be either horizontal or

vertical; in the latter case the temperature is varied at different levels, being highest at the bottom.

A. B. MANNING.

Manufacture of water-soluble products from lignite and similar fossil materials. I. G. FARBER-IND. A.-G. (B.P. 284,670, 2.2.28. Ger., 3.2.27).—“Degraded” lignite is treated with chlorine in the presence of sufficient alkali to keep the solution neutral or weakly alkaline until the end of the process, when it is permitted to become acid. The products, which are precipitated from the acid solution or may be extracted therefrom by suitable organic solvents, are light-coloured water-soluble substances containing chlorine; they give the Congo-violet reaction and precipitate gelatin from solution. By “degraded” lignite is meant lignite which has been rendered soluble in water, e.g., by boiling with sodium sulphite solution.

A. B. MANNING.

Operation of internal-combustion engines with pulverulent fuel. J. Y. JOHNSON. From I. G. FARBER-IND. A.-G. (B.P. 310,220, 31.5.28).—Coke, particularly that derived from the low-temperature carbonisation of carbonaceous materials, is conveyed directly from the coking retort through a pulverising mill to the engine in such a way as to conserve as much as possible of its sensible heat. The fuel may be further preheated, if desired, and used with or without the addition of superheated steam. Pulverised coke at the ordinary temperature may be preheated, preferably to about 600°, by admixture with highly superheated steam, and then supplied to the engine.

A. B. MANNING.

Production of active charcoal. SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (Addn. No. 32,364, 9.9.26, to F.P. 623,455; B., 1928, 593).—Charcoal is heated gradually to 100° with constant stirring in the presence of sulphuric and phosphoric acids, and the mixture is subsequently heated at 500°.

L. A. COLES.

Production of active charcoal. G. WEGELIN (F.P. 631,332, 23.3.27).—Products obtained by the incomplete combustion of hydrocarbons are calcined in the presence of suitable gases or treated with activating material.

L. A. COLES.

Preparation of highly-active charcoal. A. SCHREINER (B.P. 292,566, 22.6.28. Ger., 22.6.27).—The carbonaceous matter (wood, peat, lignite, etc.) is impregnated with a solution of potassium thiocyanate (or ferrocyanide), dried, distilled at 300–350°, heated to bright redness in the absence of air for $\frac{1}{2}$ hr., and leached with as little water as possible.

J. A. SUGDEN.

Gas producers. F. L. BROUGHTON and D. HADLINGTON (B.P. 301,208, 3.12.27).—A gas producer is provided with a combination of a supply hopper and a valve chamber, the latter containing three valves which are operated by the rotation of suitably-shaped discs about a vertical axis. One of the valves is rotated intermittently to maintain the required supply of fuel to the producer, whilst another serves to open or close communication between the hopper and the producer. The third valve, which is normally held by a spring device, is designed to move with the intermittently rotated valve in the event of any hard body becoming jammed within the valves. The fuel delivered by the

valves falls on a rotary distributor, which spreads it uniformly over the fuel bed. A. B. MANNING.

Gas producers. S. C. G. EKLUND (B.P. 301,241, 30.1.28).—A gas producer for use with an uncoked fuel comprises an upper distillation chamber and a lower gasification chamber, preferably of the ash-melting type. The fuel is distilled by passing part of the producer gas through the upper chamber. The mixed gas so formed, after separation of the tar, if desired, is caused to re-enter and pass through the hot zone of the producer. The gas withdrawn from the main gas outlet in the upper part of the gasification chamber consists substantially of carbon monoxide, hydrogen, and nitrogen only. Moreover, it is at a sufficiently high temperature to enable it to be used for certain metallurgical purposes without further preheating. Instead of an upper distillation chamber, a retort or rotary furnace placed outside, preferably above, the producer may be used. A. B. MANNING.

Gas generators. HUMPHREYS & GLASGOW, LTD., Assees. of W. I. BATTIN and C. S. CHRISMAN (B.P. 294,523, 11.4.28. U.S., 25.7.27).—A gas generator which is provided with a cooling jacket has a metallic facing interposed between the fuel bed and the jacket wall. The facing presents a smooth surface permitting the unimpeded descent of the hot fuel; at the same time it reduces the flow of heat to the jacket, and so prevents undue cooling of the periphery of the fuel bed. The facing is preferably made up of detachable blocks, of which only the lower courses need be of metal, the upper courses being of refractory material. Air spaces may be provided in the facing if desired. A. B. MANNING.

Generation of gas. C. W. ANDREWS and W. B. CHAPMAN, ASSRS. to H. A. BRASSERT & Co., and WESTERN GAS CONSTRUCTION CO. (U.S.P. 1,709,335, 16.4.29. Appl., 3.7.26).—Water-gas is made in a generator having a relatively thin fire bed, an intermediate zone of which is maintained at a sufficiently high temperature during the air blast to melt most of the ash. The molten ash passes down to a solidifying zone in which an agitating member breaks up the clinker. A. B. MANNING.

Manufacture of water-gas. C. W. ANDREWS (U.S.P. 1,709,107, 16.4.29. Appl., 8.1.25).—Water-gas is made by alternately air- and steam-blasting the fuel bed of a dry-bottom generator with a stationary grate, maintaining in the bed an upper fuel zone, an intermediate clinker-formation zone, and a lower ash zone. The walls of the intermediate zone are water-cooled, and any large masses of clinker which form are broken up by grinding them against one another and against the walls of the generator by imparting an up-and-down agitation to the fuel and ash beds, the latter being also agitated from the centre outwards. A. B. MANNING.

Production of mixtures of water-gas and gases of distillation. W. SCHWEDER (B.P. 238,336, 5.4.28. Ger., 6.4.27).—The hot coke discharged from a gas retort is transferred to a water-gas generator into which steam is then introduced until the temperature of the coke has fallen too low for any further production of water-gas. Air is then blown through the coke and the

combustion gases thereby formed are used to preheat the fuel gas supplied to the retort and/or for steam raising. Part of the combustion gases may be used, before or after their utilisation in the waste-heat boiler, for dry-quenching the coke not used in the water-gas generator. A. B. MANNING.

Separation of gas mixtures. A. B. RAY, ASSR. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,685,883, 2.10.28. Appl., 4.5.23).—The absorption power of activated carbon is independent of its water-content, and in the separation, *e.g.*, of gasoline from natural gas, the carbon may be cooled with running water during the absorption process, and used again for absorption immediately after expulsion of the absorbed gas. R. BRIGHTMAN.

Gas filter. C. C. BOARDMAN, ASSR. to THERMATOMIC CARBON CO. (U.S.P. 1,710,469, 23.4.29. Appl., 24.9.27).—A number of vertical groups of filters for separating solid particles from a gas are connected to a common horizontal discharge duct, in which a liquid is maintained at a suitable level to form a seal. A pipe associated with the liquid seal controls the back-pressure which is set up in the duct. A. B. MANNING.

Gas purifier. F. E. LAMMERT (U.S.P. 1,709,530, 16.4.29. Appl., 24.6.24).—A central flue has lateral openings communicating with a surrounding, concentric cooling chamber. At each end of the latter are annular chambers for the cooling medium, connected by tubes passing through the cooling chamber and provided with partitions for suitably directing the flow of the cooling medium through the tubes. The flue itself is closed by an inclined partition wall, which also extends laterally into the cooling chamber. A. B. MANNING.

Elimination of sulphur compounds from gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,063, 20.10.27).—The gases are mixed with a sufficient amount of air to oxidise the sulphur compounds therein, and are passed over solid active adsorbents, *e.g.*, active charcoal, at 150–250°, or over alloys or compounds of the alkaline-earth metals, aluminium, or the heavy metals with non-metals of the 5th and 6th periodic groups (with the exception of oxygen) at about 400°. Ammonia, in amount necessary for the formation of the corresponding ammonium salts, may be added to the gases either before or after the catalytic treatment and the salts separated, or the sulphur oxides may be washed out with aqueous ammonia. A. B. MANNING.

Ionisation and hydrogenation of hydrocarbon starting material, vapour, and oil. Dissociation of hydrocarbon fluids and solids for production of gases and liquids of lighter mol. wt. I. W. HENRY, ASSR. to IONIZING CORP. OF AMERICA (U.S.P. 1,709,814–5, 16.4.29. Appl., [A] 4.8.27, [B] 20.3.28).—(A) The reaction chamber is surrounded by a coil through which a high-frequency oscillating current can be passed. The chamber is packed with stationary conducting elements which serve as short-circuited secondaries in the electric system of which the outer coil is the primary. The material to be treated and superheated steam are passed through the interspaces between the elements. (B) A chamber similar in design to that described above is constructed in the form of a still mounted on a chambered

foundation. Sludge which may be deposited on the heating elements or the walls of the still is burnt off by passing a current through the coil when the still is otherwise empty.

A. B. MANNING.

Distillation of tar and recovery of products therefrom. BARRETT CO., ASSEES. OF G. E. BRANDON (B.P. 282,367, 28.11.27. U.S., 14.12.26).—Tar, preferably dehydrated and preheated, is sprayed into the hot coke-oven gases in the uptake pipe from the coking chamber; it is thereby rapidly distilled and the resulting pitch is immediately withdrawn. The tar spray is placed at the upper end of the uptake pipe, whilst at the lower end is an inner pipe extending upwards from the chamber and having a baffle above its open end. The pitch is withdrawn from the annular space round this inner pipe. A pitch of any desired m.p. can be produced by suitably regulating the spraying operation or by recirculating the pitch through the still. It is necessary to modify only a few of the individual uptake pipes of a battery in order to distil the whole of the tar produced.

A. B. MANNING.

Manufacture of asphalt emulsions. W. H. SCHMITZ (B.P. 284,330, 24.1.28).—Bituminous material (more especially waste liquor obtained in the refining of lubricating oils etc.) is agitated with an alkaline slime containing 1% (calc. on the weight of emulsion) of fuller's earth; a stabiliser (*e.g.*, starch, gum, soap) may be added.

J. A. SUGDEN.

Removing amorphous wax and asphaltic material from [lubricating] oil. T. CLARKSON and H. R. HEAL (U.S.P. 1,686,437, 2.10.28. Appl., 29.10.21).—Petroleum-still residue, after removal of gasoline, kerosene, and lighter lubricating oil by distillation, and partial removal of asphaltic impurities by sulphuric acid and alkali treatment (either before or after the distillation), is treated with naphtha or other light petroleum distillate to reduce the viscosity. The mixture, *e.g.*, 60–80% of naphtha and 40–20% of heavy mineral oil, is cooled to 0° to –35°, depending on the "cold test" desired, and filtered under pressure at this temperature, using a filtering material of a close texture comparable with fine filter paper. The filtrate is passed in heat-exchange with the incoming mixture of oil and solvent, to cool the latter, and after treatment with, *e.g.*, fuller's earth, if desired, the solvent is distilled, giving a "bright stock" or cylinder oil of low cold test. The residue in the press is extracted with hot naphtha to dissolve almost the entire residue, and distillation of the naphtha gives a petroleum residue plastic at room temperature, m.p. 55–60°.

R. BRIGHTMAN.

Economical treatment of mixtures containing hydrocarbon and nitrogen in the electric arc. SOC. CHEM. IND. IN BASLE, and H. ANDRIESENS (B.P. 296,355, 29.8.28. Switz., 29.8.27).—In the preparation of acetylene from methane, and of hydrocyanic acid from hydrocarbon and nitrogen in the arc, it is necessary to dilute the gases with hydrogen in order to prevent the formation of soot. This dilution, however, reduces the yield per unit of energy consumed. By allowing both reactions to occur simultaneously the efficiency is greatly increased. The portion of acetylene

which is not used in the formation of hydrocyanic acid in the second reaction is separated in amounts sufficient to prevent the formation of soot. A mixture of methane, nitrogen, and hydrogen is circulated through a suitable arc system (B.P. 195,239; B., 1923, 498 A) until the concentration of hydrocyanic acid is about 1% and of acetylene 2–3% by vol. A fraction of the gases is withdrawn and passed first through a solution of caustic soda and then through active charcoal in order to remove the hydrocyanic acid and acetylene. The gases are then returned to the circulator.

J. A. SUGDEN.

Production of liquid hydrocarbons. F. J. M. HANSEN (B.P. 284,224, 24.1.28. Switz., 24.1.27).—In the process for hydrogenating hydrocarbons in the electric discharge, the dissociation of the hydrogen is inhibited by the presence of the hydrocarbon. This difficulty is overcome by allowing the dissociation to take place before admixing the hydrocarbon vapour. The dissociation of the hydrogen is maintained by periodically interrupting the admission of hydrocarbon vapour.

J. A. SUGDEN.

Apparatus for cracking oil. G. EGLOFF and W. R. HOWARD, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,683,766, 11.9.28. Appl., 23.3.21. Renewed 3.7.26).—The heating tube contains a helical agitator rotated by the passage of the oil, and the agitator has a thrust bearing at the discharge end of the tube.

R. BRIGHTMAN.

Flue-gas recirculating system for cracking-still operations. H. A. ATWATER, ASSR. to COMBUSTION EQUIPMENT CO. (U.S.P. 1,709,764, 16.4.29. Appl., 9.5.27).—Combined with the furnace combustion chamber is an initial fuel-combustion zone and a second heat-exchange zone for the still-heating surfaces. A portion of the waste flue gases from the second zone is returned to the combustion chamber through separate discharge ducts opening at opposite sides of the initial combustion zone, which is protected from any chilling effect by partition structures above and below.

H. S. GARLICK.

Cracking of hydrocarbons. J. E. BELL, ASSR. to SINCLAIR REFINING CO. (U.S.P. 1,688,325, 23.10.28. Appl., 22.1.25).—Hydrocarbon oil is circulated from a main supply drum to the lower end of vertical heating tubes, over which the heating gases pass downwards, the velocity being increased, by means of baffles, as their temperature falls. The heated oil leaves the upper end at cracking temperature, and is discharged to the main supply drum, where cracking continues. The vapours escape to a reflux tower and the oil continues in circulation.

R. BRIGHTMAN.

Cracking of hydrocarbons. G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,688,859, 23.10.28. Appl., 20.12.20. Renewed, 21.11.27).—From 10 to 60% by vol. of a solid bitumen in colloidal form is dispersed in a hydrocarbon oil, *e.g.*, fuel oil, heavy Mexican or Californian crudes, and the mixture is cracked at 400–480° under at least 50 lb./in.²

R. BRIGHTMAN.

Cracking of hydrocarbons. J. PERL, ASSR. to SINCLAIR REFINING CO. (U.S.P. 1,689,362—3, 30.10.28.

Appl., 20.11.24).—To prevent or reduce the formation of carbonaceous deposits, the tubular heating coil is lined with (A) tin or (B) iron sulphide. R. BRIGHTMAN.

Manufacture of lighter products from hydrocarbons. F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,686,490, 2.10.28. Appl., 16.8.22).—The still containing the hydrocarbon oil is submerged in a liquid heating medium, *e.g.*, molten lead, contact being broken immediately distillation is complete. R. BRIGHTMAN.

Treating [cracking hydrocarbon] oils. R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,683,801, 11.9.28. Appl., 7.3.23. Renewed, 15.3.28).—The raw oil is fed to the heating coil after heat-exchange in the dephlegmator out of contact with the cracked vapours. R. BRIGHTMAN.

Treatment [cracking] of hydrocarbon oils. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,686,654, 9.10.28. Appl., 18.8.28).—Relatively heavy hydrocarbon oil is brought to cracking temperature while circulating under pressure with relatively high velocity in a heating coil. The heated oil is passed into tanks under lower pressure, the vapours escaping to a condenser, and the unvaporised oil is recirculated to the heating coil, its relatively slow passage through the tanks allowing free carbon and other solids to deposit therein. R. BRIGHTMAN.

Cracking of [hydrocarbon] oil. **Cracking of petroleum oil.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,688,860—1, 23.10.28. Appl., [A] 31.10.21, [B] 17.10.23. Renewed, [A] 12.3.26, [B] 28.5.28).—(A) Hydrocarbon oil is raised to cracking temperature under pressure, and the unvaporised oil drawn off from the vaporiser is submitted to increased pressure and temperature in a top-fired secondary zone. The dephlegmated products from each stage are collected separately. (B) The residuum accumulating in the vaporiser or reaction vessel of a cracking apparatus is cooled and agitated with liquid sulphur dioxide. The upper layer is withdrawn, freed from sulphur dioxide by sodium hydroxide treatment, and worked up for lubricating stock; the bottom layer after distilling off liquid sulphur dioxide stock affords cylinder oil stock. R. BRIGHTMAN.

Treating [cracking] residual [hydrocarbon] oils. L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,683,826, 11.9.28. Appl., 26.5.24).—Residual oil is withdrawn from a cracking still at above 200° and the pressure released in an expansion chamber. The vapours are taken off and the residual oil is strained and circulated to burners, any excess returning under pressure to the bottom of the expansion chamber with turbulence to prevent deposition of carbon. R. BRIGHTMAN.

Preparation of fuel oil. W. S. HUGHES and J. HARROP, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,686,491, 2.10.28. Appl., 19.1.27).—The residual oil resulting from the distillation of hydrocarbon oil in presence of alkali is cooled, fluxed with 5–50% of gas oil or other hydrocarbon oil, and emulsified with water. The emulsion is broken, *e.g.*, with dilute sul-

phuric acid, sufficient acid being added to decompose the salts of naphthenic acids or other organic acids and to leave an acid solution, and the oil and aqueous layers are separated. The oil may be distilled to recover the fluxing oil and the naphthenic acids. R. BRIGHTMAN.

Treating [cracked hydrocarbon] oils. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,683,767, 11.9.28. Appl., 24.3.24. Renewed, 20.2.28).—Residue, $d_{40}^{20} 0.909$ —1.06, from a cracking process is cooled below 95°, mixed with lighter oil, and centrifuged to give a coke-free fuel oil. R. BRIGHTMAN.

Petroleum-cracking process and apparatus. J. J. JAKOSKY (U.S.P. 1,689,590, 30.10.28. Appl., 15.10.25).—A series of vertical tubes allow the cracked vapours to escape readily from the horizontal cracking tube. Liquid particles are removed from these vapours by electrical precipitation methods, the liquid being returned to the cracking tube and the stripped vapours removed and condensed. The temperature of the vertical-tube walls and of the precipitation electrodes is high enough to prevent condensation of desired products, but low enough to avoid further cracking. R. BRIGHTMAN.

Conversion [cracking] of petroleum oils. A. D. DAVID, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,688,855, 23.10.28. Appl., 14.2.23. Renewed, 24.4.28).—Petroleum oil is raised to cracking temperature in a heating coil and passed into an expansion chamber, where a swirling motion is maintained by means of baffle plates and local circulation of oil with a pressure pump. The carbon particles resulting from the cracking process remain in suspension, and the residual oil is continuously drawn off into settling tanks, the carbon being separated before the oil comes into circulation again. The vapours escape from the expansion chamber through a dephlegmator in the usual manner, the condensate being circulated through the heating coil. R. BRIGHTMAN.

Refining of hydrocarbon oils. E. B. PHILLIPS and J. G. STAFFORD, Assrs. to GRAY PROCESSES CORP. (U.S.P. 1,687,992, 16.10.28. Appl., 29.5.26).—Petroleum distillates free from water are treated successively with crystalline cupric chloride and fuller's earth, or other suitable solid absorbent. R. BRIGHTMAN.

Refining of oils etc. W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,686,493, 2.10.28. Appl., 23.8.23).—Oil is clarified with, *e.g.*, fuller's earth, and passes forward by decantation in a series of units. Refined oil is drawn off at the end of the series, and the earth in the first unit is used for a fresh charge of oil until it is no longer efficient. The soakage oil is removed, and the spent earth is extracted with solvent naphtha, dried, and discharged, for revivifying if desired, as a sandy powder. The unit is recharged with fresh earth and brought back into operation as the final unit of the series, crude oil being introduced at the second, third, fourth, etc. units in turn as the earth in each becomes inefficient. R. BRIGHTMAN.

Production of hydrocarbons of higher b.p. from gaseous and low b.p. hydrocarbons. A. S.

RAMAGE, ASSR. to GYRO PROCESS CORP. (U.S.P. 1,687,890, 16.10.28. Appl., 13.5.25).—Hydrocarbon vapours, *e.g.*, from the lighter fractions of casing-head gasoline, are mixed with about 10% of steam and passed over ferrous oxide at 600–700°, giving a motor fuel, b.p. 20–210°, consisting essentially of *cycloparaffins*, *cycloolefines*, and polymerised olefines. R. BRIGHTMAN.

Imparting fluorescence to [lubricating] oil. Fluorescent product. J. C. BLACK, W. D. RIAL, and J. R. MCCONNELL, ASSRS. to PAN AMER. PETROLEUM CO. (U.S.P. 1,708,563 and 1,708,602, 9.4.29. Appl., [A] 13.8.26, [B] 6.5.27).—(A) Fluorescent substances are extracted from uncracked petroleum pitch with a non-fluorescent lubricating oil, which is then separated therefrom. (B) A concentrated extract of green fluorescent substances is obtained by mixing a mineral lubricating oil with a coal-tar product containing them and then separating the lubricating oil from the insoluble coal-tar residue. H. S. GARLICK.

Renovation of journal-box oil. T. W. POTTER, L. C. McNAMARA, C. N. LAMMERS, and J. R. McLAVY, ASSRS. to JOURNAL-BOX SERVICING CORP. (U.S.P. 1,709,230, 16.4.29. Appl., 9.5.28).—Oil is separated from lint and foreign matter by distillation in a closed tank under a controlled pressure insufficient to bring about cracking. H. S. GARLICK.

Preparation of valuable compounds from the refining wastes of cracked products, particularly cracked benzenes. GALICYJSKIE TOWARZYSTWO NAF-TOWE "GALICJA" S.A., W. DE PIOTROWSKI, and J. WINKLER (B.P. 309,718, 26.3.28).—Waste sulphuric acid from the refining of cracked benzene is diluted with water or dilute alkali (*e.g.*, waste lye from the subsequent soda-washing of the benzene) and the aqueous and oily layers are treated separately. The aqueous layer is neutralised and distilled with superheated steam, and the distillate (b.p. 80–150°) is then rectified giving, *e.g.*, butyl, amyl, and hexyl alcohols. The oily layer is washed with alkali and with water and is then heated to 110° and steam is introduced. The fraction of b.p. 50–200° is refined by means of alkali hydroxide and sodium plumbite and redistilled, giving a yellow fraction, b.p. 140–200°, flash point not below 50°, which has a characteristic odour and possesses insecticidal properties; the fraction of b.p. 200–280° is boiled with a manganese or cobalt siccative to give a varnish-like product. The residue, b.p. above 280°, is cooled to 140° and is then air-blown, yielding asphalt-like products of high elasticity, which, being soluble in benzene, can be employed as lacquers. W. S. NORRIS.

Heat-treatment of hydrocarbon gases. ANGLO-PERSIAN OIL CO., LTD., A. E. DUNSTAN, and R. V. WHEELER (B.P. 309,455, 8.10.27).—For the production of aromatic hydrocarbons, a stream of gas containing gaseous paraffins is gradually heated to a temperature (*e.g.*, below 550°) substantially below that at which eventual decomposition is to be carried out. It is then heated in a determined course to a temperature (650–975°) at which the paraffins are decomposed and aromatic hydrocarbons are formed. The stream of gas is then suddenly reduced in velocity, *i.e.*, expanded, and cooled, and the free carbon formed is deposited; finally

the aromatic hydrocarbons are condensed. If necessary, the original gas is previously freed from hydrogen sulphide, *e.g.*, by limited oxidation and deposition of elementary sulphur, and from condensable hydrocarbon vapours. The yield of aromatic hydrocarbons is increased by diluting the gas with an inert gaseous material (steam), which is preferably introduced into the gas stream between the preheater and the reaction tubes. The reaction temperature is controlled according to which paraffin or mixture of paraffins is present. The temperatures are 850–975°, 800–900°, 750–850°, 700–800°, or 650–750° for methane, ethane, propane, butane, or pentane, respectively; for mixtures, a range of temperature is chosen according to the average proportions of the constituents. The velocity of the gas in the preheater tubes and in the reaction tubes is so maintained that the flow is turbulent, giving good heat-transference and maintaining the carbon particles in suspension. W. S. NORRIS.

Manufacture of nitrogenous bases from hydrocarbon materials. H. K. IHRIG, ASSR. to S. E. CAMPBELL and ASSOCIATED OIL CO. (U.S.P. 1,686,136, 2.10.28. Appl., 21.12.26).—Crude gasoline, after removal of sulphur compounds by treatment with sodium hydroxide, is treated with an acidic reagent, *e.g.*, 25% sulphuric acid. The acid liquor is made alkaline and steam-distilled to separate the nitrogenous bases soluble in water from those insoluble therein. The products have insecticidal properties. R. BRIGHTMAN.

Apparatus for distilling bituminous substances. H. KOPPERS (U.S.P. 1,712,083, 7.5.29. Appl., 29.9.22. U.K., 28.6.22).—See B.P. 203,444; B., 1924, 548.

Apparatus for treatment of liquid hydrocarbons. S. J. M. AULD, A. E. DUNSTAN, and P. H. HERRING, ASSRS. to ANGLO-PERSIAN OIL CO., LTD. (U.S.P. 1,711,219, 30.4.29. Appl., 11.2.24. U.K., 20.2.23).—See B.P. 220,664; B., 1924, 899.

Apparatus for grinding coal and like material. W. T. BELL and J. F. BENNETT (B.P. 311,505, 20.3.28).

Conveying and quenching of coke. W. J. JENKINS & CO., LTD., and R. M. GOODMAN (B.P. 311,487, 28.2.28).

Preparation of charges for use in internal-combustion engines. A. S. JOHN (B.P. 311,165, 4.9.28).

Burner for coal dust firing. BÜTTNER-WERKE A.-G., and H. ZIKESCH (B.P. 311,019, 13.2.28).

Agitators or mixing devices. [Fuel burners.] A. C. PRIOR (B.P. 308,054, 21.2.28).

Carbide cartridge for use in acetylene gas lamps. C. A. MONHAM and E. G. FIRTH (B.P. 311,517, 31.12.28).

Apparatus [press] for expressing liquid from solid materials [*e.g.*, peat]. H. SKÖLDBERG (B.P. 284,318, 13.1.28. Swed., 29.1.27).

Drying etc. of granular materials (U.S.P. 1,705,617). **Simultaneous drying and grinding** (U.S.P. 1,702,333). **Separating volatile fluids from liquids** (B.P. 308,010). **Separating dust, smoke, etc. from smoke** (B.P. 307,168).—See I. Lead tetraethyl (U.S.P. 1,690,075).—See VII. Road surfaces (B.P. 301,891 and 309,634).—See IX. Flotation

oil (U.S.P. 1,688,975). Anti-corrosive preparation (B.P. 310,640).—See X. Dryers for drying oils (U.S.P. 1,686,484—6).—See XIII.

III.—ORGANIC INTERMEDIATES.

Preparation of acetic anhydride. E. FRITZMANN (J. angew. Chem. [Russia], 1928, 1, 1, 27—36; Chem. Zentr., 1928, ii, 2548).—Optimal conditions for the preparation of acetic anhydride by the action of chlorine on a mixture of acetate and sulphur (G.P. 372,528; B., 1923, 1200 A), and by the action of toluene-*p*-sulphonyl chloride on an acetate are described.

A. A. ELDRIDGE.

Simple method for the determination of acetaldehyde. Y. TOMODA (J.S.C.I., 1929, 48, 76—77 T).—The relation between the p_H and the dissociation of the sodium hydrogen sulphite compound of acetaldehyde has been investigated; at p_H 6—8 dissociation is inappreciable, but above p_H 8 it increases rapidly, becoming complete at about p_H 12. Thus, using 0.1*N*-iodine, below p_H 2 free sodium hydrogen sulphite only is titrated, and at about p_H 8 the combined sodium hydrogen sulphite is also titrated. Since hydriodic acid is produced, the p_H of the medium falls, necessitating the addition of alkali; if the alkalinity of the solution rises above p_H 9 oxidation of the aldehyde by the iodine occurs, so that in order to titrate both free and combined sodium hydrogen sulphite the p_H of the medium must be maintained at about p_H 8; this is accomplished by addition of a large excess of sodium hydrogen carbonate. By employing these conditions, a simple and accurate method for the determination of acetaldehyde is obtained, which can be applied not only to the direct determination of acetaldehyde in the presence of sulphite, but also to the converse case. Procedure is described in detail.

C. W. SHOPPEE.

Determination of alcohol in the presence of acetaldehyde. Y. TOMODA (J.S.C.I., 1929, 48, 77—79 T).—When an aqueous solution of acetaldehyde is subjected to aeration, volatilisation of the aldehyde can be prevented completely by sodium hydrogen sulphite, provided that the solution has an acid reaction. On this basis a method for the determination of alcohol in the presence of acetaldehyde is described, which is accurate to 3%. By employing a large excess of sodium hydrogen sulphite the volatilisation of the alcohol is accelerated owing to its increased relative partial pressure, due to the presence of the dissolved salt. The presence of formic acid, acetic acid, glycerol, or sucrose does not interfere with the method, and with dilute solutions containing less than 1% of alcohol, increased accuracy may be obtained by using a larger volume of the solution under test and an increased amount of sodium hydrogen sulphite. Apparatus and procedure are fully described.

C. W. SHOPPEE.

Bromometric determination of phenol and the cresols. H. DITZ (Z. anal. Chem., 1929, 77, 186—202).—Polemical against Järvinen (B., 1927, 596) and a claim for priority together with a critical survey of recently published methods for the bromometric determination of phenols.

A. R. POWELL.

Determination of nitrogen in dyes and their intermediate products by the Kjeldahl method. P. SISLEY and M. DAVID (Bull. Soc. chim., 1929, [iv], 45, 312—324).—Low results obtained in the determination of nitrogen in nitrobenzene, *p*-nitrotoluene, and *o*-chloronitrobenzene, using the modified Kjeldahl method of Sisley (B., 1907, 605), are due to volatilisation; accurate results are obtained if the sample is first sulphonated, *e.g.*, by heating with 30% oleum at 100—120°. The method is not general, the loss being reduced only with *o*-nitrotoluene, *m*-dinitrobenzene, *p*-chloronitrobenzene, picric acid, and dinitronaphthol (calcium salt), whilst in the case of dinitrophenetole and hexanitrodiphenylamine preliminary sulphonation leads to lower results. With picric acid the loss of nitrogen by Sisley's method (*loc. cit.*) is 25%, and is not reduced by the addition (to 0.5 g. of picric acid) of 1 g. of any of the following substances: benzene, toluene, benzoic acid, phthalic anhydride, oxalic, acetic, or formic acids. Phenol, furfuraldehyde, tartaric acid, acetone, and gallic acid appreciably reduce the loss, and accurate results are obtained in the presence of 1 g. of dextrose, sucrose, benzoquinone, resorcinol, anthracene, lactic acid, β -naphthol, quinol, pyrocatechol, and pyrogallol, the last three giving the best results; accurate results are obtained in presence of 0.3 g. of pyrogallol with many nitro-compounds and with nitroso- and azoxy-compounds, but not with all azo compounds. Since replacement of the pyrogallol by 5 g. of phosphorous acid gives accurate results, the favourable action of pyrogallol is attributed to its reducing action on the sulphuric acid. Sisley's method (*loc. cit.*) is satisfactory for azo compounds containing the azo group *para* to the hydroxyl or amino-group, but *o*-hydroxy- or *o*-amino-azo compounds yield low results. The following method gives accurate results for nitrogen in azo compounds generally, and in intermediate products with the exception of pyrazolones (cf. Ranker, B., 1927, 536). The substance (0.5—1 g.) is warmed in a 250 c.c. pyrex flask with 10 c.c. of alcohol and 5 c.c. of water, 2—4 g. of sodium thiosulphate are added, 1 g. at a time and with boiling and cooling after each addition, followed by 10 c.c. of sulphuric acid, *d* 1.84, and the flask is gently heated to expel alcohol; when the liquid becomes spongy 0.5 g. of copper sulphate, 6—8 g. of potassium sulphate (10 g. less the weight of thiosulphate used), and 12 c.c. of sulphuric acid are added. The mixture is heated until the liquid is clear blue (20—30 min.), diluted to 300 c.c., and the ammonia determined as usual, 5 c.c. of 20% sodium sulphide solution and a little granulated zinc being added with the sodium hydroxide before distillation.

R. BRIGHTMAN.

Unsaturated compounds as germicides. CHEESEWORTH and COOPER.—See XXIII.

PATENTS.

Apparatus for production of carbon disulphide. I. G. FARBERNIND. A.-G. (B.P. 291,382, 30.5.28. Ger., 31.5.27).—A subsidiary superheating retort (A) for the sulphur vapour is combined in one casting with the main retort (to contain carbon) and extends along its entire length. Retort A is filled with refractory material resistant to sulphur, or, preferably, has horizontal ribs

cast in its inner wall to support flat dishes of refractory material which are assembled so that openings therein are staggered with respect to each other, thereby lengthening the path of the sulphur vapour. The superficial area of this retort is at least one half of that of the main retort, and the lining of the latter covers the opening to retort A from the top nearly to the bottom.

W. G. CAREY.

Manufacture of olefine alcohols. F. W. LOMMEN, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,688,083, 16.10.28. Appl., 7.6.26).—Olefinic aldehydes are reduced by heating with a paraffin alcohol in presence of aluminium alkoxide as catalyst. Thus cinnamaldehyde, when heated at 100° with a slight excess of butyl alcohol containing aluminium ethoxide, affords a 95% yield of cinnamyl alcohol, with butyl butyrate as by-product.

R. BRIGHTMAN.

Dehydration of alcohols. S. R. MERLEY, Assr. to DOHERTY RES. CO. (U.S.P. 1,688,731, 23.10.28. Appl., 22.12.25).—Amyl and higher alcohols are dehydrated by adding a lower alcohol, *e.g.*, *sec.*-butyl alcohol from the still butts obtained in its rectification, which forms an azeotropic mixture with water. Volatile odorous constituents are removed at the same time.

R. BRIGHTMAN.

Hydrolysis of methyl chloride. R. H. MCKEE (U.S.P. 1,688,726, 23.10.28. Appl., 7.9.22).—Methyl chloride, from the limited chlorination of natural gas or methane, is scrubbed to remove hydrochloric acid, mixed with an equal volume of steam, *e.g.*, by bubbling through water at an appropriate temperature, and passed over an alkaline-earth hydroxide, *e.g.*, aluminium, calcium, barium, or magnesium hydroxide, at 350–375° in an aluminium tube. The aqueous solution obtained on cooling the issuing gases affords methyl alcohol and some methyl ether, free from acetone.

R. BRIGHTMAN.

Synthesis of [organic] nitrogen compounds, and of liquid hydrocarbons. H. E. POTTS. From H. PLAUSON (B.P. 309,001–2, 30.12.27).—(A) Mixtures of nitrogen and water-gas or Dowson gas containing suspended, finely-divided particles of magnesia or other catalyst are subjected to the action of β - and X-rays, and the product is then subjected to high temperature and pressure. Carbamide, uric acid, and ammonium carbamate are stated to be among the products of the reaction. (B) Mixtures of gaseous saturated and/or unsaturated hydrocarbons or of hydrogen and carbon monoxide are subjected to a similar treatment as in (A) to obtain liquid hydrocarbons having b.p. below 150°.

A. R. POWELL.

Catalytic manufacture of thiocarbamides. C. N. HAND and H. P. ROBERTS, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,688,707, 23.10.28. Appl., 1.10.24).—Aromatic amines are heated at 46–46.2° with about 60% of carbon disulphide in presence of an alkaline catalyst volatile below 75°, *e.g.*, 0.1–0.25% of aqueous ammonia, *d* 0.897. The hydrogen sulphide vapours evolved are scrubbed by passing through a tank containing a second charge of amine and catalyst, this charge being admitted to the mixer when the reaction in the first charge is complete, and the excess of carbon disulphide and catalyst have been recovered by distillation at 75°.

R. BRIGHTMAN.

Purification of benzoic acid and its derivatives.

A. O. JAEGER, Assr. to SELDEN CO. (U.S.P. 1,686,913, 9.10.28. Appl., 18.10.27).—Benzoic acid is separated from phthalic acid by distillation with pressure-steam below 191° (at which temperature phthalic acid is converted into its anhydride). The aqueous solution may be distilled under pressure, and the process made continuous by means of a countercurrent of steam, or the mixture may be vaporised and blown into a vessel counter to a blast of superheated steam; the vapours may be removed and cooled so that benzoic acid but very little water condenses, or the benzoic acid may be extracted from the steam above 100° with high-boiling solvents. Benzoic acid may similarly be recovered from effluent converter gases by treating the gases with steam or steam and water to reduce the temperature below 191°, when the phthalic acid separates. By treating the gases from a vapour-phase catalytic oxidation of naphthalene with a carbon dioxide-splitting catalyst and sufficient steam, the production of benzoic acid from naphthalene can be effected in one operation.

R. BRIGHTMAN.

Production of styrene from chloroethylbenzene.

O. H. SMITH, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,687,903, 16.10.28. Appl., 4.10.27).—Halogen derivatives of alkylbenzenes, *e.g.*, the mixture of α - and β -chloroethylbenzenes obtained by chlorinating ethylbenzene below 30°, or the mixture of *o*-, *m*-, and *p*-chloroethylbenzenes, b.p. 90–105°/50 mm., obtained from chlorobenzene and ethylene in presence of aluminium chloride, are heated to 600–725° in a cracking tube, and the styrene or alkylstyrene is separated in known manner.

R. BRIGHTMAN.

Production of phthalic anhydride.

F. A. CANON and C. E. ANDREWS, Assrs. to SELDEN CO. (U.S.P. 1,689,860, 30.10.28. Appl., 7.11.25).—Temperature control in the catalytic oxidation of naphthalene is effected by a cadmium-mercury alloy, containing 25% Cd, b.p. about 400°.

R. BRIGHTMAN.

cycloHexyl alkyl phthalates.

R. ADAMS and J. F. HYDE, Assrs. to NEWPORT CO. (U.S.P. 1,689,761, 30.10.28. Appl., 11.6.26).—Phthalic anhydride is heated with 1 mol. of cyclohexanol or a substituted cyclohexanol, and the reaction product esterified with an alcohol in presence of an acid catalyst. *cycloHexyl hydrogen phthalate*, m.p. 95°, *cyclohexyl methyl phthalate*, b.p. 168°/2 mm., *cyclohexyl ethyl phthalate*, b.p. 180°/5 mm., 219–220°/21 mm., *cyclohexyl n-propyl phthalate*, b.p. 173–178°/1.5–2 mm., *cyclohexyl isopropyl phthalate*, b.p. 168–172°/1.5–2 mm., and *cyclohexyl n-butyl phthalate*, b.p. 186–188°/1.5–2 mm., are described.

R. BRIGHTMAN.

Organic solvents. [cycloHexyl alkyl phthalates.]

R. ADAMS and J. F. HYDE (U.S.P. 1,689,762, 30.10.28. Appl., 8.12.26).—*cycloHexyl alkyl phthalates* are used as solvents for cellulose esters. The following are described: *4-Methylcyclohexyl ethyl phthalate*, b.p. 160°/3 mm.; *4-methylcyclohexyl n-butyl phthalate*, b.p. 183°/2–2.5 mm.; *3-methylcyclohexyl ethyl phthalate*, b.p. 168°/3.5 mm.; *3-methylcyclohexyl n-butyl phthalate*, b.p. 178°/2 mm.; *2-methylcyclohexyl ethyl phthalate*, b.p. 168°/3.5 mm.; *2-methylcyclohexyl n-butyl phthalate*, b.p. 185°/3 mm.

R. BRIGHTMAN.

Reduction of aromatic nitro-compounds. P. DIETERLE, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,689,014, 23.10.28. Appl., 4.1.26).—Aromatic nitro-compounds are reduced to azoxy-, azo, hydroxy-, amino-, and nitroamino-compounds by heating with ferrous sulphide in neutral or alkaline media in presence or absence of organic solvents. Thus 2:4-dinitrophenol is converted into 4-nitro-2-aminophenol by heating its sodium salt at 40–80° for $\frac{1}{2}$ –2 hrs. with precipitated ferrous sulphide. Similarly, equal parts of *m*-dinitrobenzene and ferrous sulphide in water at 80–85° for 1–2 hrs. yield *m*-nitroaniline, and nitrobenzene is converted into aniline by heating it in 3% sodium hydroxide solution at 99–102° with ferrous sulphide for 2 hrs. With 6 pts. of nitrobenzene and 4.5–6 pts. of ferrous sulphide in 15 pts. of 40–48% sodium hydroxide at 80–85° for 2–3 hrs. about 20–25% of aniline and 65–75% of azobenzene are obtained. After removal of aniline and dilution to give 20–30% sodium hydroxide, the azobenzene can be converted into hydrazobenzene by heating at 70–80°.

R. BRIGHTMAN.

Production of polymerised vinyl acetate. W. O. HERRMANN and W. HAEHNEL, Assrs. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,710,825, 30.4.29. Appl., 6.11.26. Ger., 14.11.25).—See B.P. 261,406; B., 1927, 823.

Manufacture of *o*-aminodiaryl ethers. A. ZITSCHER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,703,915, 5.3.29. Appl., 16.12.27. Ger., 24.12.26).—See B.P. 307,130; B., 1929, 349.

Preparation of homogeneous pure 2:3- and 2:5-dichloro-4-amino-1-methylbenzene [2:3- and 2:5-dichloro-*p*-toluidines]. W. SCHUMACHER and C. SEIB, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,712,173, 7.5.29. Appl., 3.2.28. Ger., 30.3.26).—See B.P. 294,078; B., 1928, 704.

Manufacture of tetranitrodianthrone. B. STEIN, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,686,992, 9.10.28. Appl., 28.6.26. Ger., 25.6.25).—See B.P. 289,958; B., 1928, 516.

Preparation of monodiazo compounds of 1:4-diaminoanthraquinone- β -sulphonic acids. G. KRÄNZLEIN and F. ROEMER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,021, 30.10.28. Appl., 18.1.27. Ger., 25.1.26).—See B.P. 264,879; B., 1928, 8.

Apparatus for chemical reactions (U.S.P. 1,705,614).—F.p. depressants (U.S.P. 1,687,094).—See I. Treating hydrocarbon-nitrogen mixtures (B.P. 296,355). Products from waste cracked benzenes (B.P. 309,718).—See II. Flotation of ores (U.S.P. 1,686,529).—See X. Light filter (U.S.P. 1,688,259).—See XI. Stable sulpho-acids and salts (B.P. 288,126).—See XII. Vapours from alcohol (B.P. 310,400 and 310,403).—See XVIII.

IV.—DYESTUFFS.

Gases emitted in the manufacture of sulphur dyes. G. V. KOGAN (J. Chem. Ind. Moscow, 1928, 5, 799–801).—A study of the quantities of hydrogen

sulphide and ammonia which are evolved in definite time intervals in the manufacture of sulphur dyes, and of the conditions of maximum evolution.

CHEMICAL ABSTRACTS.

Determination of nitrogen in dyes. SISLEY and DAVID.—See III.

PATENTS.

[Preparation of] dyes of the anthraquinone series. K. WEINAND, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,688,256, 16.10.28. Appl., 23.12.27. Ger., 20.12.26).—*N*-Substituted aminoanthraquinone dyes giving light-fast, pure blue shades on animal fibres are obtained by the action of alicyclic amines, not of the hydrogenated benzene or naphthalene series, on a halogenated anthraquinonesulphonic acid in presence of a copper catalyst. Thus sodium 4-bromo-1-aminoanthraquinone-2-sulphonate in aqueous alcohol in presence of copper sulphate affords 1-amino-4-cyclopentylaminoanthraquinone-2-sulphonic acid and 1-amino-4-bornylaminoanthraquinone-2-sulphonic acid.

R. BRIGHTMAN.

[Production of] monoazo dyes from pyrazolones and anilinepolysulphonic acids. L. W. GELLER, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,688,044, 16.10.28. Appl., 23.12.25).—Yellow to red or brown dyes for wool are obtained by coupling a diazotised anilinedisulphonic acid with a 1-aryl-3-methyl-5-pyrazolone or 1-aryl-5-pyrazolone-3-carboxylic acid, e.g., aniline-2:4-disulphonic acid \rightarrow 1-(4-sulpho-2-tolyl)pyrazolone-3-carboxylic acid. R. BRIGHTMAN.

[Production of] monoazo dyes from disulphonaphthylpyrazolones and *o*-aminophenols. L. W. GELLER, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,688,045, 16.10.28. Appl., 23.12.25).—Monoazo dyes for lakes and pigments or for dyeing wool from an acid bath in yellow to red or brown shades, changed to yellowish- or bluish-red on after-chroming, are obtained by coupling a diazotised *o*-aminophenol-carboxylic or -sulphonic acid with a 1-(4:8-disulpho-2-naphthyl)-5-pyrazolone-3-carboxylic acid or the corresponding 3-methylpyrazolone.

R. BRIGHTMAN.

Readily-soluble vat colour preparations. W. BAUER, A. HERRE, and A. FUNKE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,711,008—9, 30.4.29. Appl., [A] 29.11.24, [B] 17.5.26. Ger., 10.12.23).—See F.P. 589,914; B., 1926, 154.

Preparation of dyes derived from diphtalimidoanthraquinones. E. G. BECKETT and J. THOMAS, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,711,873, 7.5.29. Appl., 28.7.24. U.K., 14.9.23).—See B.P. 231,206; B., 1925, 393.

Monoazo dye. W. DUISBERG, W. HENTRICH, and L. ZEH, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,711,930, 7.5.29. Appl., 16.1.25. Ger., 28.2.24).—See B.P. 230,055; B., 1925, 875.

Manufacture of azo dyes for dyeing cellulose esters. O. KNECHT, Assr. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,711,390—1, 30.4.29. Appl., [A] 23.12.25, [B] 16.3.27. Ger., [A, B] 1.6.25).—See B.P. 245,758; B., 1927, 469.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of wool in raw- and wool-felt pastes by chemical means. B. SCHULZE (Papier-Fabr., 1929, 27, 299—301).—Dissolution of the wool by treating a weighed sample of the material with boiling 2% sodium hydroxide solution under standard conditions and reweighing the residue gives results for the wool content which are too high, since other materials besides the wool are thereby partially dissolved. Thus, cotton loses 4.2%, jute 16.3%, wood cellulose 4.9%, and mechanical wood pulp 4.7% by weight under these conditions. On the other hand, if the vegetable fibres are removed by sulphuric acid, leaving the wool unchanged, better results are obtained. The material is treated with 80% sulphuric acid for 3 hrs. with shaking, the mixture is poured into a large volume of water, the wool residue washed to neutrality, dried at 105—110°, and weighed. It is then found that a 0.04% increase in weight of the wool occurs, cotton is completely dissolved, whilst jute leaves 0.8%, wood cellulose 0.1%, and mechanical wood pulp 12.1% of insoluble matter. These differences are negligible except in the case of mechanical wood pulp, but use of the latter in the materials referred to is forbidden, so that its adverse effect may be disregarded. Results obtained for the analysis of different known mixtures show that the sodium hydroxide method gives values 5—9% higher than the theoretical, whereas those for the sulphuric acid method vary from 0.5% too low to 0.1% too high. Natural silk, and viscose, nitrate, cuprammonium, and acetate silks are all completely dissolved by the above acid treatment, but Tussah silk leaves a residue of about 10%. B. P. RIDGE.

PATENTS.

Article made of fibrous material and its production. W. H. KOBBE, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,689,394, 30.10.28. Appl., 14.3.27).—Fibrous material is treated with wet, calcined gypsum, shaped, and, after hardening and drying, impregnated with molten sulphur. R. BRIGHTMAN.

Production of fibres from coconuts. L. THOMAS and E. ELÖD (B.P. 310,396, 24.1.28).—Coconut husks are steeped in hot water which is maintained in motion, sufficient lime being added to prevent discoloration of the fibre. After subjection to mechanical disintegration they are given a further steeping, the fibres being finally freed and isolated in the usual way. F. R. ENNOS.

Manufacture of cellulose acetate. J. W. BULMER (B.P. 300,824, 2.1.28).—After steeping cellulose in glacial acetic acid for several hours in order to secure uniform absorption, the mixture is cooled until the acid crystallises, thereby causing some mechanical disruption of the cellulose, which thus becomes more receptive to the action of the acetylating mixture. F. R. ENNOS.

Acetylation of cellulose material. S. A. OGDEN (B.P. 310,563, 28.1.28).—Cellulose material, prepared according to B.P. 246,746 (B., 1926, 975), is treated with acetic acid and dried by heat, the partially acetylated product being then further acetylated with anhydride in the usual way. F. R. ENNOS.

Esterification of mercerised cellulose with lower aliphatic acids. H. T. CLARKE and C. J. MALM, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,687,059, 9.10.28. Appl., 23.4.27).—Mercerised cellulose, after washing free from alkali and drying at 30—40°, is heated with an aliphatic acid, C_2 — C_7 , for about 48 hrs. at 100—170° until the product contains at least 8% of acyl group. With heptonic acid the temperature of acylation should be 145—165°. The operation may also be effected under pressure, and acetylation at 140—160° is complete in 10 hrs. Acylation may also be effected with the acid vapours under normal or reduced pressure. During the acylation water is removed by use of a fractionating column.

R. BRIGHTMAN.

Pulping of wood. F. G. RAWLING (U.S.P. 1,705,827, 19.3.29. Appl., 17.9.27).—Wood is digested under pressure at 190° with a solution containing sodium sulphide, sodium hydrogen carbonate, and carbon dioxide. A. R. POWELL.

Manufacture of artificial threads, bands, films, etc. from solutions of cellulose esters and ethers. RUTH-ALDO Co., Inc., Assees. of H. L. BARTHELEMY (B.P. 308,271—3, 28.12.27. Fr., 28.12.26).—A cellulose ester or ether is precipitated in a suitable form from its solution in a volatile solvent by means of (A) a hydrocarbon of b.p. above 80°, e.g., toluene, or an alicyclic hydrocarbon, e.g., cyclohexane, (B) a higher fatty acid ester of a monohydric alcohol, e.g., ethyl stearate, (C) a halogenated hydrocarbon, e.g., trichloroethylene, an aromatic nitrile, e.g., benzonitrile, or a nitrated aromatic hydrocarbon, e.g., nitrobenzene.

F. R. ENNOS.

Manufacture of filaments or threads of cellulose derivatives. H. DREYFUS (U.S.P. 1,688,532, 23.10.28. Appl., 16.9.26. U.K., 29.3.26).—Addition of 0.5—15% (preferably 0.5—5%) of a high-boiling solvent or plasticiser, e.g., diacetin, triacetin, toluene- or xylene-sulphon-methyl- or -ethyl-amide, to the ripened cellulose ester solution, especially those with viscosities above 50°, gives on dry-spinning threads of considerably enhanced strength. The plasticiser may be removed from the resulting filaments by washing or extraction with liquids non-solvent for the cellulose derivative.

R. BRIGHTMAN.

Spinning of artificial silk. I. G. FARBENIND. A.-G. (B.P. 279,888, 28.10.27. Ger., 30.10.26).—Viscose is spun into fine-denier threads of high extensibility and uniform dyeing capacity by projecting it vertically upwards from nozzles through a short length of the precipitating liquid, without the use of a second bath, in such a manner that the threads are not subjected to stretching or bending at an angle smaller than 150° during their passage to the bobbins. F. R. ENNOS.

Manufacture of artificial threads, filaments, etc. COURTAULDS, LTD., and E. A. MORTON (B.P. 310,172, 10.4.28).—The cake of fibre is washed while still in the rapidly rotating centrifugal box by allowing a jet of water to impinge vertically on to the middle of a circular disc placed at the centre of the bottom of the box. The centre of this disc is domed whilst the outer part has alternate ridges and hollows, so that the water

jet is broken up into a spray which reaches all parts of the cake. F. R. ENNOS.

Manufacture of hollow artificial threads. ERSTE BÖHMISCHE KUNSTSEIDEFABR. A.-G. (B.P. 282,687, 20.12.27. Czechoslov., 21.12.26).—An insoluble liquid or solid substance, *e.g.*, petroleum, which does not yield any bubble-forming vapours, is mixed with the solution to be spun, and is subsequently removed from the textile threads, after finishing, by means of a vacuum at ordinary or increased temperature. F. R. ENNOS.

Paper-sizing agents. E. C. R. MARKS. From ALOX CHEM. CORP. (B.P. 309,383, 6.10.27).—A petroleum hydrocarbon oil, mixed with 0.1% of an oxidising catalyst, is subjected to a fine spray of air and heated at 120–155° under pressure until not more than 10% of heavy organic acids insoluble in the hydrocarbon mixture are formed. After separation these acids are purified by treatment with steam above 150°, separated from sludge, and converted by treatment with alkali into neutral soaps, which are mixed with paper pulp and then converted by means of alum into insoluble aluminium soaps. F. R. ENNOS.

Apparatus for treatment of webs or sheets of paper or textile or other material with chemicals or colouring matters or other fluids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,135, 27.2., 23.5., and 7.8.28).—By passing the material over orifices in the cover of one or more boxes, the appropriate liquid or gaseous staining or colouring materials contained therein are drawn up by suction on to the sheet. Various effects may be produced by giving the sheets a varying absorption capacity by application of pressure, by treatment with suitable reagents, or by imparting a reciprocating movement to the boxes. F. R. ENNOS.

Treatment of cellulosic material. Manufacture of cellulose derivatives. H. DREYFUS (U.S.P. 1,711,110—1, 30.4.29. Appl., 31.10.24. U.K., [A] 25.9.24, [B] 6.10.25).—See B.P. 249,173 and 263,939; B., 1926, 436; 1927, 247.

Manufacture of [compound] textile threads. O. RASCH (B.P. 311,447, 11.2.28).

Spinning bowls or boxes for artificial silk. J. C. HENSING, Assec. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 293,871, 13.7.28. Holl., 15.7.27).

[Guider-starter for] spinning of artificial silk and the like. W. P. DREAPER (B.P. 311,454, 13.2.28).

Centrifugal [laundry] washing and drying machines. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE GES. M.B.H. (B.P. 292,512, 19.6.28. Ger., 20.6.27).

Solvents for cellulose esters (U.S.P. 1,689,761—2).—See III. Rubber-covered metal sheets (B.P. 301,300).—See X. Plates for electric condensers (B.P. 310,625).—See XI. Wool-washing water (B.P. 300,386).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Stability to light of records made with type-writer ribbon, duplicate paper, and copying ink pencils. H. I. WATERMAN, J. GROOT and M. J. VAN

TUSSENBROEK (Chem. Weekblad, 1929, 26, 276—277).—Whilst impressions made with ribbon and papers in which carbon-black has been used do not change on keeping except for the fading of the dye, papers and pencils containing only methyl-violet do not give permanent impressions, since the dye fades completely on exposure to light. S. I. LEVY.

PATENTS.

Dyeing composition. A. DAVID, Assr. to DIP-IT, INC. (U.S.P. 1,687,751, 16.10.28. Appl., 25.3.22).—1% of a pure soap is incorporated with 59–66% of water, about 30% of glycerol, and 3–10% of a suitable dye, preferably a water-soluble acid, direct, or basic dye. R. BRIGHTMAN.

Mineral dyeing. C. B. WHITE, Assr. to VIVATEX PROCESSES, INC. (U.S.P. 1,686,540, 9.10.28. Appl., 23.12.26).—1–5% of formic or other organic acid is added to a solution of ferric sulphate and the sulphate ions are removed by means of barium acetate; textile material is passed through the bath, dried, and treated with an alkaline solution in the usual manner. The increased solubility of the ferric acetate in presence of formic acid leads to deeper, faster, and more uniform shades. R. BRIGHTMAN.

Dyeing and printing. M. BATTEGAY, Assr. to CALCO CHEM. CO. (U.S.P. 1,686,224, 2.10.28. Appl., 1.11.26).—With acid dyes and their salts, the mordants obtained from sulphur and phenols (*cf.* U.S.P. 1,523,340; B., 1925, 204) can be applied simultaneously with the dyestuff. After the dyeing, padding, or printing operation, the material is passed into a fixing bath containing dilute organic or mineral acid or mineral acid salt such as aluminium sulphate, zinc sulphate, barium chloride, etc. R. BRIGHTMAN.

Differential coloured fabric and its manufacture. C. DREYFUS and H. PLATT, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,689,426, 30.10.28. Appl., 13.8.27).—Fabric, *e.g.*, cellulose acetate or other organic derivative of cellulose, is printed with a solution of viscose or other material of different behaviour towards dyestuffs, and after regenerating the cellulose the mixed fabric is dyed in a bath containing, *e.g.*, a cellulose acetate dye which does not affect cellulose, and a cellulose dye which does not affect cellulose acetate. R. BRIGHTMAN.

Treatment of artificial silk and articles made thereof for rendering same water-repellent. J. P. BEMBERG A.-G. (B.P. 286,257, 1.3.28. Ger., 1.3.27).—Artificial silk materials are impregnated with a solution of a basic aluminium soap (containing not more than twice the equivalent weight of fatty acid to three times the equivalent weight of aluminium) in an organic solvent, the latter being then removed by evaporation; other fats and waxes, *e.g.*, paraffin wax, may be added to the solution. *E.g.*, into an 8% solution of a soap containing 80% of fat is poured an excess of a concentrated solution of a basic aluminium sulphate having 1.2 hydroxyl groups to 3 atomic equivalents of aluminium; 2 kg. of the precipitated basic aluminium soap (9.6% Al_2O_3) are then dissolved in 100 kg. of trichloroethylene. A. J. HALL.

Treatment [sizing] of artificial silk yarn. O. PLOETNER (B.P. 311,137, 29.6.28).—Artificial silk yarn

is rendered smooth and pliable by sizing it with a mucilage containing 7—8 pts. of soft soap, 1—2 pts. of glycerin, and 1 pt. of potato meal or other colloidal substance such as egg albumin, gum tragacanth, or agar-agar, this process being effected by passing the yarn through the mucilage, removing the excess, and smoothing the yarn by passage between pieces of absorbent rubber sponge pressed together.

A. J. HALL.

Mothproofing of fabrics. H. I. JONES (U.S.P. 1,688,717, 23.10.28. Appl., 24.1.27).—Fabric is treated first with an aqueous solution of casein and borax and then with an aqueous solution of salt of copper, lead, mercury, bismuth, thallium, or a rare-earth metal. Greater waterproofing effect is obtained by adding to the casein solution an emulsion of an easily hydrolysed wax, e.g., beeswax, in excess of ammonia solution.

R. BRIGHTMAN.

Manufacture of felt and felt materials. H. STEIN, W. E. AUSTIN, and I. LIEBOWITZ, Assrs. to STEIN FOR DYEING CO., INC. (U.S.P. 1,687,801—2, 16.10.28. Appl., 10.2.25).—Fur skins, hair, or other animal fibre, preferably without previous alkaline washing, are partly carotred using 50—75% of the amount of acid mercuric nitrate solution normally employed for complete carotring. After rinsing and hydro-extraction the fibres are immersed for 8—12 hrs. in a solution of protective agent, e.g., a 0.5—5% solution of ferrous sulphate crystals, to which a stabilising agent such as ammonium chloride may be added. After further rinsing and hydro-extraction the material is bleached by treating at 15—40° with 3% hydrogen peroxide.

R. BRIGHTMAN.

Imparting a wool finish to cotton cloth. M. J. MACK (U.S.P. 1,689,208, 30.10.28. Appl., 28.7.27).—Cotton fabric is successively bleached, dyed, napped, mercerised, sized, and re-brushed.

R. BRIGHTMAN.

Dyeing of artificial silk. W. HENTRICH, R. KNOCH, and M. HARDTMANN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,711,890, 7.5.29. Appl., 10.9.27. Ger., 22.9.26).—See B.P. 301,564; B., 1929, 127.

Machines for treating fabrics by processes such as washing, dry-cleaning, etc. BRIT. AMER. LAUNDRY MACHINERY CO., LTD., Assees. of G. H. WEST (B.P. 309,980, 29.6.28. U.S., 19.4.28).

[Steaming machine for] finishing of textile fabrics. G. C. HIRST (B.P. 311,355, 6.2.28).

Treatment of paper, textiles, etc. (B.P. 310,135).—See V. Stable sulpho-acids and salts (B.P. 288,126).—See XII. Bleaching of foodstuffs (U.S.P. 1,687,803—5).—See XIX. Multicolour screens (B.P. 309,113).—See XXI.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of phosphoric acid in mineral phosphates and fertilisers. G. JORGENSEN (Ann. Falsif., 1929, 22, 199—207).—In the case of mineral phosphates a sample (5 g.) is boiled with 25 c.c. of nitric acid (*d* 1.21) and 12.5 c.c. of hydrochloric acid

(*d* 1.12) for 30 min. The solution, after cooling, is made up to 250 c.c., shaken, and filtered, the first part of the filtrate being rejected. To 50 c.c. of the solution a quantity of molybdic acid is added calculated from the given table according to the proportion of phosphate present, and the mixture is kept at 50° for 10 min. and shaken. After being kept overnight the flask is cooled, the contents are filtered, and the precipitate is washed five times with 20—25 c.c. of a 4% ammonium nitrate solution. If precipitation is thought to be incomplete, the filtrate is again heated with molybdic acid. The solution is warmed and two thirds of a standard magnesium solution (amount found from the table) are added. The flask is shaken until the precipitate becomes crystalline, the rest of the magnesium solution is added, and the liquid cooled with frequent shaking. After 15 min. the precipitate is collected, washed with ammonia solution, dried, heated, and weighed as $Mg_2P_2O_7$. For superphosphates and fertilisers the total phosphoric acid is determined on 5 g. of the sample; these are heated with 20 c.c. of nitric acid only, the procedure being as above except that different quantities of reagents are added, as found from the tables. The phosphoric acid soluble in water is next determined, and then that soluble in water and in ammonium citrate, this last determination being made on a 2.5 g. sample, which is ground first dry and then with 20—25 c.c. of water, and the decanted liquid from three grindings is filtered. The residue is washed, the filtrate made up to 200 c.c., a few drops of nitric acid are added, and the whole is shaken (solution A). To the residue with the filter paper are added 100 c.c. of Petermann's ammoniacal ammonium citrate solution, the whole is shaken, and, after 15 hrs., heated to 40°, cooled, made up to 250 c.c., shaken, and again filtered (solution B). Then 50 c.c. each of A and B are mixed, and phosphoric acid is determined as for mineral phosphates. A modified procedure is given for Thomas slag. D. G. HEWER.

Manufacture of potassium chloride from Solikamsk sylvite. II. F. F. VOLF and V. S. YATLOV (J. Chem. Ind. Moscow, 1928, 5, 909—915; cf. B., 1929, 127).—The composition of the solutions of the sylvite, and of the precipitated crystals, is discussed. The conditions of crystallisation prevent the separation of calcium sulphate with the potassium chloride.

CHEMICAL ABSTRACTS.

[Manufacture of] bleaching powder without cooling. E. HADAMOVSKY (Chem.-Ztg., 1929, 53, 321—322).—The tower-chamber system, in which lime is rabbled over successively lower floors by means of arms on a central vertical shaft, as in the ordinary eight-hearth furnace for burning fine pyrites, the chlorine gases entering at the bottom and passing upwards, is found to operate as well without cooling as with cooling.

S. I. LEVY.

Examination of alkali iodides. W. MEYER (Pharm. Ztg., 1929, 74, 628).—Samples of ammonium, sodium, and potassium iodides, which on keeping darkened and separated free iodine, were found to contain thiosulphate. The Pharmacopœia test with silver nitrate and ammonia does not show the presence of small quantities of thiosulphate; these may be detected by

decolorisation of an acidified starch solution coloured blue with iodine. S. I. LEVY.

Manufacture of aluminium sulphate from clays.

I. E. ADADUROV (J. Chem. Ind. Moscow, 1928, 5, 941—942).—Clays containing alumina as kaolinite are calcined preferably at 800° before attack by sulphuric acid (20%), whilst clays containing 34% of kaolinite-alumina are preferably calcined at 700° and treated with 50% acid. Iron is best eliminated by chlorination of the crude clay, mixed with charcoal, at 300—400° or, better, of dehydrated aluminium sulphate at 400°.

CHEMICAL ABSTRACTS.

PATENTS.

Nitre oven. E. B. RUPARD, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,709,880, 23.4.29. Appl., 2.6.27).—The oven, for use in the manufacture of sulphuric acid, is connected by a conduit with the combustion flue and in its lower part has a refractory vessel, to contain sodium nitrate and sulphuric acid, fitted with a safety overflow through the oven wall below the conduit level. A valve normally closes the outer end of the overflow, but opens outwards automatically when necessary. W. G. CAREY.

Manufacture of boric acid. AMER. POTASH & CHEM. CORP., Assees. of W. E. BURKE and H. DE ROPP (B.P. 294,236, 7.6.28. U.S., 22.7.27).—A borax solution is successively treated with sulphuric acid, cooled to a temperature just above that at which sodium sulphate starts to crystallise, and filtered to remove the boric acid which has crystallised out; the mother-liquor, after dilution and dissolution in it of more borax, is successively cooled to such a temperature that sodium sulphate decahydrate crystallises out, filtered, and returned to the process. The conditions are so adjusted that the quantity of sodium sulphate crystallising out is approximately equal to that formed during the initial reaction, and the water formed by the reaction and that added to dilute the mother-liquor are removed as water of crystallisation. L. A. COLES.

Manufacture of silica gel. M. YABLICK (U.S.P. 1,687,919, 16.10.28. Appl., 10.3.22).—A solution of sodium silicate or other soluble silicate is treated with an ammonium salt, preferably the carbonate, and the silicic acid allowed to gel in the alkaline liquor before washing and drying. R. BRIGHTMAN.

Synthetic production of ammonia. NITROGEN ENGINEERING CORP. (B.P. 287,072, 12.3.28. U.S., 12.3.27).—A portion of the ammonia formed by the combination of nitrogen and hydrogen in a single converter is recovered from the gaseous products immediately they leave the converter, and the remainder after the gases have been contaminated with impurities by passage through a pump and by the addition of fresh supplies of nitrogen and hydrogen. L. A. COLES.

Production of salts. A. E. MITCHELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,594, 16.2.28).—An acid is treated with a base or with a decomposable salt while allowing them to flow down a packed tower, using an excess of one constituent and subsequently neutralising with a volatile acid or alkali supplied to the base of

the reaction tower in a current of steam, or in a separate vessel or tower. *E.g.*, a suspension of chalk in water or in liquor from a previous operation flows down a tower, and excess of nitric acid is introduced at a lower level, the excess of acid subsequently being neutralised with gaseous ammonia. W. G. CAREY.

Production of clear brine or other liquors.

A. W. ALLEN (U.S.P. 1,687,703, 16.10.28. Appl., 2.9.27).—Turbid liquors, *e.g.*, potassium sodium chloride liquors, sodium nitrate liquors in the Shanks process, are clarified by the addition or the production, by evaporation with rapid agitation and cooling, of sufficient small crystals of a constituent, or other water-soluble compound, to act as a mechanical filter-aid, when the crystals and suspended matter are filtered off. R. BRIGHTMAN.

Manufacture of alkali nitrates.

J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 310,687, 31.5.28).—Alkali sulphates are treated with at least 2 mols. of nitric acid per mol. of sulphate, and the alkali nitrates are separated from the mother-liquor by cooling and centrifuging. L. A. COLES.

Production of pure ammonium sulphate.

R. E. SLADE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,635, 11.4.28).—Ammonium sulphate practically free from iron is obtained from the interaction of ammonium carbonate and calcium sulphate by returning a portion of the evaporated liquor, preferably from the crystal separators, to the reaction stage and removing the precipitated iron and chalk together, or to a subsequent stage where the liquor is still alkaline and removing the precipitated iron by settling. The evaporators are made of, or lined with, nickel-chrome steel or similar alloy. W. G. CAREY.

Apparatus for the production of ammonium sulphate.

BAYERISCHE STICKSTOFF-WERKE A.-G. (G.P. 453,941, 6.6.26. Addn. to G.P. 441,260; B., 1927, 965).—The circulating liquid is raised by compressed air from the equalising vessel to which the fresh acid is added as described in the prior patent and, after separation from vapour, is returned to the upper part of the saturation vessel where it serves to absorb liberated ammonia. L. A. COLES.

[Manufacture of] ammonium alum.

C. McC. BROWN (U.S.P. 1,709,166, 16.4.29. Appl., 14.10.25).—Aluminous material containing iron is heated at a temperature above the decomposition point of ferric sulphate, ammonium hydrogen sulphate sufficient to dissolve the alumina is added, and heating is continued at the same temperature until the reaction is complete. A. R. POWELL.

Production of ammonium salts.

R. TERN (B.P. 310,673, 16.5.28).—Volatile ammonium compounds are produced from salts capable of reacting in a dry condition, by mixing such salts, reducing the mixture to a colloidal state, heating in a revolving inclined drum to which air is supplied, withdrawing the product as vapour, and treating in an "electro-filter". W. G. CAREY.

Production of barium hydroxide.

C. DEGUIDE (Swiss P. 122,352, 8.10.26).—Basic barium silicate

($2\text{BaO}, \text{SiO}_2$) is treated with hot water whereby barium hydroxide dissolves, leaving an insoluble residue of the normal silicate which, after removal, is converted into the basic silicate by treatment with barium carbonate.

L. A. COLES.

Recovery of oxalates. G. H. BUCHANAN and G. BARSKY, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,687,480, 9.10.28. Appl., 4.9.24).—Low-grade calcium oxalate cake, containing, e.g., 20% of calcium oxalate and 80% of calcium carbonate, is made into a slurry and heated at 100° with an excess (10–35%) of sodium carbonate solution, sufficient to prevent reversion on cooling when the insoluble calcium salts are filtered off. The oxalate solution is treated with milk of lime and filtered, the precipitate (about 80% of calcium oxalate and 20% of calcium carbonate) being decomposed with sulphuric acid in the usual manner and the filtrate converted into (sodium) carbonate and used again. If the crude oxalate cake contains lime, carbon dioxide may be passed into the mixture during the treatment with sodium carbonate until the hydroxide has been converted into carbonate.

R. BRIGHTMAN.

Treatment of [ferrous] chloride solutions. S. I. LEVY and G. W. GRAY (B.P. 309,268, 10.2.28).—The ferrous chloride solutions obtained by dissolution of the ferrous sulphide produced by heating pyrites out of contact with the air are saturated with hydrogen chloride at 100° and utilised in dissolving fresh quantities of ferrous sulphide until they are 90% saturated with ferrous chloride at 100° . On filtering and cooling to 20° , about half the ferrous chloride separates as $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$. The mother-liquor is saturated with hydrogen chloride and returned to the cycle. When the zinc accumulated in the liquor reaches 80–100 g./litre, the solution is removed from the cycle and evaporated to recover crude ferrous chloride crystals, and a final mother-liquor from which the zinc is readily recovered; alternatively, most of the ferrous chloride is precipitated by saturation of the solution at 20° with hydrogen chloride.

A. R. POWELL.

Production of ferrous bicarbonate. C. PEETERS (Swiss P. 122,987, 28.2.27).—The compound is precipitated by mixing aqueous solutions saturated with carbon dioxide containing, respectively, a ferrous salt and an equimolecular proportion of an alkali bicarbonate, together with 1–5% of an alkali tartrate, lactate, citrate, etc.

L. A. COLES.

Production of alumina from clay and other aluminiferous material. M. BUCHNER (B.P. 309,621, 13.1.28).—The clay or aluminiferous material containing but small quantities of alkali compounds is digested with nitric acid, and, after filtration, the solution is treated with nitric acid or oxides of nitrogen to precipitate crystalline aluminium nitrate. The product is converted into alumina with simultaneous recovery of oxides of nitrogen by heating it alone or mixed with alumina, or by adding it to molten alkali nitrates or calcium nitrate, the decomposition being aided by working under reduced pressure and by passing steam, hot gases, etc. through the material.

L. A. COLES.

Production of magnesium salts or magnesia from waste material containing magnesium.

E. SEIDLER (Austr. P. 108,413, 28.8.26).—The material is dissolved in concentrated hydrochloric acid and impurities such as silica, iron, lime, alumina, etc. are precipitated by the addition of basic material containing magnesium, e.g., filter residues or calcined magnesite waste which has been rendered readily soluble by exposure to the air.

L. A. COLES.

Production of hypochlorites. G. ORNSTEIN (G.P. 454,406, 2.2.23. Addn. to G.P. 406,452; B., 1925, 169).—An aqueous suspension containing material capable of forming hypochlorites and free hypochlorous acid circulates continuously through a circuit comprising a container and absorption apparatus; in the latter the suspension is treated with liquid or gaseous chlorine in quantity sufficient to form some free hypochlorous acid, which in the container is converted into hypochlorites. The hypochlorite solution after clarification is withdrawn continuously from the container and replaced by equivalent quantities of water and the reacting substances.

L. A. COLES.

Evaporation of cyanide solutions. STOCKHOLMS SUPERFOSFAT FABR. AKTIEBOLAG, Assees. of H. G. A. RAMSAY (Swed. P. 59,652, 23.2.24).—The solutions are evaporated to dryness by spraying *in vacuo* at a temperature below that at which the cyanides hydrolyse.

L. A. COLES.

Process of alkylation. [Lead tetraethyl.] C. A. KRAUS and C. C. CALLIS, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,690,075, 30.10.28. Appl., 5.4.23).—Magnesium and an alkyl chloride are heated in ether with the substance to be alkylated, e.g., plumbous chloride, to above the normal b.p. of the alkyl chloride.

R. BRIGHTMAN.

Obtaining pure oxygen from a single or double oxygen plant, with a greater output. W. F. DALTON (B.P. 309,632, 14.1.28).—The liquid oxygen is conveyed by siphon tubes to an inner column contained in the evaporating coil of the collecting vessel, the liquid being evaporated during its passage through the tubes and the pure gas being withdrawn from the inner column. Alternatively, the siphon tubes may be closed and the liquid oxygen conveyed to the inner column for rectification, when necessary.

L. A. COLES.

Liquefaction of oxides of nitrogen. J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 310,212, 2.12.27).—Gases containing oxides of nitrogen are cooled and compressed in the presence of, e.g., 2–4% of anhydrous nitric acid, which may be introduced as a spray or may be formed *in situ* by the addition of suitable quantities of water and oxygen to the gases.

L. A. COLES.

Oxidation of phosphorus vapour. A./S. MALMIN-DUSTRI (Norw. P. 42,753, 20.12.24).—Roasting gases or finely-divided metal oxides are used for the oxidation.

L. A. COLES.

Ammonia oxidation and catalyst therefor. E. I. DU PONT DE NEMOURS & Co. (B.P. 306,382, 25.6.28. U.S., 18.2.28).—See U.S.P. 1,706,055; B., 1929, 432.

Manufacture of hydrocyanic acid. R. FICK, Assr. to I. G. FARBERIND. A.-G. (U.S.P. 1,712,297, 7.5.29. Appl., 21.12.25. Ger., 19.12.24).—See B.P. 261,559; B., 1927, 74.

Ferric chloride (B.P. 284,991 and 288,319).—See X. Titanium compounds (B.P. 309,051).—See XIII. Halogens from sea-water (B.P. 294,655).—See XXIII.

VIII.—GLASS; CERAMICS.

Kilns and kiln firing. II. The Dunnachie continuous gas-fired kiln. III. The Belgian ring kiln. S. R. HIND (Trans. Ceram. Soc., 1929, 28, 148—164, 204—219).—II. The kiln has two parallel but unequal sets of chambers, eight on one side and ten on the other. The main producer-gas flue runs between the two sets of chambers, the exhaust flues being on the outer sides. The method of firing is that common to all chamber kilns, but two chambers are fired simultaneously in different parts of the round. The method of testing the behaviour of the kiln is described; 14.9 cwt. of fuel are consumed per 1000 bricks fired to cone 15. A heat balance is presented, and methods of improving the efficiency of the plant are indicated.

III. The kiln consists of two long parallel galleries, 12 ft. wide and 8 ft. 6 in. high internally, which communicate at the ends to form a continuous passage. Room is left between the galleries to form a smoke chamber, which leads to an outside stack. The method of operating the kiln and of setting the bricks is described. The results of an exhaustive test show that 1.83 cwt. of fuel are consumed per ton of fired ware. A summarised heat-balance is given, together with full data on the fuel, draught and temperatures, kiln atmospheres, exhaust gases, rates of firing, etc. F. SALT.

Use of pulverised coal as a fuel for periodic kilns. D. J. WATSON (J. Amer. Ceram. Soc., 1929, 12, 336—341).—Attempts to use pulverised fuel in periodic kilns have been unsuccessful because sufficient heat for combustion must be maintained in the fire-box. This too soon raises the whole kiln above the water-smoking temperature. A fire-box has been constructed the size of which can be varied by the movement of two baffle plates. In this way the small hot spot necessary for combustion may be maintained at low kiln temperatures.

J. A. SUGDEN.

Glass-tank refractories and their chemical relationship with the molten glass. K. C. LU (Ohio State Univ. Eng. Exp. Sta. Bull., 1928, No. 44, 40 pp.).—The life of a refractory depends on the rate of formation and dissolution of a porcelain-like surface at the glass interface. Digestion of this layer with hydrofluoric acid yielded mullite. The rate of dissolution was less in an aluminous than in a highly siliceous refractory. The brown, glassy layer covering the white layer contained labradorite crystals and a little mullite. In a highly siliceous refractory the brown glass was replaced by a white layer containing cristobalite.

CHEMICAL ABSTRACTS.

Application of colloid chemistry to the study of clays. II. A. E. J. VICKERS (Trans. Ceram. Soc., 1929, 28, 124—147; cf. B., 1929, 394).—A comprehensive review is presented of the literature dealing with clays from the colloid aspect. The relation between the general properties of clays and their origin is discussed, and the present position regarding the nature of clay substance is summarised. The colloid chemistry of

clays is outlined in some detail and its practical applications are indicated. F. SALT.

Obtaining raw clays which are not washed away by water. P. P. BUDNIKOV (Trans. State Exp. Inst. Silicates, Moscow, 1927, No. 21, 97—106).—The times required for clay, mixed with reagents and then air-dried, to disintegrate when immersed in water are tabulated. Calcium hydroxide (more than 5%) markedly increased the stability of the clay. Of the salts and acids, phosphoric acid (3—5%) was most effective. Addition of phosphoric or sulphuric acid decreases the burning shrinkage. Clays containing 5.5—42.5% SiO_2 , when treated with sulphuric acid or a mixture of this with calcium phosphate, manifested stability up to 21.7% SiO_2 . CHEMICAL ABSTRACTS.

Action of sulphuric acid on clay at the ordinary temperature. P. N. GRIGORIEV and P. I. GALKIN (Trans. State Exp. Inst. Silicates, Moscow, 1927, No. 21, 91—95).—The production of "waterproof" clay ("keramolite") by the action of sulphuric acid (cf. preceding abstract) is attributed to dehydration. The presence of impurities, especially iron, appears favourable. CHEMICAL ABSTRACTS.

Rate of drying of a plastic porcelain mass due to reduced pressure and heat. L. NAVIAS (J. Amer. Ceram. Soc., 1929, 12, 305—318).—A body consisting of china clay 20%, ball clay 25%, flint 25%, and felspar 30% was subjected to pressures of 0.7, 5, 15, and 35 cm. and temperatures of 23°, 50°, and 65°. The relationship between the logarithms of the amount of evaporation and of the time was found to be linear. At the lowest pressure the relationship has a single slope, but at the higher pressures two distinct slopes indicate two stages in the drying. The connexion between rate of drying and lowering of temperature of the drying mass is indicated, and the effect of the humidity of the air and the shape of the specimen is discussed.

J. A. SUGDEN.

Functions of regenerators in relation to the refractory materials of construction. A. T. GREEN (Trans. Ceram. Soc., 1929, 28, 165—191).—The principles of regeneration, with particular reference to the open-hearth furnace, are explained, and the theory of regenerator action is discussed. Essential properties of refractories for use in regenerators are: suitable thermal characteristics to ensure maximum absorption and transmission of heat; resistance to the action of slags and dust; and mechanical strength and resistance to spalling. Data are presented on the heat capacities and other relevant properties of firebrick, silica, magnesite, zirconia, and carborundum refractories. For economic reasons, the choice of regenerator fillings is restricted to silica, siliceous, and fireclay bricks. The available data indicate that silica is less efficient from the point of view of operation and thermal capacity than are firebrick and siliceous products. To guard against warping and deformation of the bricks, the material should be tested for refractoriness under a load of 20—25 lb./in.² at about 1200°, and for after-contraction, which should not exceed 0.5—0.7%. F. SALT.

Drying cracks in firebricks. C. E. MOORE (Trans. Ceram. Soc., 1929, 28, 193—200).—Drying cracks in

firebricks are classified as due to faulty drying treatment and to methods of preparation of the material, *i.e.*, mixing, extruding, moulding, and pressing. A "danger zone" is reached in drying when movement of moisture through the mass is rapidly becoming more difficult, when the rate of contraction with loss of water is decreasing, and when the strength is slowly increasing. The effects of additions of grog are described. The various causes of drying cracks are enumerated, and the advantages of humidity drying over the hot-floor method are pointed out.

F. SALT.

Sand-blast abrasion test for glazes. E. SCHRAMM (J. Amer. Ceram. Soc., 1929, 12, 356—359).—The essentials of a good test are (1) the use of a stream of sand which may easily be reproduced, (2) the rotation of the test plate so that a large area can be covered and a weighable amount removed without cutting deeply into the glaze. A suitable apparatus for the purpose is described in which a $\frac{1}{8}$ -in. jet is inclined at an angle of 60° to the plane of the test plate. Tests showed that the weight of sand delivered and extent of abrasion are independent of the humidity of the atmosphere (*cf.* Harrison, B., 1927, 749). The comparative hardness of a number of typical glazes is given; it is found that glaze fire within normal maturing range has little effect on hardness. Glaze composition is the chief factor governing hardness.

J. A. SUGDEN.

Prediction of service value [of ceramic materials] from laboratory test data. A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1929, 12, 319—329).—A statistical treatment shows that such prediction depends not only on the applicability of the particular test but also on certain precision conditions which become very important in work with materials such as refractories the properties of which are variable. Charts have been constructed which show the effect of errors of measurement on accuracy of prediction for cases arising in ceramic work.

J. A. SUGDEN.

Monolithic cements. CLARK.—See IX.

PATENTS.

Tunnel kilns. W. L. HANLEY, JUN. (U.S.P. 1,705,475 and 1,705,477, 19.3.29. Appl., [A, B] 30.8.26. Renewed [A, B] 8.8.28).—Tunnel kilns are provided with central firing zones into which the products of combustion enter, and with preheating and cooling zones in opposite directions therefrom. A single line of goods passes through the three zones in succession. In (A), the products of combustion are removed from the firing zone without permitting much longitudinal flow and are conducted by outside passages into the preheating zone. In (B), air is admitted to the cooling zone, heated by contact with the goods therein, withdrawn, and conducted outside the kiln to the preheating zone.

B. M. VENABLES.

Pottery kiln. A. C. WARD (U.S.P. 1,710,558, 23.4.29. Appl., 20.12.27).—A special sub-floor flue system is described.

J. A. SUGDEN.

Laboratory muffle [for metal recovery]. E. B. FORCE, ASSR. to CARBORUNDUM CO. (U.S.P. 1,710,870, 30.4.29. Appl., 2.9.27).—The walls of a heating chamber comprise at least two sections, that more directly

exposed to the action of oxidising or easily reducible materials in the chamber being made of a refractory metallic oxide material, whilst that through which heat is principally transmitted to the chamber is made of silicon carbide.

J. S. G. THOMAS.

Production of opacifying media from zirconium silicate. DEUTS. GASLÜHLICHT-AUER GES. M. B. H. (B.P. 306,019, 9.7.28. Ger., 14.2.28).—Zirconium silicate is fused with about an equimolecular quantity of oxide of calcium, strontium, barium, magnesium, or zinc in presence of a small quantity of a flux.

W. G. CAREY.

Apparatus for drawing sheet glass. P. HADDAN. From VIRGINIA PLATE GLASS CORP. (B.P. 311,171, 24.9.28).

Manufacture of non-splintering glass sheets. G. E. HEYL (B.P. 310,995, and Addn. B.P. 311,098 and 311,151, [A] 4. and 17.2.28, [B] 10.5.28, [C] 8.8.28).

Manufacture of compound sheets of glass and cellulose derivative compositions. G. E. HEYL (B.P. 311,128, 15.6.28. Addn. to B.P. 310,995).

Protecting wood or metal (B.P. 310,554).—See IX.
Furnace for glass (Swiss P. 122,176). **Marking of lamps** (Dutch P. 17,399).—See XI.

IX.—BUILDING MATERIALS.

Factors governing the durability of clay building materials. I. W. A. MCINTYRE (Trans. Ceram. Soc., 1929, 28, 101—123).—The destructive agencies involved in the "weathering" of building materials are classified as biological, physical, and chemical. Bacterial action is not an important factor in this connexion. The various physical and chemical factors, such as moisture expansion, thermal expansion, erosion, rupture by frost and by crystallisation of salts, the presence of soluble salts, etc., are described, and an attempt is made to estimate their relative importance. Texture has an important bearing on the durability, but the crystallisation of soluble salts, mainly sulphates, is the chief disintegrating influence. The salts may be derived from the soil, the atmosphere, the mortar, or from the material itself, but the various actions governing the formation, distribution, and crystallisation of salts in brickwork are not yet elucidated. Frequent washing prevents or greatly reduces the action; it is noted that the effects of these salts are absent in roofing tiles, which are subjected to more constant washing by rain.

F. SALT.

Comparative tests on monolithic refractory cements. H. N. CLARK (J. Amer. Ceram. Soc., 1929, 12, 330—335).—Nine leading cements on the market (plastic and dry) were tested for composition, fineness, drying and firing shrinkages, dried and fired strengths, refractoriness, and fire behaviour. The "dry" cements had much lower shrinkages and higher strengths. Two cements had low refractoriness. A small cylindrical furnace 18 in. long, 6 in. internal diam., with 3 in. walls was rammed or poured from each of the cements. These small furnaces were used to test fire behaviour and spalling tendency by firing them to 1500° and rapidly reheating three times after cooling. As a result of

the small-scale tests the three best cements were chosen and submitted to a large-scale test on the walls of an oil-fired boiler.

J. A. SUGDEN.

Litharge-glycerin cements. H. STÄGER (Z. angew. Chem., 1929, 42, 370—379).—Seven different samples of lead oxide to be used for the cements were examined microscopically; samples 1—4 were prepared by precipitation and ignition under different conditions, sample 5 was commercial litharge, samples 6 and 7 were obtained by burning lead in vapour form. Samples 1—4 showed irregular particle size, doubtless due to agglomeration; sample 3 (red) appeared most uniform (cf. Glasstone, B., 1921, 846A), whilst samples 6 and 7 showed very fine regular structure. Sedimentation tests confirmed that the red oxide (3) had average particle size 2—6 μ , whilst the particle size in the yellow samples varied up to 30 μ . The oil numbers (c.c. of linseed oil required to make a uniform paste from 100 g. of oxide) and readiness of reaction to form soaps and glycerides were also recorded, and conclusions drawn as to the surface activity of the different samples. It was observed that the agglomerated particles of some of the yellow samples were readily broken down by rubbing. The highest surface activity was shown by the high-dispersion sample 7. The time of setting and the breaking strains of the cements were observed for mixtures of all the oxides with 85% glycerin in varying molecular proportions. The best results were obtained with different proportions for the varying samples, the ratio $\text{PbO} : \text{C}_3\text{H}_5(\text{OH})_3$ for suitable cements being 2:1 for samples 1—4, 3:1 for sample 5, and 1:1 for samples 6 and 7. Varying the dilution of the glycerin employed also gave results varying with the sample; for the less active (1, 2, 4, and 5), 97% glycerin gave as good cements as 85%, but with 75% they were definitely weaker, whilst the more active oxides (samples 3, 6, and 7) gave better cements with 75% and worse with 97% glycerin than with 85%. Greater dilutions gave worse results in every case. Atmospheric moisture has no appreciable effect on the setting of the cement. Tests on absorption of carbon dioxide by the cements showed that the finer oxides, with the higher oil numbers, give more open and porous cements, which are more readily acted on by the gas; this was confirmed by hardness tests carried out by the die-pressure method (cf. Meyer, Elektrotechn. Z., 1922, 42, 1285). The times of setting for maximum hardness and strength depend on the nature of the oxide and proportion of glycerin; where these are correctly chosen, maximum values were reached in every case in 20 hrs.

S. I. LEVY.

Relation between the temperature curve and the expansion curve in the setting of plaster. F. J. WILLIAMS and F. C. WESTENDICK (J. Amer. Ceram. Soc., 1929, 12, 377—381).—A form of volumeter was used to determine the time-temperature and time-expansion curves. The relationship of the two curves suggests that the calcium hemihydrate first goes into solution, causing the decrease in volume and evolution of heat which are observed. When the solution becomes saturated with respect to the hemihydrate the crystallisation of the dihydrate begins. This causes an expansion to occur, whilst the temperature reaches a maximum

owing to the adsorption of heat by the crystallisation of the dihydrate.

J. A. SUGDEN.

Refractory construction materials. GREEN.—See VIII.

PATENTS.

Kiln for calcining finely-divided material, e.g., lime. O. DEMISCH (G.P. 454,402, 13.3.25).—A powdered mixture of fuel and the material to be calcined is forced through a jet leading upwards into a kiln of approximately semi-ellipsoidal vertical cross-section. The mouth of the jet and the outlets for the waste gases lie on the horizontal, diametrical plane, and the receiver for the calcined material lies below this plane.

L. A. COLES.

Burning of argillaceous material. S. J. HAYDE, Assr. to AMER. AGGREGATE Co. (U.S.P. 1,707,395, 2.4.29. Appl., 10.2.20).—The material is suddenly subjected to a temperature above 820° to cause rapid expulsion of combined water and the production of a cellular aggregate, the burning of which is completed at a lower temperature.

A. R. POWELL.

Production from magnesia cement of articles resistant to chemical attack. S. J. BENNETTER (Norw. P. 42,716, 5.5.25).—Aqueous emulsions of cellulose derivatives are added to the material prior to moulding.

L. A. COLES.

Production of building stones. A. TETZNER (B.P. 292,925, 25.6.28. Ger., 24.6.27).—Clay and sand (6 pts. each) are ground to a meal-like mass, and cement (1 pt.) and water are added to form a moist mass.

H. ROYAL-DAWSON.

Production [by a cold process] of articles with a glaze-like covering. K. and C. FRIEDRICH (B.P. 309,755, 7.5.28).—Articles constructed of porous material, e.g., mixtures of cement with ground pumice-stone, slag, etc., are coated with a paste of finely ground cement containing small quantities of metallic (preferably zinc) soaps, and, while the coating is still plastic, a thin layer of cellulose varnish is applied.

L. A. COLES.

Protection of wood and metal from corrosive influences. H. KREILL (B.P. 310,554, 25.1.28).—The material is coated with glass, fused or sprayed, molten on to the surface by electrical means. Wood may be soaked in a mixture of water-glass and asbestos before treatment. [Stat. ref.]

L. A. COLES.

Manufacture of road surfaces. W. H. SCHMITZ (B.P. 301,891, 3.10.28. Ger., 8.12.27).—Mineral aggregates are coated uniformly with a mineral, vegetable, or animal oil, e.g., tar oil or turpentine oil, before admixture with bitumen emulsions.

L. A. COLES.

Manufacture of road-surfacing materials or compounds. J. Y. JOHNSON. From AMIESITE Co. OF AMERICA (B.P. 309,634, 14.10.27).—Stone aggregate is treated with a partly neutralised solution of oleic acid in hydrocarbon oils, and lime is added after admixture of the aggregate with bitumen to form calcium oleate in the product.

L. A. COLES.

[Concrete] mixing machines. KOEHRING Co., Assees. of G. E. WEBB (B.P. 307,869, 7.6.28. U.S., 15.3.28).

Manufacture [laying] of stone asphalt roadways. J. Y. JOHNSON. From AMIESITE ASPHALT CO. OF AMERICA (B.P. 310,294, 14.10.27).

Asphalt emulsions (B.P. 284,330).—See II. Rubber surfacing (U.S.P. 1,705,273).—See XIV.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Corrosion of iron. J. F. G. HICKS (J. Physical Chem., 1929, 33, 780—790).—The corrosion of iron has been investigated by measuring the increase in weight of a spiral of pure iron wire suspended in various atmospheres of dry or moist air, oxygen, carbon dioxide, oxygen-carbon dioxide mixtures, and of water vapour. It is concluded that the primary cause of corrosion is the actual dissolution of iron in water which takes place before any other chemical reaction sets in, and that a film of liquid water adhering to the metallic surface is essential for corrosion. The mechanism given is that iron dissolves as ferrous hydroxide until $p_H = 9.4$, the equilibria being $\text{Fe (solid)} = \text{Fe (dissolved)} + 2\text{HOH} = \text{Fe(OH)}_2 + \text{H}_2$. These are shifted to the right, and corrosion is maintained by oxygen and carbon dioxide in the following ways. Oxygen removes ferrous hydroxide by oxidation to the less soluble ferric hydroxide, and also hydrogen (depolarisation). Oxygen also produces the maximum *E.M.F.* with respect to dissolving iron, and hence acts as an accelerator; its oxidising action is a maintenance factor. Carbon dioxide removes ferrous hydroxide by conversion into the less soluble carbonate (maintenance factor), and forms carbonic acid, the hydrogen ions of which dissolve metallic iron and favour corrosion (accelerating effect).

L. S. THEOBALD.

Influence of copper on mild steel. S. KODAMA (J. Study Ferrous Met., 1928, 8, 1—23).—Experimental results accord with Ruer's equilibrium diagram, according to which 0.3% Cu can be completely dissolved in iron as a solid solution. The diffusibility of copper in solid iron is small at 1100°, but great at 1200°; copper tends to destroy the ferrite grain. In the surface oxidation of steel copper remains unoxidised and enters the boundary of ferrite at the surface, causing fissures in hot-rolling.

CHEMICAL ABSTRACTS.

Cause of the thermal brittleness of cupriferous steel. I. KOTAIRA (J. Study Ferrous Met., 1928, 8, 25—39).—Steels containing more than 1% Cu have the dendritic structure of a solid solution rich in copper; this cannot be removed by simple annealing. When cupriferous steel is heated at 1000° for 22 hrs. the surface oxide contains only 10—20% of the original copper, the copper content of the unoxidised steel increasing by 5—10%.

CHEMICAL ABSTRACTS.

Analysis of zinc dust. S. S. FROLOV and K. O. SVETLYAKOV (J. Chem. Ind. Moscow, 1928, 5, 942—943).—The zinc dust, moistened with alcohol, is warmed with a known quantity of copper sulphate in aqueous solution; after 30 min., a few drops of sulphuric or acetic acid are added, the liquid is filtered, the filtrate and washings are treated with ammonia, and the copper is determined colorimetrically. CHEMICAL ABSTRACTS.

Protection from corrosion of aluminium armatures by anodic oxidation. W. E. VON GRONOW (Gas- u. Wasserfach, 1929, 72, 429, 436).—When aluminium is coated with a hard film of oxide, it will often resist further attack of oxidising agents; this coating is best obtained by anodic oxidation of the perfectly clean metal, using a graphite cathode. The process is carried out at 40° in a 2% solution of chromic acid; the current necessary is 0.3—0.4 amp./dm.² of metal surface, and it is applied at 0—40 volts during the first 15 min., then at 40 volts for 35 min., and at 50 volts for 5 min. The precautions necessary to obtain completely satisfactory results are described, and the extent to which the treatment can be applied to alloys is discussed.

R. H. GRIFFITH.

Properties of carbonised tungsten. B. T. BARNES (J. Physical Chem., 1929, 33, 688—691).—The spectral emissivity for $\lambda = 0.660 \mu$ and $\lambda = 0.472 \mu$ and the total emissivity of tungsten carbonised to the carbide W_2C have been investigated. Photomicrographs of filaments carbonised at 1950—2150° Abs. showed the presence of only the carbides W_2C and WC . The m.p. of the carbide W_2C , or more probably the eutectic point for W_2C and tungsten, is estimated to be 3000° Abs. $\pm 15^\circ$.

L. S. THEOBALD.

Effect of certain cations on the flotation of galena. N. W. TAYLOR and H. B. BULL (J. Physical Chem., 1929, 33, 641—655).—The flotation recovery of galena by means of eucalyptus oil in the presence of the nitrates of thorium, cerium, chromium, aluminium, cadmium, lead, nickel, cobalt, barium, magnesium, and silver has been measured. In every case the cation has a toxic effect on flotation and reduces the yield, the reduction being greatest with chromium, thorium, aluminium, and cerium. The shape of the curve obtained by plotting the loss in yield against concentration of added electrolyte suggests that of an adsorption isotherm and leads to the conclusion that adsorption of the cation by the galena is responsible for the toxic effect. This appears to be due to the electrostatic repulsion of similarly charged mineral particles which, in turn, reduces the adherence to air bubbles necessary for flotation. (Cf. following abstract.)

L. S. THEOBALD.

Electrostatics of flotation. H. B. BULL (J. Physical Chem., 1929, 33, 656—658).—The *E.M.F.* produced by the fall of crystallised galena through solutions of thorium, chromium, silver, lead, and cobalt nitrates of various concentrations have been measured. The results indicate that the toxicity of lead, silver, and cobalt, but not of thorium and chromium, on the flotation of galena (cf. preceding abstract) and the electrostatic charge carried by galena in solutions of their nitrates are closely connected. The large effect of the silver ion compared with those of the lead and cobalt ions is attributed to greater adsorption.

L. S. THEOBALD.

Determination of sulphur in galena and metallic lead. H. LEYSART (Z. anal. Chem., 1929, 77, 209—213).—Sulphur is determined in lead by heating 5 g. of fine filings with 50 c.c. of hydrobromic acid (*d* 1.49) and a few drops of stannous chloride solution, and collecting

the hydrogen sulphide evolved in 50 c.c. of cadmium acetate solution (25 g. of the salt in 1 litre of 20% acetic acid). The precipitated cadmium sulphide is determined iodometrically. Sulphur may be determined in galena in a similar manner using 0.15–0.2 g. of the ore and 2 g. of pure zinc instead of stannous chloride.

A. R. POWELL.

Theory of chromium plating. E. A. OLLARD (Korrosion u. Metallschutz, 1928, 4, 208–209; Chem. Zentr., 1928, ii, 2507).—The electrolyte must contain chromic hydrogen chromate, chromic dichromate, sulphuric acid or a sulphate, and a colloidal compound of the type $\text{Cr}_2\text{O}_3 \cdot x\text{CrO}_3$. The chief cathodic processes are: $\text{Cr}^{+++} \rightarrow \text{Cr}$, $\text{H}^+ \rightarrow \text{H}$, and $\text{Cr}^{+++} \rightarrow \text{Cr}$.

A. A. ELDRIDGE.

Comminution of nickel catalyst. E. MASCH-KILLEISSON (Oil Fat Ind. [Russia], 1928, No. 1, 24–27; Chem. Zentr., 1928, ii, 2417).—After an optimal degree of fineness of division of a nickel catalyst has been attained, further dispersion is accompanied by reduction and, finally, cessation of activity. Nickel, which is thus removed from the catalytic surface, should therefore be deposited on the carrier after it has been reduced to the desired state of division.

A. A. ELDRIDGE.

Apparatus for reduction of nickel catalyst. E. WOLFSON (Oil Fat Ind. [Russia], 1928, No. 2, 8–11; Chem. Zentr., 1928, ii, 2417).

PATENTS.

Sintering apparatus. A. HOLMBERG (B.P. 310,089, 21.1.28).—Portable sintering containers are conveyed for discharge to a mechanical tipper which can be rotated on a horizontal axis.

C. A. KING.

Metallurgical furnaces. B. TALBOT (B.P. 293,010, 2.12.27. Belg., 30.6.27).—A tilting, reversible, regenerative furnace is provided with auxiliary baths at both ends of the main hearth which serve as reservoirs for molten metal or slag or may be used for melting material to be charged into the main hearth. The auxiliary hearths are movable longitudinally to the main hearth, joined thereto by means of unitary removable throats, and are provided with two gas ports oppositely directed across the bath, one of these ports having an air port associated with it.

A. R. POWELL.

Apparatus for subjecting a mass of powdered or granular material to the action of gases, particularly applicable to the calcining and roasting of ores and similar materials. H. M. RIDGE (B.P. 305,883, 1.5.28).—In a roaster the ore or material to be roasted lies upon a bed of inert granular material, which, in turn, lies on grate bars bridging gas-supply chambers. The granular material may be either of uniform size or in strata of several sizes, with the object of providing a good distribution of the reacting gas or air which passes upward through the ore. Rabblers, if used, extend into the ore but not into the inert material. The grate bars may be so formed that hood-like projections cover the spaces between the bars.

B. M. VENABLES.

Mechanical roasting furnaces. METALLGES. A.-G. (B.P. 297,053, 7.9.28. Ger., 13.9.27).—In mechanically-rabbed multiple-hearth furnaces in which the charge

travels alternately towards the centre and towards the periphery on succeeding hearths, the middle hearths are provided with further openings located between the centre and periphery at various distances apart so as to accelerate the passage of the charge through the furnace.

A. R. POWELL.

Mechanical [ore-roasting] kilns. ERZRÖSTUNG GES.M.B.H., and G. BALZ (B.P. 309,830, 14.4.28. Addn. to B.P. 288,823; B., 1928, 451).—The lowest roasting chamber of a kiln discharges the material into a hopper which is an intrinsic part of the kiln structure, and which is fitted with a perforated pipe to supply an air blast to the material. The hopper may be situated centrally with a bottom discharge opening, or laterally with a discharge chute outside the kiln.

C. A. KING.

Furnaces for extracting a metal from an ore. S. C. G. EKELUND (B.P. 309,651, 26.1.28).—Ore is caused to move on a travelling base through a horizontal or inclined tunnel furnace in opposite direction to a stream of gas generated in a slagging gas producer. By a heat interchange with the cooling charge, the gas is preheated and a portion may be diverted and burned at the hot zone of the furnace.

C. A. KING.

Rotary furnace for melting metals. K. SCHMIDT, Assee. of K. SCHMIDT GES.M.B.H. (B.P. 297,432, 20.9.28. Ger., 21.9.27).—The axis of a metal-melting furnace is disposed obliquely to increase the capacity. The charging aperture, burner, and outlet for waste gases are combined at one end, the burner being fitted through the furnace cover, which is made adjustable to allow of the directional heating of the furnace.

C. A. KING.

Reduction of ores of metals capable of volatilisation. F. L. DUFFIELD (B.P. 310,252, 4.8.28).—A charge of zinc, lead, or similar volatile metal, carbon, and lime is introduced at the bottom of a vertical furnace and raised through progressively hotter zones of the furnace. The metal vapours evolved are condensed, and the gases are burned in a combustion chamber surrounding the reduction furnace.

C. A. KING.

Treatment of moulds for producing soft castings. H. A. MYERS, Assr. to METAL CASTINGS HOLDING CO. (U.S.P. 1,706,858, 26.3.29. Appl., 30.8.26).—The mould is faced with a mixture of graphite, magnesia, and potassium cyanide, then heated to such a temperature that the surface is case-hardened by the cyanide.

A. R. POWELL.

Cooled rim on cyanide pots. C. H. CARPENTER and A. D. KEENE, Assrs. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,705,714, 19.3.29. Appl., 4.6.27).—Cyanide case-hardening pots are provided with a wide curved flange dipping into a shallow annular trough, supported by the surrounding refractory brickwork. The pot is heated electrically, and the flange kept cool by air circulation.

A. R. POWELL.

Recuperative annealing furnace. J. SCHNEPF, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,705,686, 19.3.29. Appl., 20.10.27. Ger., 14.8.26).—The furnace comprises an open-end furnace chamber in which the main heating takes place, and two laterally adjacent chambers communicating with the main chamber and open at their outer ends for charging and

discharging the material undergoing heat treatment. One of the lateral chambers receives the material from the main chamber to allow it to cool slowly, and the heat from this chamber is used to preheat a new charge in the other lateral chamber.

A. R. POWELL.

Recovery of iron from iron-containing ores with formation of ferric chloride. VEREIN. STAHLWERKE A.-G. (B.P. 284,991 and Addn. B.P. 288,319, [A] 25.1.28, [B] 5.4.28. Ger., [A] 7.2.27, [B] 7.4.27).—(A) The ore is heated below 600° in an atmosphere of hydrogen chloride or chlorine, and the volatilised ferric chloride is reduced by hydrogen to obtain sponge iron and hydrogen chloride for use again in the process. A small quantity of sponge iron is added as catalyst in the reduction stage. If the ore contains arsenic or antimony it is prechlorinated at about 100°. (B) Iron ore is mixed with magnesium chloride and hydrochloric acid, and the mixture is heated at 150–250° to produce ferric chloride and magnesia. The product is then heated more strongly to volatilise the ferric chloride, which is subsequently reduced to iron by hydrogen. Alternatively, the chloridised product is heated at 150–250° in hydrogen, and the iron is separated from the magnesia by screening and magnetic separation. The hydrochloric acid and magnesia are allowed to interact for the production of magnesium chloride for further use in the process.

A. R. POWELL.

Manufacture of cast iron. E. PIVOVARSKY, ASSR. to EDELGUSSVERBAND G.M.B.H. (U.S.P. 1,705,995, 19.3.29. Appl., 11.2.26. Ger., 21.2.25).—Cast iron is heated in the cupola to a temperature above 1400° at which the carbide present begins to decompose with the separation of finely-divided graphite. On subsequently casting, the whole of the uncombined carbon separates in a finely-divided form, so that a particularly even-grained, tough casting is obtained.

A. R. POWELL.

Metallurgical process and product [cast iron]. L. H. MARSHALL, ASSR. to OHIO BRASS CO. (U.S.P. 1,707,529, 2.4.29. Appl., 19.1.25).—Cast iron is rendered malleable by annealing above 750° until graphitisation is complete, cooling slowly to about 670°, and quenching in water or in air.

A. R. POWELL.

Welding of cast iron and filler rod therefor. H. V. WILLE (U.S.P. 1,708,815, 9.4.29. Appl., 14.4.21).—A welding rod for producing a soft weld in cast iron comprises an iron alloy containing 4–20% C.

A. R. POWELL.

Production of pure iron. Manufacture of steel. H. J. VAN ROYEN (B.P. 282,365–6, 26.11.27. Ger., [A] 14.12.26, [B] 15.12.26).—(A) The charge of pig iron is blown in the converter until it contains less than 0.01% P and 0.02% (Si+Mn), and its ferrous oxide content is then reduced to zero by blowing through it a mixture of carbon dioxide and monoxide in the ratio 1:2.5–7 together with an excess of nitrogen. (B) The charge is converted into steel of any desired carbon content by introducing aliphatic hydrocarbons into the gas mixture when the iron is completely deoxidised.

A. R. POWELL.

Manufacture of iron and steel insensitive to cold-shortness, blue-shortness, and ageing. H. J.

VAN ROYEN (B.P. 295,315, 27.3.28. Ger., 9.8.27).—Iron or steel obtained by the methods described in B.P. 282,365–6 (preceding) is used as the raw material for the process described in B.P. 285,814 (B., 1929, 360).

A. R. POWELL.

Sherardising [iron and steel]. S. O. COWPER-COLES, L. TAYLOR, A. A. GOULD, and P. G. LUCAS (B.P. 308,180, 19.9., 29.11., and 2.12.27).—To obtain a bright lustrous coating of zinc in the sherardising process, 0.5% of arsenic or phosphorus is added to the zinc dust mixture with or without powdered mica or talc. A highly corrosion-resisting coating on iron and steel is obtained by sherardising with electrolytic chromium powder. The sherardising operation may be made continuous by enclosing the articles to be treated in a rotating perforated drum through which the sherardising mixture falls, and which can be replaced by a similar drum in the treatment vessel without introducing air or cooling the apparatus.

A. R. POWELL.

Pickling of metals. IMPERIAL CHEM. INDUSTRIES, LTD., E. CHAPMAN, and A. HILL (B.P. 309,314, 14.4.28).—In pickling iron and steel in dilute sulphuric acid the addition of a thiazole derivative, e.g., dehydrothio-*p*-toluidine, prevents the action of the acid on the metal. The inhibitor may be prepared by heating 7 pts. of *o*-toluidine and 2 pts. of sulphur at 185° for 6 hrs., then at 210–215° for 18 hrs., and dissolving the product in 9 times its wt. of 70% sulphuric acid.

A. R. POWELL.

Bessemer process of making steel. F. W. DAVIS, ASSR. to S. G. ALLEN (U.S.P. 1,709,389, 16.4.29. Appl., 2.6.24).—Non-Bessemer pig iron is blown with a blast enriched in oxygen sufficiently to supply the necessary heat for the reaction.

A. R. POWELL.

Treatment of freshly-cast steel ingots. GEBR. BÖHLER & CO. A.-G., WIEN (Austr. P. 108,125, 4.5.26).—The ingots are cast in double-walled moulds, the space between the walls being evacuated or packed with heat-insulating material. The thickness of the inner wall is so adjusted that considerable supercooling of the steel takes place before solidification. Subsequent annealing is thus rendered unnecessary.

A. R. POWELL.

Surface-hardening of metal bodies or articles by heat-treatment. CAMMELL, LAIRD & CO., LTD., J. MCN. ALLAN, and A. P. HAGUE (B.P. 310,347, 23.1.28).—Steel articles are heated in a furnace maintained at 1100–1500° by rotating either the article in the furnace or the furnace around the stationary article. The article on cooling is withdrawn when it has attained a suitable hardening temperature (about 800°) and is then chilled.

C. A. KING.

Preparation of low-carbon [iron-chromium] alloys. V. B. BROWNE (U.S.P. 1,710,446, 23.4.29. Appl., 21.10.25).—Rustless chromium-iron alloys are obtained by melting in a carbon-arc furnace a charge comprising a low-carbon iron-chromium alloy, lime, and manganese oxide so proportioned as to prevent access of carbon to the bath.

A. R. POWELL.

Malleable iron alloy. A. L. BOEGEHOLD, ASSR. to GEN. MOTORS RES. CORP. (U.S.P. 1,707,753, 2.4.29. Appl., 14.3.27).—The alloy contains 1.3–2.5% Si,

2—2.75% C, and about 0.5% Mo, the last ingredient serving to obstruct the formation of flake graphite during solidification so that a white iron is obtained on casting.
A. R. POWELL.

Annealing of silicon-steel sheets. W. E. CAUGHERTY and C. J. S. STROBLE, Assrs. to ALLEGHENY STEEL CO. (U.S.P. 1,706,438, 26.3.29. Appl., 30.6.27).—Thin silicon-steel sheets are laid one, two, or three at a time on a flat base in an annealing furnace, and as soon as the first charge has reached the annealing temperature a second equal charge is placed on top of it, heated to the same temperature, and the operation repeated until the furnace is full; it is then closed and maintained at the annealing temperature (830°) for 1—3 hrs.

A. R. POWELL.

Manufacture of iron or alloys thereof. A. A. FREY (B.P. 234,976, 6.2.28. U.S., 5.2.27).—Iron or steel scrap is melted under a lime-carbon charge until the sulphur is completely eliminated, the slag is removed, and the metal tapped into a basic converter in which it is blown until all the carbon, phosphorus, manganese, and silicon are removed and the metal contains 1% or more of ferrous oxide. At this stage the blast is turned off and the charge allowed to settle before running off the slag. The converter is again turned on but, instead of air, a reducing gas, preferably hydrogen, is blown through the metal until the latter is thoroughly de-oxidised. Silicon, arsenic, or aluminium may be added at the end of the reduction to obtain alloys of these metals with iron.

A. R. POWELL.

Manufacture of materials [steel] containing tungsten. H. G. BATCHELLER and J. O. KELEY, Assrs. to LUDLUM STEEL CO. (U.S.P. 1,709,817, 16.4.29. Appl., 6.10.25).—Tungsten is introduced into the steel bath in the form of a friable conglomerate comprising partially reduced tungsten trioxide and a slag composed of sodium carbonate and the manganese, arsenic, and tin compounds present as impurities in the oxide.

A. R. POWELL.

Metallic alloy. BARBER ASPHALT CO., Assees. of A. B. DAVIS (B.P. 300,249, 16.4.28. U.S., 10.11.27).—An alloy of iron containing 15—25% (20) Ni, 10—20% (14.5) Cr, 3—8% (5) Co, 2—8% (4.5) Cu, the quantity of the last two elements being more than 7%. Tungsten, molybdenum, and silicon may also be included as modifiers.

C. A. KING.

Refractory iron alloys. E. O. VOGEL and C. F. C. WEICHELT (MEIER & WEICHELT) (B.P. 287,876, 27.3.28. Ger., 28.3.27).—The alloys contain 12—20% Ni, 16—20% Cr, 0.3—3% Si, and up to 1% C, the silicon content being at least equal to 2.5 times the carbon content and the sum of the silicon and chromium exceeding 19%. [Stat. ref.]

A. R. POWELL.

[Iron-nickel-chromium] alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 308,643, 23.12.27. Addn. to B.P. 284,789; B., 1928, 271).—Alloys having a differential permeability greater than iron and a high electrical resistance comprise 40—50% Fe, 1—5% Si or Al, 1—5% Cr or similar element, and the remainder (not less than 42%) nickel. In addition the alloys may contain 0.2—0.3% Mn and up to 5% Cu. The

alloys are substantially free from carbon; they are heat-treated for 15 min. at 900—1100°, then slowly cooled to the ordinary temperature. A. R. POWELL.

[Nickel-molybdenum-iron] alloy. F. M. BECKET, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,710,445, 23.4.29. Appl., 10.1.28).—The alloy contains Ni 40%, Mo 15—40%, and Fe not less than 10% nor more than five times the amount by which the molybdenum exceeds 10%.

A. R. POWELL.

Alloy steels [for permanent magnets]. C. K. EVERITT, and E. ALLEN & CO., LTD. (B.P. 308,549, 20.9.28).—The steel contains 0.5—2% C, 6—12% Cr, 15—40% Co, 0.3—1.5% Si, 1—6% W, and 0.5—4% Mo. The cast alloy is heated at 750°, cooled, reheated at 950—1000°, and quenched in oil or air. Magnets made from this steel are characterised by a high remanence and coercive force.

A. R. POWELL.

Treatment of slags containing metals obtained in the production of ferrochromium from ores. D. W. BERLIN (Swed. P. 59,975, 4.10.22).—The slags are fused in an electric furnace with aluminium or silicon, and the resulting metal is fused with ferric oxide to remove excess of the reducing agent.

A. R. POWELL.

Coating of metallic surfaces for prevention of corrosion. J. DAVIES (B.P. 310,696, 7.6.28).—For the protection of iron and steel, and particularly water tanks, the metal is coated with a mixture of 65% of Portland cement, 10% of casein, 15% of calcined magnesia, and 10% of asbestos powder to which an equal volume of water is added. Magnesia and asbestos may be omitted from the composition. After drying, the coating is washed with formaldehyde and sprayed with cellulose lacquer.

C. A. KING.

Coating band iron with a rust-protection material. C. RÖTZEL (B.P. 284,191, 23.1.28. Ger., 22.1.27).—Band iron after passing through a hot-rolling mill is immediately immersed, while still hot, in oil or grease.

J. S. G. THOMAS.

Froth-flotation concentration of ores. R. B. MARTIN, Assr. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,686,529, 9.10.28. Appl., 11.12.26).—In the concentration of zinc ores the copper sulphate used in the froth-flotation process may be replaced with advantage by an organic copper compound, e.g., aniline cupric chloride, benzidine cupric chloride. R. BRIGHTMAN.

[Flotation] concentration of oxidised ores. J. T. TERRY, Assr. to C. E. TERRY (U.S.P. 1,709,329, 16.4.29. Appl., 23.11.25).—Oxidised ores of lead, zinc, silver, mercury, or bismuth are subjected in the form of a finely-ground pulp to the action of a soluble chromate which converts the surfaces of the mineral particles into insoluble chromates; the pulp is then subjected to froth-flotation.

A. R. POWELL.

Flotation oil. R. LUCKENBACH (U.S.P. 1,688,975, 23.10.28. Appl., 23.5.25).—Flotation oil suitable for the recovery of, e.g., oxidised portions of copper ore, consists of 50—70% of heavy residuum, 10—15% of petroleum diluent, and 10—30% of a frothing agent containing 20—50% of soft soap, together with, e.g., cotton-seed foots, pine creosote, and Turkey-red oil.

R. BRIGHTMAN.

Concentration of manganese ore. H. and A. J. TRUMBO, Assrs. to MANGANESE PATENTS CORP. (U.S.P. 1,710,522, 23.4.29. Appl., 20.9.27).—The ore is ground, classified, cleaned with a solution of sodium carbonate, and concentrated on jigs, tables, and magnetic separators. The tailings from the concentration processes are digested with sulphur dioxide solution to recover their manganese content.

A. R. POWELL.

Recovery of copper from copper-rich material. S. I. LEVY and G. W. GRAY (B.P. 309,269, 10.2.28).—The insoluble residue containing copper, obtained by digestion with hydrochloric acid of the product resulting from heating pyrites at 600° in a neutral atmosphere, is dried at 100° and again digested with hot hydrochloric acid to remove the greater part of the zinc and iron. The residue from this treatment is roasted, with or without the addition of ferrous chloride, and leached to recover copper and zinc sulphate or chloride.

A. R. POWELL.

Melting and refining of copper. H. H. ALEXANDER (B.P. 310,356, 16.1.28. Cf. B.P. 309,848; B., 1929, 479).—Dissolved oxide is removed from molten copper by blowing beneath the surface of the metal fine streams of steam or other gas containing powdered coal, coke, or charcoal in suspension.

A. R. POWELL.

Recovery of copper and nickel. CHEM. FABR. JOHANNISTHAL G.M.B.H., and F. TROSTLER (B.P. 283,132, 19.12.27. Ger., 4.1.27).—Copper-nickel alloys, mattes, or speisses are oxidised anodically in slightly alkaline 10–20% sodium chloride solution to produce an anode slime containing copper and nickel hydroxides. The slime is collected, washed, and digested with copper sulphate solution, whereby the nickel hydroxide dissolves with the precipitation of an equivalent quantity of copper hydroxide. This is collected, washed, and converted into metal in the usual way.

A. R. POWELL.

Manufacture of moulding bodies of metals. GEBR. SIEMENS & Co., Asses. of E. BIRNBRÄUER (Austr. P. 108,103, 4.11.22. Ger., 5.11.21 and 5.8.22).—Solutions of copper salts or of salts of the metals of the iron group are treated in such a way as to obtain the metal or its hydroxide in a finely-divided form. The precipitate is collected, washed, and reduced above 600° with hydrogen, carbon monoxide, or methyl alcohol. The metal is thus obtained in a plastic, difficultly oxidisable form suitable for use as a solder or metallic cement.

A. R. POWELL.

Metallurgy of ores etc. containing tin. E. A. ASHCROFT (B.P. 309,307, 5.4.28).—The material is heated with ammonium chloride and, if necessary, with finely-divided iron to convert the tin into stannous chloride, which is recovered by volatilisation or by lixiviation. The ammonia evolved in the first stage is utilised in recovering the metal subsequently obtained in solution.

A. R. POWELL.

Recovery of tin from ores etc. H. L. SULMAN and H. F. K. PICARD (B.P. 310,639, 14.4., 5.7., and 1.8.28).—A mixture of low-grade tin concentrates and pyrites is bonded with coal dust and tar, and the briquettes are heated to effect sulphidisation of the tin without volatilisation. The porous masses so formed are heated

on a grate in a current of air, whereby the tin sulphide volatilises and is immediately converted into tin oxide fume, which is recovered in a bag plant or other collector.

A. R. POWELL.

Production of metallic tin. CAYZER TIN SMELTING Co. (PROPRIETARY), LTD., Asses. of W. J. CAYZER (B.P. 306,445, 29.9.28. S. Africa, 20.2.28).—A mixture of tin ore or concentrate and an excess of carbonaceous material is charged on to a bed of spongy metal forming the hearth of a furnace which is maintained at 900–1000°, the furnace atmosphere being not less than 60% CO. Molten tin, as produced, percolates through the bed of sponge metal (iron), which may be formed or maintained by the introduction of hæmatite with the charge.

C. A. KING.

Recovery of zinc [from sulphate leach liquors]. L. F. W. LEESE (B.P. 309,288, 19.3. and 13.9.28).—Zinc sulphate solutions obtained in the hydrometallurgical treatment of ores containing zinc are passed over scrap iron to remove copper, treated with zinc hydroxide to precipitate ferrous hydroxide, and passed through a granular mass of magnesia to precipitate zinc hydroxide.

A. R. POWELL.

Manufacture of electrolytic zinc. I. G. FARBERIND. A.-G. (B.P. 285,373, 6.2.28. Ger., 14.2.27).—A solution of zinc sulphate containing 15–25% of free sulphuric acid is electrolysed at a current density of 200–500 amp./m.²

J. S. G. THOMAS.

Electrodeposition of pure chromium in thick layers. G. GRUBE (G.P. 454,168, 20.7.21).—The electrolyte contains chromic acid, chromic oxide, and a mineral acid in such proportions that the concentration of the chromic acid is more than double that of the total other constituents and that of the chromic oxide is more than sufficient to neutralise the mineral acid.

A. R. POWELL.

Smelting of tungsten ores. F. M. BECKET and W. C. READ, Assrs. to ELECTRO METALLURGICAL Co. (U.S.P. 1,705,655, 19.3.29. Appl., 26.3.28).—Tungsten ores containing sulphur, arsenic, tin, and/or copper are ground finely together with silicon, bleaching powder, and sulphur chloride, with or without a reaction promoter such as sodium chloride. The mixture is heated to such a temperature that an exothermic reaction takes place and the tungsten ore is reduced to ferrotungsten without fusion; complete volatilisation of the impurities as chlorides takes place simultaneously.

A. R. POWELL.

Aluminium-base alloy. S. DANIELS (U.S.P. 1,710,148, 23.4.29. Appl., 27.3.25).—The alloy contains 2.25–3% Cu, 0.75–1.5% Fe, and a small percentage of magnesium and silicon, the ratio Mg:Si being about 1.7:1.

H. ROYAL-DAWSON.

Apparatus for centrifugal purification of metals and the like. E. R. HEWITT (U.S.P. 1,709,939, 23.4.29. Appl., 7.4.23).—The apparatus is similar to a centrifuge, the basket being replaced by a container lined with refractory material. After centrifuging, the container is brought steadily to a standstill and the purified metal is run off from the bottom.

C. A. KING.

Purification of light metals. H. E. BAKKEN, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,710,398,

23.4.29. Appl., 21.7.26).—Entrained impurities are removed from a readily oxidisable light metal by passing the liquid metal through a porous mass of a neutral metal.
C. A. KING.

Production of metallic caesium. R. E. MIESSE, Assr. to NEW PROCESS METALS CORP. (U.S.P. 1,707,637, 2.4.29. Appl., 3.1.27).—A mixture of caesium chloride and a rare-earth metal alloy is heated at 270° in an exhausted glass vessel until all traces of gas are removed, and is then more strongly heated by radiation from an electric heater until reaction takes place with the production of caesium metal and rare-earth chlorides.
A. R. POWELL.

Manufacture of thin metallic foil. C. MÜLLER, Assr. to K. MEY (U.S.P. 1,709,801, 16.4.29. Appl., 9.3.25. Ger., 4.3.24).—A coherent foil-forming layer of desired thickness produced by deposition is sandwiched between two other layers, which are then removed by molecular decomposition, the outer layers being of sufficient thinness to avoid detrimental stress being exerted on the inner layer during their removal.
J. S. G. THOMAS.

Production of porous metal articles from metal powder. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 311,141, 9.7.28).—Iron or nickel powder produced by the thermal decomposition of the corresponding carbonyl is charged into a flat, rectangular mould until the mould is loosely filled, and the whole is heated at 500–600° in a current of hydrogen with or without a previous application of pressure. The product is a porous sintered plate suitable for use as an electrode in alkaline storage batteries.
A. R. POWELL.

Coating of perforated metal sheets and articles with rubber. ANODE RUBBER CO., LTD., ASSEES. OF ANODE RUBBER CO. (ENGLAND), LTD. (B.P. 301,300, 5.4.28. Ger., 26.11.27).—Perforated metal sheets or articles, *e.g.*, reels or spools for the rayon industry, are covered with a protective seamless layer of rubber, which also rounds off any sharp edges, by dipping or by electrophoretic deposition, using aqueous dispersions containing rubber. According to the composition of the deposit produced, the layer, after drying and vulcanisation, may be either of soft rubber or vulcanite.
D. T. TWISS.

Treatment of metal articles for prevention of tarnish and rust. H. A. SCRIVEN and B. F. G. GUISE (B.P. 309,339, 7.6.28).—The articles are plated with a coating of nickel and silver in equal proportions, then dipped in a hot solution of celluloid and soap in amyl acetate, and finally dried at 32° for several hrs.
A. R. POWELL.

Anti-rust or anti-corrosive preparation. E. W. TERRY (B.P. 310,640, 16.4.28).—Bitumen and coal tar are mixed with petroleum to the consistency of treacle.
C. A. KING.

Steel alloy. E. HOUDREMONT and V. EHMCKE, Assrs. to F. KRUPP A.-G. (U.S.P. 1,711,733, 7.5.29. Appl., 11.1.28. Ger., 28.1.27).—See B.P. 284,314; B., 1928, 756.

Refinement of nickel alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,710,846, 30.4.29.

Appl., 3.5.28. U.K., 25.4.27).—See B.P. 296,112; B., 1928, 820.

Solder for aluminium or its alloys. L. R. PRESTON (U.S.P. 1,711,807, 7.5.29. Appl., 8.12.26. U.K., 14.12.25).—See B.P. 265,733; B., 1927, 303.

[Magnetic alloys for] loaded conductor. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,710,805, 30.4.29. Appl., 3.5.28. U.K., 30.4.27).—See B.P. 296,137; B., 1928, 790.

Mercury boilers (B.P. 293,756).—See I. **Treating ferrous chloride solutions (B.P. 309,268).**—See VII. **Muffle (U.S.P. 1,710,870).**—See VIII. **Protection of metal (B.P. 310,554).**—See IX. **Heating strip metal (B.P. 297,311).**—See XI. **Bonding rubber to metal (U.S.P. 1,689,628).**—See XIV. **Precious metals from sea-water (B.P. 294,655).**—See XXIII.

XI.—ELECTROTECHNICS.

Insulating oils. STADNIKOV and VOSSCHINSKAJA.—See II. **Aluminium armatures.** VON GRONOW. **Theory of chromium plating.** OLLARD.—See X.

PATENTS.

Electric furnace. P. K. DEVERS, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,708,833, 9.4.29. Appl., 13.7.25).—A heater is arranged between a vacuum-tight outer envelope and an enclosed, charge-receiving graphite vessel which can be evacuated. J. S. G. THOMAS.

Electric furnace. O. A. COLBY, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,709,658, 16.4.29. Appl., 14.7.27).—Gas is forced through oxidisable material to be heated in the furnace chamber, and then passes into and through the furnace chamber so that a non-oxidising atmosphere is maintained therein. J. S. G. THOMAS.

Electric furnace. H. ELDRIDGE, Assr. to M. K. ELDRIDGE (U.S.P. 1,711,088, 30.4.29. Appl., 19.5.25).—An electrode extends into a stationary side-wall structure of a furnace having a portable bottom formed with a central cavity the side walls of which can be raised into intimate contact with the stationary side wall. A discharge port communicates with the cavity and can be closed by a second electrode arranged therein. J. S. G. THOMAS.

Electrical [resistance] furnace for production or fusion of silicates, *e.g.*, glass. C. E. CORNELIUS (Swiss P. 122,176, 4.5.26).—The material under treatment serves as the resistance, and the electrodes are constructed of metals having the property of storing heat, *e.g.*, iron, are of such a shape that they are in maximum contact with the charge, and are provided with openings through which the molten material is withdrawn. L. A. COLES.

Electric furnace for heating strip metal by induction currents. HIRSCH KUPFER- u. MESSING-WERKE A.-G., and M. TAMA (B.P. 297,311, 15.3.28. Ger., 17.9.27).—Metal strip wound into the form of a coil is placed in an ironless induction furnace and forms a secondary circuit in which currents are induced by the field generated by a current of 50–60 cycles in the primary. J. S. G. THOMAS.

Copper oxide electrode and its manufacture. E. W. WESCOTT, Assr. to COMSTOCK & WESCOTT, INC. (U.S.P. 1,711,462, 30.4.29. Appl., 10.11.25).—Granular copper oxide is bonded by fine soft copper flowed under high pressure. J. S. G. THOMAS.

Copper oxide electrodes for galvanic purposes. W. ADLER and F. SCHIEBALY (Austr. P. 108,153, 6.10.25).—A plastic mass composed of copper oxide, a plastic binder, e.g., clay, chamotte, and water is moulded, if desired about wire gauze etc., coated with strengthening material, and burned. J. S. G. THOMAS.

[Oxide electrode for] galvanic batteries. E. WOLFF (B.P. 305,009, 30.4.28. Ger., 28.1.28).—Copper oxide is carried between two undulated sheet-metal plates having numerous narrow perforations and placed so that the apices of the respective sets of undulations cross each other at right angles. J. S. G. THOMAS.

Manufacture of electrodes for Edison accumulators. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,383, 25.11.27).—Extremely fine, pure, non-pyrophoric iron and nickel powders suitable for use, respectively, in the cathodes and anodes of Edison accumulators are produced by thermal decomposition of the corresponding metallic carbonyls. Thus, iron carbonyl is decomposed in the free space of a heated vessel at about 250°, whilst nickel carbonyl vapour is decomposed at 200°. The powders may subsequently be treated with hydrogen at about 500°, and/or may be subjected to pressure. J. S. G. THOMAS.

Electrodes for use in electric arc-welding. QUASI-ARC Co., LTD., and A. P. STROHMENGER (B.P. 308,475, 27.3.28).—A core metal containing 2–4% Mn is covered with a flux-forming material, e.g., blue asbestos with a strip of aluminium, the percentage of manganese being proportioned relatively to the gauge of the core metal so that the fused metal deposited contains about 0.45% Mn. J. S. G. THOMAS.

Electrode for use in apparatus for the decomposition of gases. NORDISKE FABRIKER DE NO FA, A./S., and C. F. HOLMBOE (Austr. P. 108,289, 28.11.25).—Fluted ribs separated by conducting distance pieces are held together by screw bolts. J. S. G. THOMAS.

Manufacture of electron-emitting bodies [cathodes]. E. Y. ROBINSON, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (B.P. 307,099, 1.12.27).—A core of metal, e.g., nickel or platinum, or of carbon, immersed in alkaline-earth metal powder or amalgam, e.g., of barium, is heated *in vacuo* or in an inert or reducing atmosphere at about 1000° so that metal vapour penetrates the core, which is afterwards assembled in a valve. J. S. G. THOMAS.

Production of oxide incandescence cathodes. V. H. WOHL (Austr. P. 107,954, 26.5.26).—Electrically heated metallic filaments are drawn a number of times through solutions or suspensions of compounds of the alkaline-earth metals from which the alkaline-earth metal is deposited on heating, or through liquid amalgams of the alkaline-earth metals, and the deposited metal is oxidised by heating in air. J. S. G. THOMAS.

[Gas-filling for] electron tubes, more especially for incandescence cathode Röntgen tubes. C. H. F. MÜLLER (Swiss P. 122,456, 18.6.26).—Attack of metal parts of the tubes is prevented by filling the tubes with neon containing a small proportion, e.g., 10%, of hydrogen or helium, at a pressure exceeding 0.0006 mm. of mercury. J. S. G. THOMAS.

Marking of incandescence lamps and the like. N. V. PHILIPS' GLOEILAMPENFABR. (Dutch P. 17,399, 10.3.26).—The internal surfaces of the glass bulbs etc. are marked by heating a stamped mixture of silica, alumina, and oxides of lead (litharge), boron, calcium, magnesium, cobalt, and an alkali oxide. J. S. G. THOMAS.

Luminescent tube. P. F. J. LEBRUN (U.S.P. 1,709,858, 23.4.29. Appl., 26.8.27).—The tube is filled with argon, neon, and mercury vapour. H. ROYAL-DAWSON.

Electro-endosmosis method and apparatus. V. A. LAPENTA (U.S.P. 1,711,416, 30.4.29. Appl., 10.8.25).—A membranous bag containing material to be treated, and a pair of porous cells containing respectively a positive and a negative electrode, are arranged in a vessel containing electrolyte, and current is passed between the electrodes. J. S. G. THOMAS.

Magnetic material. [Nickel-iron-cobalt alloy.] P. P. CIOFFI, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,708,936, 16.4.29. Appl., 23.12.26).—The heated alloy is subjected, during cooling, to a field which substantially neutralises the effect of extraneous fields, in order to obtain a material of constant magnetic permeability over a wide range of field intensities. J. S. G. THOMAS.

Manufacture of bodies [dynamo brushes] from metal powder. C. F. SHERWOOD (U.S.P. 1,708,192, 9.4.29. Appl., 7.5.27).—Powdered metal in the form of a brush moulded integrally with a part of a preformed conductor is sintered in a fluxing bath of molten salt, m.p. 650–815°. J. S. G. THOMAS.

Production and application of plates for electric condensers etc. J. Y. JOHNSON. From I. G. FARBENIND, A.-G. (B.P. 310,625, 22.3.28).—Finely-divided, electrically conducting materials, e.g., metals, graphite, charcoal, etc., incorporated with paper pulp are worked up into foil of suitable thickness as, e.g., in the process of paper manufacture. J. S. G. THOMAS.

Impregnation material for use in electrical condensers. TELEPHON-APPARAT-FABR. E. ZWIETUSCH & Co., G.M.B.H. (B.P. 299,697, 27.7.28. Ger., 29.10.27).—A mixture of ozokerite and colophony (9 : 1) is used. For use in an atmosphere permeated with steam or varying materially in temperature 0.5 pt. of carnauba wax is used in place of 0.5 pt. of colophony. J. S. G. THOMAS.

Electrical [electrolytic] condenser. S. RUBEN (U.S.P. 1,710,073, 23.4.29. Appl., 21.3.27).—A highly viscous paste composed of glycerin and a film-forming, supersaturated, plastic composition of boric acid and sodium borate is introduced between film-forming electrodes separated by a spacer medium, impregnated

to prevent electrolytic action between its fibres, the electrolyte, and the surface of the electrodes.

J. S. G. THOMAS.

Apparatus for sorting or classifying articles by means of light. WESTINGHOUSE ELECTRIC & MANUF. CO., ASSEES. OF V. K. ZWORYKIN (B.P. 292,474, 18.6.28. U.S., 16.6.27).—The surface of such materials as tin-plated or galvanised sheets, cardboard, paper, etc. is inspected for imperfections by a number of closely-spaced photo-electric cells illuminated (preferably from a single linear source of light) by reflection from the article to be tested, which is moved beneath the cells by means of rollers or other means. If no sheet is present under the cells, light is reflected from a special reflector just below the plane of travel of the sheets to prevent unnecessary operation of the rejecting mechanism. Any diminution of the light reaching any cell operates, through a thermionic valve and amplifiers, a solenoid-deflecting mechanism causing rejection of defective plates. A balanced circuit is used in which the cells are connected in series in two groups (like a Wheatstone bridge) so that one thermionic valve only need be provided for all the cells.

B. M. VENABLES.

[Colourless] light filter [for ultra-violet rays]. F. ZERNIK (U.S.P. 1,688,259, 16.10.28. Appl., 27.4.25. Ger., 8.5.24).—Hydroxyquinolinesulphonic acids and their alkyl or aryl others, e.g., 8-methoxyquinoline-5-sulphonic acid, m.p. 302° (decomp.), 8-ethoxyquinoline-5-sulphonic acid, m.p. 283° (decomp.), and sodium 6-methoxyquinoline-5-sulphonate [free acid, m.p. 345° (decomp.)], are used.

R. BRIGHTMAN.

Electrolytes for electrolytic rectifiers or the like. FANSTEEL PRODUCTS CO., INC., ASSEES. OF E. W. ENGLE (B.P. 283,208, 30.12.27. U.S., 7.1.27).—See U.S.P. 1,680,210; B., 1928, 760.

Electric discharge tubes. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 310,829, 17.7.28).

Ionisation etc. of hydrocarbon materials (U.S.P. 1,709,814—5). Treating hydrocarbon-nitrogen mixtures (B.P. 296,355).—**Liquid hydrocarbons** (B.P. 284,224).—See II. **Synthesis of nitrogen compounds etc.** (B.P. 309,001—2).—See III. **Iron-nickel-chromium alloys** (B.P. 308,643). **Steel for magnets** (B.P. 308,549). **Recovery of copper and nickel** (B.P. 283,132). **Porous metal articles** (B.P. 311,141). **Zinc** (B.P. 285,373). **Chromium** (G.P. 454,168).—See X. **Plastic insulating materials** (U.S.P. 1,688,500).—See XIV. **Beet juice** (G.P. 452,436).—See XVII.

XII.—FATS; OILS; WAXES.

Relation between titre and refractive index of fat [during hardening]. A. MARKMAN and M. SERGEJEV (Oil Fat Ind. [Russia], 1928, No. 7, 27—29; Chem. Zentr., 1928, ii, 2418).—No definite and constant relation was observed.

A. A. ELDRIDGE.

Hardening of linseed oil. V. VASSILIEV (Oil Fat Ind. [Russia], 1928, No. 7, 9—11; Chem. Zentr., 1928, ii, 2417—2418).—Chemical constants of linseed oil before and during saturation with hydrogen were determined; 11.7% of the hydrogen was required for the

reduction of hydroxyl groups, the original acid value being high (37.1).

A. A. ELDRIDGE.

Determination of moisture and oil in soya beans. L. BURSCH (Oil Fat Ind. [Russia], 1928, No. 2, 13—15; Chem. Zentr., 1928, ii, 2418).—The moisture content of cleaned and ground soya beans is about 1.94% smaller than corresponds with the analysis of the whole bean, the value for the oil content being correspondingly (0.46) high. It is necessary to determine (by distillation with xylene) the moisture content and the oil content of the ground bean, and the moisture in the whole bean, and to calculate the oil content of the latter.

A. A. ELDRIDGE.

Detection of adulterants in cacao butter by oxidation with perbenzoic acid. K. BODENDORF (Pharm. Ztg., 1929, 74, 384—385).—The degree of unsaturation and the rates of reaction determined with perbenzoic acid are highly characteristic for fats commonly used as adulterants. These fall into two classes, respectively much more and much less unsaturated than cacao butter itself. The fat dissolved in chloroform is treated with the acid in the same solvent, and samples withdrawn at intervals of 20 min. are titrated iodometrically; the results are plotted. The method will not detect simultaneous presence of a highly unsaturated and a nearly saturated adulterant.

S. I. LEVY.

Solubility tests of castor oil. H. P. TREVITHICK and M. F. LAURO (Oil & Fat Ind., 1929, 36, [3], 27—29).—The sp. gr., free fatty acids, and viscosity of castor oil increase slightly on ageing; the most important change, however, is the decreased solubility in alcohol. It is recommended that failure to pass solubility tests specifying alcohol below 95% in strength should not be considered proof of adulteration of castor oil since samples of oil, possessing otherwise the characteristics of pure castor oil and completely soluble in 95% alcohol, yielded turbid mixtures in 90% alcohol (1:2) and by the Finkener test after keeping for 10 months. The insoluble portion was isolated and had acetyl value 136, iodine value 79.4, showing that this portion was hydroxylated and not an adulterant. Concordant results could not be obtained by the filtration method of determining the acetic acid liberated from the acetylated oil; the distillation method is recommended, and details are given of a modified procedure wherein phosphoric acid is substituted for sulphuric acid.

E. LEWKOWITSCH.

Alkali-refining of castor oil. M. BAUMAN (Oil Fat Ind. [Russia], 1928, No. 7, 12—14; Chem. Zentr., 1928, ii, 2520).—Before treatment with alkali the oil is mixed at 20° with an equal volume of petroleum. The refined oil had d_{20}^{20} 0.9682, iodine value 82.4, ash less than 0.01%, ignition temperature (Brenken) about 300°.

A. A. ELDRIDGE.

Extraction of castor oil with benzene. A. SLASCHTSCHEV (Oil Fat Ind. [Russia], 1928, No. 3, 9—11; Chem. Zentr., 1928, ii, 2520).—The yield of castor oil on extraction with benzene is 1.5—2% less than when alcohol is used.

A. A. ELDRIDGE.

Petroleum and alcohol as solvents for castor oil. A. SLASCHTSCHEV (Oil Fat Ind. [Russia], 1928, No. 7,

15—20; Chem. Zentr., 1928, ii, 2520).—Benzine, near the b.p., is a satisfactory solvent for castor oil; benzene, carbon tetrachloride, and carbon disulphide are effective, but the resulting oil has a dark colour. Alcohol gives inaccurate values, since other substances are also extracted.

A. A. ELDRIDGE.

Synthetic crude oil from cholesterol and from *Lycopodium clavatum*. N. A. ORLOV (J. Appl. Chem. Russia, 1928, 1, 117—118).—Cholesterol (50 g.) was heated with a mixture of alumina and ferric oxide (3 g.) in hydrogen at 400—450° and 175 atm. for 14 hrs. The hydrogen then contained 7% of saturated compounds (calc. as CH₄), and 43 g. of a transparent, yellow oil, $[\alpha] +1^{\circ}54'$, remained. The oil was fractionated and its behaviour with sulphuric acid and with potassium permanganate was examined. The oil from lycopodium heated under pressure with hydrogen was likewise examined.

CHEMICAL ABSTRACTS.

Alkaline-earth stearate [emulsions]. A. P. LEE and J. E. RUTZLER (Oil & Fat Ind., 1929, 6, [3], 15—18).—The relative properties of emulsions of purified cottonseed oil stabilised by the stearates of magnesium, calcium, strontium, and barium have been investigated. All the emulsions were of the water-in-oil type. The magnesium and strontium stearates were insoluble in the oil at 25°, although traces of the metals could be found spectroscopically. The stability of emulsions stabilised with magnesium stearate (as measured by the degree of separation after 15 days) was the greater as the amount of peptiser was increased (0.001—0.006 mol./50 g. of oil), the curve showing a tendency to flatten. The stability and viscosity of emulsions containing equimolar (0.004/50 g.) quantities of the alkaline-earth stearates increased with the weight of the metal: barium stearate produced the only truly stable emulsion, which further showed a disproportionally great viscosity, perhaps attributable to a specific action of this peptising agent. Preliminary experiments showed that the viscosity of emulsions stabilised by strontium stearate increased continuously for a short time after production.

E. LEWKOWITSCH.

Electrical apparatus for the extraction in the laboratory of fats and oils. F. GOGOLEV (Oil Fat Ind. [Russia], 1928, No. 3, 18—19; Chem. Zentr., 1928, ii, 2417).

Litharge-glycerin cements. STÄGER.—See IX. **Nickel catalyst,** MASCHKILLEISSON; WOLFSON.—See X. **Butter fat in dairy products.** CHAPMAN.—See XIX.

PATENTS.

[Manufacture of soft] soap. O. H. CARLSON (U.S.P. 1,705,852, 19.3.29. Appl., 16.9.27).—Soft soap is obtained by adding to a boiling mixture of Castile soap, sodium bicarbonate, borax, and glycerol a boiled mixture of stearic acid and sodium carbonate, boiling the product, cooling, and adding ether until the mass gels.

A. R. POWELL.

Extraction of unsaponifiable and difficultly-saponifiable matter from fatty material. J. K. MARCUS (U.S.P. 1,690,091, 30.10.28. Appl., 30.4.27).—Fatty material, e.g., cod-liver oil, is saponified with alcoholic potassium hydroxide, and the viscous-solid

soap mass, adjusted to 30% aqueous alcohol, is extracted with ethylene dichloride, the heavier extract layer being run off free from dissolved soap.

R. BRIGHTMAN.

Production and use of stable sulpho-acids of high mol. wt. and their salts. ORANIENBURGER CHEM. FABR. A.-G., ASSOCES. OF CHEM. FABR. MILCH A.-G. (B.P. 288,126, 20.12.27. Ger., 1.4.27).—When neutral fats, fatty acids, resins, etc. are mixed with aliphatic or aromatic carboxylic acids, hydroxy-acids, keto-acids, or their anhydrides or chlorides, and treated with strong condensing-sulphonating agents such as halogenated sulphonic acids or mixtures of sulphuric acid and phosphoric pentoxide, water-soluble products of enhanced utility in the pasting of dyes, stabilising of dye solutions, splitting of fats, and for cleaning, wetting, and oiling purposes, are obtained.

E. HOLMES.

Manufacture of compressed cakes [from soap powder]. F. KILIAN (B.P. 311,070, 14.4.28).

Composition for bleaching (U.S.P. 1,687,303—5). See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Chemical composition of Russian turpentine oil from *Pinus sylvestris*. B. ARBOUSOV (J. Russ. Phys. Chem. Soc., 1929, 61, 255—268).—A résumé of previous researches on the chemical composition of turpentine from various conifers. The presence of *d*- α -pinene in the turpentine from *Pinus sylvestris* had been definitely established, but data concerning the two other main constituents (nopinene and *d*- Δ^3 -carene) has been indefinite. In the present investigation the turpentine was thoroughly dried and fractionated, and the sp. gr. b.p., sp. rotation, and coefficient of dispersion were determined. The turpentine contained 80—85% of *d*- α -pinene, b.p. 49.6°/13 mm., d^{15}_D 0.8625, $[\alpha]_D^{15}/[\alpha]_C = 1.97$ —1.98. Examination of the physical constants of the higher fractions showed the presence of about 14% of *d*- Δ^3 -carene, b.p. 165—168°, $d^{17.2}_D$ 0.8593, $[\alpha]_D +13.34^\circ$, $[\alpha]_D^{17.2}/[\alpha]_C = 2.12$; there were no indications of the presence of nopinene. That *d*- Δ^3 -carene was present in the turpentine fraction was confirmed by Aschan's method—a purple coloration with acetic anhydride and concentrated sulphuric acid, and by the formation of a crystalline nitroso-compound, m.p. 145.5°, on treatment with amyl nitrite, glacial acetic acid, and sulphuric acid.

M. ZVEGINTZOV.

Synthetic resins as construction materials in chemical industry. E. KALMAN (Chem. Fabr., 1929, 169—170).—On account of their indifference to acids and alkalis, chlorine, and organic and inorganic solvents and reagents, the phenol-formaldehyde resins are employed for the construction of vessels and plant of all kinds; a container weighing 1.8 tons, constructed in one piece without strengthening by iron or otherwise in any way, is claimed to be the biggest piece of apparatus of any kind ever constructed without joints from a single piece of material.

S. I. LEVY.

Litharge-glycerin cements. STÄGER.—See IX.

PATENTS.

Manufacture of titanium-containing compounds. F. G. C. STEPHENS, L. J. ANDERSON, and W. A. CASH

(B.P. 309,051, 1.10.27).—A titanium salt solution is hydrolysed by adding it to warm or boiling water to which has previously been added silicic acid or other colloid (excluding colloidal titanium dioxide) in order to produce a hydrated titanium dioxide that is filterable, although sufficiently fine for use as a pigment. The pigment may be precipitated or subsequently associated with an insoluble alkaline-earth sulphate. S. S. WOOLF.

Production of stable [colour] pastes and lakes. I. G. FARBENIND. A.-G. (F.P. 629,764, 24.2.27. Belg., 2.3.26).—The products comprise zinc lakes and solvents which yield or contain free fatty acids. L. A. COLES.

[Nitrocellulose] lacquers. CARBIDE & CARBON CHEMICALS CORP. (B.P. 289,373, 12.3.28. U.S., 25.4.27).—Nitrocellulose lacquers and thinners for the same containing ethylene glycol monoethyl ether and its acetate in suitable proportions are claimed.

S. S. WOOLF.

Nitrocellulose composition. H. BRADSHAW, E. H. NOLLAU, and R. G. WOODBRIDGE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,703,415, 26.2.29. Appl., 21.12.21. Renewed, 21.3.24).—Nitrocellulose containing more than 12.4% N is dehydrated by displacement with denatured alcohol, the excess of alcohol being expressed under high pressure, and the block is colloided with ether in presence of 0.5% of diphenylamine, comminuted, and seasoned first for 15–25 days at 60° and finally for 4 months or more until the viscosity is less than 200 sec. at 28° by the steel-ball method. The product is mixed with a softener, *e.g.*, castor oil, blown rapeseed oil, butyl phthalate, and a pigment and dissolved in a volatile solvent.

R. BRIGHTMAN.

Driers and their combination with drying oils. H. E. BUC, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,686,484—6, 2.10.28. Appl., [A] 16.9.27, [B, C] 26.9.27).—The oil-soluble sulphonc acids formed by sulphonating petroleum distillates, particularly lubricating distillates, are converted into the corresponding oil-soluble sodium sulphonates. These are extracted by a selective solvent, *e.g.*, 35–65% aqueous alcohol, and freed from oil by treatment with 10–20% of gasoline by vol. The sodium sulphonate, when converted into the corresponding oil-soluble lead (A), cobalt (B), or manganese (C) sulphonates and freed from insoluble inorganic matter by dissolution in an organic solvent, *e.g.*, a mixture of benzene and 75% of ethyl or isopropyl alcohol, affords driers which may be used alone or in combination with other driers.

R. BRIGHTMAN.

Artificial resin and its manufacture. F. SEEBACH, Assr. to BAKELITE GES.M.B.H. (U.S.P. 1,683,701, 11.9.28. Appl., 21.3.27. Ger., 29.3.26).—Fusible resins containing about 4% N and of mol. wt. 370–380, which are soluble in organic solvents, are obtained by the action of formaldehyde on a mixture of 1 mol. of an aromatic amine or a salt or derivative thereof (including, *e.g.*, anthranilic acid, 1-naphthylamine-5-sulphonic acid) with more than 0.5 mol. of phenols. The condensation product is heated alone or in presence of organic acid anhydrides.

R. BRIGHTMAN.

[Artificial] resin and its manufacture. A. E. MAZE (U.S.P. 1,683,835, 11.9.28. Appl., 14.9.26).—

Diacetone alcohol is condensed with 3 mols. of formaldehyde in presence of an alkali or alkaline-earth hydroxide or soluble carbonate or phosphate. R. BRIGHTMAN.

Manufacture of phenol resins. BAKELITE CORP. (B.P. 304,659, 27.10.27. U.S., 11.11.26).—A phenol (2 mols.) is condensed with formaldehyde (more than 2 mols.) in the presence of aniline (1 mol.) and a basic catalyst, *e.g.*, hexamethylenetetramine. The aniline is subsequently expelled (and recovered) under conditions avoiding complete transference of the reactive resin to the "resinoid" state.

S. S. WOOLF.

Manufacture of resin-impregnated sheets. G. E. WIGHTMAN, Assr. to BAKELITE CORP. (U.S.P. 1,703,414, 26.2.29. Appl., 22.6.26).—Fibrous sheets impregnated with reactive and plastic phenolic resin are dried by continuous movement below 40° through a zone of reduced pressure.

R. BRIGHTMAN.

Producing a viscous phenol condensation product. R. GÜNTZEL, Assr. to SCHIEFERWERKE AUSDAUER A.-G. (U.S.P. 1,711,411, 30.4.29. Appl., 8.3.28. Ger., 10.3.27).—See B.P. 286,731; B., 1929, 294.

Impregnation of articles with varnishes of synthetic resins. MANUF. DE MACHINES AUXILIAIRES POUR L'ELECTRICITÉ & L'INDUSTRIE (B.P. 286,305, 28.2.28. Belg., 3.3.27).

Grinding mill (B.P. 306,630).—See I. **Products from waste cracked benzines** (B.P. 309,718).—See II. **Antifouling coatings** (U.S.P. 1,689,008).—See XXIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Reclamation of rubber. J. J. MORIARTY, Assr. to J. H. S. KEER (U.S.P. 1,710,100, 23.4.29. Appl., 8.12.27).—Fibrised, vulcanised rubber is disintegrated, mixed with a plasticiser, and milled; during the last operation a reclaiming agent is introduced. The mixture is then subjected to the action of steam under pressure.

D. F. TWISS.

Manufacture of reinforced rubber. H. H. KELLER (U.S.P. 1,710,481, 23.4.29. Appl., 31.3.27).—Rubber (100 pts.) is mixed with sulphur (3), zinc oxide (26½), extremely fine aluminium oxide (26), and an appropriate quantity of a vulcanisation accelerator.

D. F. TWISS.

Manufacture of reinforced hard rubber. W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,689,570, 30.10.28. Appl., 23.12.26).—Comminuted hard rubber, disintegrated partly-cured rubber, sulphur, cotton, or other fibre, and rubber latex are mixed, dried, moulded, and vulcanised.

R. BRIGHTMAN.

[Accelerators for] manufacture of vulcanised rubber. W. SCOTT, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,688,755—S, 23.10.28. Appl., [A] 17.2.25, [B—D] 8.4.27).—(A) The condensation products of aryl- or diaryl-guanidines and polyphenols, *e.g.*, quinol and its derivatives, give improved ageing properties. Further, 4-dimethylamino-4'-ethoxy-s-diphenylguanidine (B) from *p*-phenetylthiocarbimide and *p*-aminodimethylaniline with subsequent desulphurisation, 4-dimethyl-

amino-s-diphenylguanidine (c), and 4-dimethylamino-2'-methyl-s-diphenylguanidine (d) are used as accelerators.

R. BRIGHTMAN.

Manufacture of rubber surfacing on a rigid base. M. C. TEAGUE, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,705,273, 12.3.29. Appl., 22.4.25).—A concrete base is scored while still moist, allowed to set, and surfaced with a rubber mixture comprising latex, fillers, sulphur, a vulcanising agent, and sulphonated castor oil.

A. R. POWELL.

Fixing a thin layer of sponge rubber to the surface of india-rubber goods. S. MORIMOTO (U.S.P. 1,685,954, 2.10.28. Appl., 14.4.27. Japan, 24.6.26).—A sponge-rubber solution, obtained by mixing, *e.g.*, 600 pts. of unvulcanised india-rubber, 100 pts. of zinc white, 8 pts. of lithopone, 8 pts. of sulphur, 5 pts. of ammonium carbonate, 7 pts. of white substitute, 2 pts. of colouring matter, and 140 pts. of spindle oil, dissolving the mixture in carbon disulphide, and diluting with naphtha, is applied to the surface of unvulcanised rubber goods and the whole is hot-cured.

R. BRIGHTMAN.

Manufacture of plastic and mouldable composition. C. KULAS (U.S.P. 1,688,500, 23.10.28. Appl., 2.1.26).—Plastic material for electrical insulation purposes is obtained, *e.g.*, by adding to the viscous solution of 50 pts. of resol in 50 pts. of alcohol or acetone, 100 pts. of pulverised scrap or waste rubber, or 50 pts. of rubber and 50 pts. of filler or colouring material. The mixture is stirred and gently boiled, the solvent recovered, and the residual mass rapidly calendered to expel volatile constituents, seasoned at about 30–40° for, *e.g.*, 14 days, moulded, and dried at 60–160° at a gradually increasing temperature.

R. BRIGHTMAN.

Bonding of rubber to metal. H. GRAY, Assr. to B. F. GOODRICH CO. (U.S.P. 1,689,628, 30.10.28. Appl., 27.4.27).—The metal surface is coated with sulphur chloride or a solution thereof and placed under pressure in contact with a rubber composition, which is then vulcanised.

R. BRIGHTMAN.

Stabilised latex and its production. M. R. DAY, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,689,581, 30.10.28. Appl., 3.1.27. Renewed 27.3.28).—See B.P. 279,336; B., 1928, 62.

Rubber-covered metal sheets (B.P. 301,300).—See X.

XV.—LEATHER; GLUE.

Water penetration tests for sole leather. F. O. SPRAGUE (J. Amer. Leather Chem. Assoc., 1929, 24, 87–88).—A copper cylinder, 2 ft. high and 2 in. in diam., is filled with water after clamping to a disc of the leather to be tested. At the first drop of water penetrating through the leather, electrical contact is made which stops a clock. The time of penetration varies from a few minutes up to more than one week for samples from different parts of a waterproofed hide. (Cf. Gayley, *ibid.*, 1916, 11, 37.)

D. WOODROFFE.

PATENTS.

Chrome tanning material. C. D. MARLATT, Assr. to INDUSTRIAL WASTE PRODUCTS CORP., and W. H. DICKERSON (U.S.P. 1,698,505, 8.1.29. Appl., 15.4.26).—A dry powder is produced by heating chrome tanning

liquor to 113° and spraying it into a hot gaseous drying medium.

H. S. GARLICK.

Water-soluble product from lignite (B.P. 284,670).—See II.

XVI.—AGRICULTURE.

Significance of water movements in a sandy soil in its management. K. WIESENTHAL (Z. Pflanz. Düng., 1929, 8B, 128–136).—Records of moisture contents at varying depths, and depth of permanent water level in a sandy soil for considerable periods are presented. There is a saturated layer of soil just above the permanent water level, and above this an "intermediate layer" with the lowest mean water content rising to the top soil. The higher mean water content of the top soil is due to its organic matter. The "intermediate layer" is characterised by the widest variations in moisture content, and in periods of drought this loses more water than the top soil. In two sandy soils examined the water supply on the surface layer was mainly dependent on the rainfall, the capillary rise from the subsoil not being sufficiently great to reach the plants during considerable periods. On these soils hoeing resulted neither in increased crops nor in increased water content of the surface soil.

A. G. POLLARD.

Nutrient requirement of soils as indicated by the Mitscherlich and Neubauer methods. H. WIESSMANN and E. SCHRAMM (Z. Pflanz. Düng., 1929, 8B, 105–128).—In the Mitscherlich method comparison of nutrient contents based on the total, straw, and grain yields shows the grain : straw ratio to depend on the potash content of the soil, and crop yields in soils of varying potash deficiency are not strictly comparable. Again, soils deficient in potash suffer by the dilution with sand in these experiments. Differences in nutrient values obtained on a straw basis and on a grain basis increase with potash deficiency, and only in rich soils do these values approximate. In practice nutrient values based on the total crop yields are preferable. Differences in nutrient values for phosphate based on grain and straw yields are less than in the case of potash, and these differences decrease with increased phosphate deficiency. Grain yield values are 16–21% higher than those calculated on straw yields, and show a greater divergency from the corresponding Neubauer values. In the numerous soils examined agreement between Mitscherlich and Neubauer values was very imperfect, although there was a general tendency for relative sufficiency and deficiency to be similarly indicated by both. Mitscherlich values for potash content averaged about one half those by Neubauer's method, and for the phosphate content 47–80% greater. Probably Mitscherlich's growth factor is too high and the potash values correspondingly low. The relatively lower phosphate values shown by Neubauer's method in rich soils is ascribed to the less complete assimilation in this case compared with poor soils. Preliminary heating of the soil (Dirks) leads to poorer results for potash in both methods, and to an improved but by no means good agreement in phosphate values. On the average the poor soils among those examined showed lower p_H values than the richer ones. A. G. POLLARD.

Nitrogen content of Red River Valley soils. J. H. ELLIS and W. SHAFER (Sci. Agric., 1928, 9, 231—248).—The high nitrogen content of these Manitoba soils is established. Dark intrusions of surface material extending into the underlying horizon have a higher nitrogen content than the normal profiles.

CHEMICAL ABSTRACTS.

Soil moisture at permanent wilting of plants. F. J. VEHMEYER and A. H. HENDRICKSON (Plant Physiol., 1928, 3, 355—357).—The quantity of water available for plant growth cannot be determined by the "moisture equivalent" alone. It is necessary to know the residual moisture at permanent wilting for a particular soil.

CHEMICAL ABSTRACTS.

Huminit. DENSCH (Z. Pflanz. Düng., 1929, 8B, 142—147).—Huminit is a finely-powdered preparation from peat or lignite in which the humus acids are neutralised. Its objects appear to be to act as a soil ameliorant, to improve the physical condition, to act as a nitrogen fertiliser, and to provide material inducing greater bacterial activity, notably the fixation of nitrogen. Pot and field trials showed no proof of any effects other than the improved soil texture commonly associated with added organic matter. A. G. POLLARD.

Effects of nitrogen fertilisers. HASELHOFF (Z. Pflanz. Düng., 1929, 8B, 136—142).—General comparisons are made of the common fertilisers. The iodine content of Chile saltpetre has no favourable influence on crop yields, on the grain : straw ratio, or on the weight per 1000 grains. In general, calcium nitrate and sodium nitrate gave similar results, and there was no confirmation of the opinion that the sodium content of the latter improved the yield of root crops. As regards efficiency calcium cyanamide was more dependent on soil characteristics than were ammonium salts or nitrates, but, in general, was rather less efficient. Injury to seedlings often associated with the use of cyanamide was not influenced by the conjoint application of iron or manganese salts. A. G. POLLARD.

Calcium, potassium, and iron balance in certain crop plants in relation to their metabolism. W. F. LOERWING (Plant Physiol., 1928, 3, 261—275).—Two acid muck soils, when treated with calcium carbonate, showed depressed yields of maize and wheat; this result is attributed to potash insufficiency and to reduction of sap acidity. Potash additions were injurious with 3 of 4 acid muck soils. With such soils high in iron content, toxic accumulations of iron may occur in tissues owing to increased sap acidity. In soils low in calcium and magnesium the addition of potassium chloride may depress the lime and magnesia to the starvation point. High yields in young grain plants were associated with high carbohydrate and organic nitrogen content, and low yields with low protein, low carbohydrate, and high nitrate content.

CHEMICAL ABSTRACTS.

Lime penetration resulting from surface application to pasture land. P. R. NELSON (Soil Sci., 1929, 27, 143—146).—The greater part of the lime applied to pastures remains at the surface, but there is a gradual and slight increase in p_H value of the soil to a depth of

9 in. Among mineral fertilisers, gypsum alone showed a slight tendency to neutralise soil acidity.

A. G. POLLARD.

Effects of crude petroleum on nitrate production, seed germination, and growth. H. F. MURPHY (Soil Sci., 1929, 27, 117—120).—Application of crude petroleum to the soil surface considerably reduced nitrate production. The effect was intensified when the oil was mixed with the soil. 1% of oil mixed with soil practically inhibited nitrification. Germination of seed was seriously reduced by treatment of the soil with petroleum.

A. G. POLLARD.

Influence of potash manuring on the hectolitre-weight of cereals. A. JACOB (Z. Pflanz. Düng., 1929, 8B, 61—76).—Numerous results of field trials show that potash manuring results in a definitely higher volume-weight of cereal grains.

A. G. POLLARD.

Nitrogen contents of weeds and their relation to soil fertility. H. J. HARPER and H. F. MURPHY (Proc. Oklahoma Acad. Sci., Univ. Oklahoma Bull., 1928, [ii], No. 410; Studies Series 30, 73—77).—Weeds transfer to the soil large amounts of organic and nitrogenous substances. The nitrogen content (usually 1.0—2.5%) of the following plants was determined: sunflower, ragweed, rosin weed, coreopsis, wild lettuce, cocklebur, crab grass, black-eyed Susan, water-hemp, Russian thistle, Bermuda grass, oat and wheat straw (lowest, 0.519%), soya beans, peas (2.36—3.24%), hairy vetch (highest, 2.67—4.1%), sweet clover, cotton burrs, sesbania, and rye.

CHEMICAL ABSTRACTS.

Analysis of insecticides. Liquid insecticides immiscible with water. Hydrocarbon mixtures, carbon tetrachloride, nitrobenzene, naphthalene, methyl salicylate. M. FRANÇOIS and L. SEGUIN (Ann. Falsif., 1929, 22, 226—232).—See B., 1929, 222.

Rhode Island soils. B. L. HARTWELL and J. B. SMITH (Rhode Island Agric. Exp. Sta. Bull., 1928, No. 214, 24 pp.).

Potential fertility of Oklahoma soils. H. J. HARPER and H. F. MURPHY (Proc. Oklahoma Acad. Sci., Univ. Oklahoma Bull., [ii], No. 409, 1928, 7, 138—141).

Soils of the Punjab. P. E. LANDER, R. NARAIN, and M. M. LAL (Mem. Dept. Agric. India [Chem.], 1929, 10, 25—142).

Determination of phosphoric acid in fertilisers. JORGENSEN.—See VII.

PATENTS.

Destruction of cacti. H. STOLTZENBERG (U.S.P. 1,686,582, 9.10.28. Appl., 20.10.25).—Cacti are subjected to the action of aromatic arsenic compounds, e.g., phenyl- and diphenyl-arsine oxide, chloride, or cyanide, phenarsazine chloride, etc., either as vapours or sprays or as injections of 5% solutions in cresol or sulphuric acid.

R. BRIGHTMAN.

Products from waste cracked benzines (B.P. 309,718). Nitrogenous bases (U.S.P. 1,686,136).—See II. Germicidal coatings (U.S.P. 1,689,008).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Coagulation of colloids from beet sugar liquors.

E. GUNDERMANN (Chem.-Ztg., 1929, 53, 305—307, 322—323).—A résumé of the literature is given. The velocity of coagulation by addition of acids reaches a maximum at p_H 3·2, and then diminishes with increasing acidity; it increases with rising temperature. The coagulated sediment goes into solution again on shaking and again separates on keeping; it is only partially retained by filter paper. The proportion of sediment is also at a maximum at p_H 3·2; relatively more is obtained from more dilute solutions than from stronger solutions. The colour of the solution improves with coagulation, the improvement being greatest at p_H 3·2; further addition of acid results in a darker colour.

S. I. LEVY.

Solid molasses. STAIGER and GLAUBITZ (Z. Spiritusind., 1929, 52, 163—164).—A sample of raw molasses in the solid form prepared by the aid of the heat of the sun contained 8·22% of moisture and 0·32% N, and for the neutralisation of 20 g. required 0·6 c.c. of *N*-sodium hydroxide. There were also present substances which reduced Fehling's solution but were not fermentable. The yield of alcohol varied from 31·75 to 34·25 c.c. per 100 g. of molasses according to the race of yeast used for the fermentation. Sterilised wort with added molasses showed a strong development of *Bacillus subtilis*, butyric and lactic acid bacteria, and streptococci, whilst a gelatin plate culture yielded over 1000 colonies of red yeast, fungi, torulæ, etc.

C. RANKEN.

Determination of starch syrup and dextrose in presence of sucrose and invert sugar.

C. I. KRUISHEER (Chem. Weekblad, 1929, 26, 254—263. Cf. B., 1926, 963; also Schoorl, B., 1929, 336).—A number of formulæ are given, from which the composition of mixtures can be calculated after the determination of six values, viz., original reducing power and lævulose content, R_1 and F_1 , the same after partial inversion with warm dilute hydrochloric acid, R_2 and F_2 , and again after complete inversion at 100° with hydrochloric acid, R_3 and F_3 . A large number of control analyses on mixtures specially prepared, and of analyses of jams, sweets, and chocolates is given.

S. I. LEVY.

PATENTS.

Improvement of crude beet juice. A. GRÄNTZ-DÖRFFER (G.P. 452,436, 22.1.25).—The juice after treatment with lime is subjected to the action of an electric current, using non-reacting electrodes brought into direct contact with the juice, which may simultaneously be treated with air, with alkalis, e.g., lime, or with carbon dioxide, sulphur dioxide, etc.

L. A. COLES.

Rotary crystallisers. Separating mother-liquor from crystals (B.P. 286,599).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Drying of hops. Institute of Brewing Research Scheme. Report of the seventh season's work at the experimental oast, 1927. A. H. BURGESS (J. Inst. Brew., 1929, 35, 235—246).—The depth of loading, air speed, and time of drying are related in the

following manner: $T = 87 \cdot 3L/a^{1 \cdot 047} + M$, where T is the time of drying in min., L the loss of water per sq. ft. of kiln floor in oz., a the air speed in ft. per min., and M is the minimum time depending mainly on the temperature employed and slightly on the air speed. Hops dried at 40° have a superior preservative value to those dried at higher temperature, the amount of α -acid being reduced at the higher temperatures. Hops dried in nitrogen have a higher preservative value than those dried in air, whilst the use of sulphur improves the aroma and colour. The amount of sulphur retained by the hops is greater if the hops are wet when the sulphur is burned. The moisture content of hops varies at different depths of the load during drying in the intermittent type of kiln, and to avoid overdrying the lowest hops and underdrying the top hops a continuous type of kiln should be used.

C. RANKEN.

Preservative principles of hops. X. Modification of Ford and Tait's gravimetric process for the evaluation of hops.

J. J. II. HASTINGS and T. K. WALKER (J. Inst. Brew., 1929, 35, 229—233; cf. B., 1928, 229).—10 g. of freshly-minced hops are either shaken vigorously or mechanically stirred for 10 min. with 100 c.c. of methyl alcohol in a wide-necked bottle of 250 c.c. capacity. The extracted hops are allowed to settle and the supernatant liquid is decanted on to a fluted filter paper. The percentage of α -soft resin in the alcoholic extract of the hops is determined by precipitation with a 1% solution of lead acetate in methyl alcohol, and the β -soft resin is determined by extraction of the filtrate from the α -lead salt with light petroleum, subsequent to dilution with twice its volume of water. Alternatively, the total soft resin can be determined by extracting a further portion of the alcoholic extract of the hops, and the amount of β -resin calculated by subtracting from the total soft resins the value found for the α -resin. It is emphasised that the precipitation of the α -lead salt should be carried out at 60°.

C. RANKEN.

Interstitial liquid and [yeast] cell moisture.

N. C. BEETLESTONE (J. Inst. Brew., 1929, 35, 260—263; cf. B., 1928, 170).—The author reviews the criticisms of his proofs that the yeast cell contains approx. 54% of moisture, and points out that the theory that the moisture content of the cell is 70% depends experimentally on results obtained from cells which are not normal, but which have been distended by the entry of interstitial liquid during pressing. The results should only be taken for yeast cake formed by drainage, where the cake consists of spheres grouped in the hexagonal form of packing. Since the amount of space between such spheres is approx. 27% of the total volume, it follows that the interstitial liquid is 25% of the weight of the yeast cake, and that the remaining 75% of the weight of the cake consists of the cells. From this latter value, and taking the calculated minimum moisture of a cake as 69%, it is calculated that the maximum cell moisture is 59% approx.

C. RANKEN.

Reproduction of yeast in open, loosely covered, and closed fermentation vessels. E. LÜHDER and W. KILP (Z. Spiritusind., 1929, 52, 160—162).—With equal seeding rates, the reproduction of yeast in wort

varied according to the type of fermentation vessel. The reproduction, which was greatest in open vessels, diminished if the vessel was loosely covered, and was lowest when the vessel was completely shut off from the air by a water-seal. With the seeding rate of 5 g. of yeast per litre of wort, the reproduction at the end of 72 hrs. with the open vessel was 6.68-fold, with the loosely closed type 6.10-fold, and with the completely closed vessel 5.97-fold. With a seeding rate of 0.5 g. per litre, the corresponding values were 45.90, 29.88, and 23.12. The loss by evaporation during fermentation in the open vessels amounted to $\frac{1}{3}$ of the original volume of the liquid. With the partly-closed vessel $\frac{1}{10}$ of the original volume was lost, whilst with the closed type the volume remained constant. The evaporation was accompanied by a loss in alcohol, and to this deficiency with its favouring effect on yeast growth was partly attributed the greater yeast reproduction in open vessels. If the volumes of the fermenting liquids were retained constant by the addition of water during fermentation, the relative reproduction rates in the three types of vessel were again as those given above. C. RANKEN.

Heating of the vintage. J. DUBAQUIÉ (Ann. Falsif., 1929, 22, 211—213).—The whole grapes ($\frac{1}{10}$ — $\frac{1}{2}$ of the total vintage) are placed in a hermetically sealed container kept at 40—50° for several hours. Conditions favourable for intercellular fermentation are set up, and the temperature favours the action of the acids on the pectoses. An intense and very desirable odour is developed and the must appears "fatter" and sweeter than usual. The heated product, which is sterile, is added to the vats after the first fermentation. The simplicity and beneficial results are lost if crushed grapes are heated. D. G. HEWER.

Determination of corrected volatile acidity of wines. R. MARCILLE (Ann. Falsif., 1929, 22, 224—225; cf. B., 1922, 911 A).—Mathieu's modification of Sadler's method (Compt. rend. du 6^e Congrès de Chimie industrielle, p. 670) for determination of volatile acidity in sulphited wines needs modification when a high proportion of aldehydes is present, since the aldehyde may combine with the total sulphuric anhydride, thus causing the two determinations to take place under similar conditions. It is necessary to determine free and total anhydride, the difference giving the combined acidity. The total volatile acidity and that of the free sulphuric anhydride, and 70% of the combined anhydride after being expressed in terms of sulphuric acid, are deducted from the total to give the corrected volatile acidity. To accomplish this the free anhydride (g./litre) is multiplied by 1.55 and the combined anhydride by 1.08 and the results are added.

D. G. HEWER.

"Bacteriophage," a new antiferment with formic acid base. R. MARCILLE (Ann. Falsif., 1929, 21, 19—22).—A sample of the Belgian product had total acidity (as sulphuric acid) 190 g./litre, fixed acidity (Roos' method) 41 g./litre, and yielded a residue (per 100 c.c.) of 10.4 g. (syrupy liquid) at 105°, of 6.6 g. at 120°, and of 3.73 g. (black, viscous mass) at 130°. Fractional distillation, which could not be completed, gave 60% of a liquid of acidity 92 g. per litre; the mol. wt. of the

acid derived from the sodium salt approximated to that of formic acid. As an antiseptic for wort the product was very active, but should be used in doses 4 or 5 times that recommended by the makers. About 140 g. of formic acid per litre are present. Formic acid added in efficacious doses to wines would cause deterioration, augment their volatile acidity, and might be injurious to the health of the consumer. D. G. HEWER.

Solid molasses. STAIGER and GLAUBITZ.—See XVII. **Essential oil of hops.** CHAPMAN.—See XX.

PATENTS.

Test reagents for determining alcohol and colour [added caramel] in alcoholic liquids. J. F. WILLIAMS, Assr. to C. J. ROBERTS and L. C. JACKSON (U.S.P. 1,689,901—2, 30.10.28. Appl., [A] 30.7.26, [B] 2.8.26).—(B) 70 pts. of amyl alcohol or refined fusel oil (d 0.8103—0.8119, b.p. 110—130°), 28 pts. of toluene (or xylene), and 2 pts. of tartaric acid, acetic acid, or other soluble acid are mixed to give a reagent of d_{20}^{25} 0.8333. (A) This reagent when added to whiskey etc. extracts alcohol and natural colouring matter. The decrease in volume of the whiskey shows by reference to a curve (given) the percentage of alcohol originally present; added colouring matter (caramel) is shown by the colour of the extracted samples. R. BRIGHTMAN.

Distillation of liquids. Apparatus for recovering alcohol from vapours. A. FREYMAN (B.P. [A] 310,400 and [B] 310,403, 25.1.28).—(A) Vapour generated by heating in a still or oven is conducted to a dephlegmator, and is thence passed into a cooler. The vapour on its way to the condenser is used to heat vessels or containers charged with condensate from the condenser for the distillation of volatile matters from such condensate. (B) Vapour containing alcohol, e.g., the vapour issuing from a baker's oven, prior to passing to a cooler where the more volatile constituents collect, is conducted to a condenser which is kept at a predetermined temperature by water or liquid the b.p. of which corresponds to the maximum temperature at which the required condensation takes place. C. RANKEN.

Apparatus or mills for treating or grinding malt. G. PORTEUS (B.P. 311,074, 21.4.28).

XIX.—FOODS.

Electrolytic conductivity of aqueous extracts of flour. K. SCHMORL (Z. ges. Mühlenwesen, 1928, 5, 62—65, 88—90, 126—127; Chem. Zentr., 1928, ii, 2299).—Of the water-soluble organic constituents, chiefly the nitrogenous substances affect the conductivity. With the degree of grinding the mineral matter and nitrogen increase regularly. The use of conductivity determinations in control is discussed.

A. A. ELDRIDGE.

Effect of lecithin in dairy products on butter fat determinations. O. W. CHAPMAN (J. Dairy Sci., 1928, 11, 429—435).—Lecithin contents (average) are reported as follows: milk 0.0447, cream 0.1981, skim milk 0.0165, buttermilk 0.1302%. The fat found in buttermilk contains 13% of lecithin.

CHEMICAL ABSTRACTS.

Lactometer as used to determine solids-not-fat in milk. C. F. HOYT, N. C. SMITH, L. M. LAMPERT, and L. G. SAYWELL (Calif. Dep. Agric. Bull., 1928, 17, 594—603).—The calibrated lactometer reading should be made at the top of the meniscus. Unless the prior treatment of a sample is known, the values are subject to uncertainty. Average values for samples of maximum sp. gr. (using the Babcock formula) agree fairly well, and those for samples in which the original sp. gr. has been restored by heating (using the formula $S - F = (L/4) + (L/5) + 0.2$, where $S - F$ = solids-not-fat, L = lactometer reading, and F = fat %) agree well with gravimetric results. CHEMICAL ABSTRACTS.

Connective tissue content of beef muscle. H. H. MITCHELL, T. S. HAMILTON, and W. T. HAINES (J. Nutrition, 1928, 1, 165—178).—Methods for the determination of elastin and collagen are described. The distribution of elastin and collagen in various "cuts" of meat has been examined. Age probably does not greatly influence the content of connective tissue in muscle. CHEMICAL ABSTRACTS.

Proximate composition of fresh fruits. C. CHATFIELD and L. McLAUGHLIN (U.S. Dep. Agric. Circ., 1928, No. 50, 1—19).—Data for 65 fresh fruits or their products are tabulated. CHEMICAL ABSTRACTS.

Hydrogen swelling of canned ready-to-serve prunes. Effect of blanching. E. M. MRAK and P. H. RICHERT (Fruit Prod. J., 1929, 8, 11—13, 14—15, 15).—The rate of spoilage under various conditions of canning has been studied. Least corrosion occurred when the amount of oxygen originally present was greatest. Blanching increases the rate of formation of hydrogen. CHEMICAL ABSTRACTS.

Flour quality: its nature and control. E. A. FISHER (Nat. Ind. Council for Flour-Milling Ind., Pamphlet No. 3, 56 pp.).

Separation of the components of frozen milk by fractional melting. V. V. WINTER (Chem. News, 1929, 138, 321—324).—See B., 1928, 108.

Adulterants of cacao butter. BODENDORF.—See XII. **Determination of mixed sugars.** KRUISHEER.—See XVII.

PATENTS.

Composition for bleaching. Bleaching of food-stuffs. W. B. STODDARD and V. R. KOKATNUR, ASSTS. to PILOT LABORATORY, INC. (U.S.P. 1,687,803—5, 16.10.28. Appl., [A] 8.3.27, [B] 7.6.27, [C] 2.9.27. Renewed [A] 29.12.27, [B] 19.12.27).—(A) Acids obtained from natural fats and oils are converted into their mixed chlorides, and the latter, by treatment with hydrogen peroxides and alkali, into acid peroxides, which are used for bleaching foodstuffs, e.g., by incorporation with the material and keeping them at ordinary temperatures. (B) Inactive or relatively inactive organic peroxides are activated by addition of small amounts (1—10%) of active organic peroxides. Suitable bleaching agents are obtained by the action of alkaline hydrogen peroxide at -5° to 0° on a mixture of 2 mols. of benzoyl chloride, 1 mol. of fumaryl chloride, and 12.5% of a fatty acid chloride. By incorporation of 5—10% of the "active"

peroxide with the "inactive" peroxide a product may be obtained which is 2—6 times as effective in bleaching power as the "activating" peroxide alone. (C) The material to be bleached is treated with a peroxidised compound, particularly peroxidised food material, which is constitutionally similar to the vehicle of the colouring matter present and physically similar to the colouring matter. When decomposed the peroxidised compound leaves a residue possessing food value.

R. BRIGHTMAN.

Manufacture of wheaten flour. R. W. DUNHAM (B.P. 311,034, 5.3.28).—The flour is matured by repeatedly subjecting it to alternate periods of light and comparative heat, and darkness and comparative cold, so as to continue the natural ripening occurring in the field. The material may be passed along under electric lamps heated to a red glow so that its temperature does not exceed 60° and preferably is slightly below 38° . It is then passed into a dark chamber at $10-16^{\circ}$.

W. J. BOYD.

Treatment of flour and the like. J. A. HALL and W. F. C. GEORGE (B.P. 310,125, 18.2. and 12.7.28).—In a series of three interconnected tubes or ducts, an improver, to be added to untreated flour, is prepared by subjecting flour, while it is being agitated, to hot steam-laden air whereby it is maintained at $77-93^{\circ}$ for 5—20 min., then allowing it to rest until the heat is distributed uniformly through it, and finally agitating it in the presence of cool air.

W. J. BOYD.

Preservation of grain, flour, and bran of cereals. R. A. LEGENDRE (B.P. 310,980, 5.11.27).—The material is treated in such a manner that the pH value of its diastase cells is increased to above 7. Preferably at least one salt of a weak acid with a strong base is used, or at least one free base. The alkaline material may be applied (a) in solution by spraying, (b) as a solid by mixing it with the cereal, or (c) as a gas, e.g., ammonia.

W. J. BOYD.

Production of metal glutins. K. KÜTTEL, ASSRS. to E. J. WATZL and H. J. TRENKAMP (U.S.P. 1,686,281, 2.10.28. Appl., 7.8.25).—A gluten in aqueous solution (20%) is treated at 28° with the equivalent amount of alkali hydroxide to produce a saturated alkali gluten, and an excess of an insoluble glutinate-forming salt, e.g., aluminium or zinc sulphate, is then added, the product thus containing the anion of the precipitating salt.

R. BRIGHTMAN.

Sterilisation of double cream. S. GRASSE (B.P. 309,932, 18.1.28).—The cream, preheated to about 37° , is maintained in a state of gentle agitation and passed under pressure through a continuous and closed conduit in which it is first heated to about 118° and then cooled to ordinary temperature. A suitable apparatus is described.

W. J. BOYD.

Cheeses and their preparation. PHENIX CHEESE CORP. (B.P. 300,113, 27.2.28. U.S., 6.11.27).—A mixture of cheese and dairy products is melted at 74° and emulsified by stirring, with or without addition of an emulsifier such as sodium citrate. The mass is then "viscolised" to impart a smooth texture and fluffy appearance, to blend the ingredients, and to stabilise the emulsion.

W. J. BOYD.

Manufacture of cheese products. SKINNER MANUF. CO., Assecs. of L. M. SKINNER and J. L. HEID (B.P. 297,025, 24.1.28. U.S., 12.9.27).—To a mixture of flour and powdered cheese water is added to form a dough which is kneaded, pressed into the form of macaroni, and dried in air first at 32° and then at normal temperature.

H. ROYAL-DAWSON.

Preparation of vegetable food. E. H. MILES and G. REILLY (B.P. 310,788, 31.1.28. Addn. to B.P. 256,765. Cf. B.P. 274,051; B., 1928, 425).—The juices of preservative fruits (grapes, apples) are mixed with the juices of fruits less rich in preservative factors (lemons, grape fruit), the mixture is heated at not above 80° until hydrolysis of the sucrose is well advanced, and the whole is concentrated rapidly *in vacuo* at low temperature to a thick syrup. The press-cakes of both classes of fruit may be treated with an acid, preferably an acid fruit juice, to extract further calcium and other salts, and the liquid then expressed and added to the other extracts before heating and concentration of the latter. The insoluble outer part of the orange peel may be added, and the essential oil of lemon rind may be expressed and used as a flavouring agent.

W. J. BOYD.

Removal of residual poisons from and the preservation of fruits. R. H. ROBINSON and H. HARTMAN, Assrs. to U.S.A. (U.S.P. 1,708,330, 9.4.29. Appl., 19.5.27).—The fruit is treated with an aqueous solution of hydrogen chloride and formaldehyde.

L. A. COLES.

Removal of substances producing turbidity from pectin solutions [e.g., fruit juice]. POMOSIN-WERKE GES.M.B.H. (B.P. 284,273, 26.1.28. Ger., 26.1.27).—The solutions are cooled until freezing commences, and the precipitate and ice formed are removed by filtration or other means.

W. J. BOYD.

Treatment of cannery waste. J. T. TRAVERS (U.S.P. 1,711,105, 30.4.29. Appl., 15.11.27).—To the waste, rendered alkaline, are added a soluble calcium salt and a coagulating agent containing a primary phosphate of calcium.

W. J. BOYD.

Treating and drying fish. W. H. HASLAM (B.P. 310,765, 26.1.28).—Fish or other animal or vegetable material is dried in an uninterrupted current of air, the temperature rising periodically during the process according to the temperature required to dry the material. The velocity of the air is approx. 1000 ft./min. at 27°, being increased 1000 ft./min. for every rise of 5½°. The temperature may vary from 21° to about 77°. A dry, uncooked, sterilised fish powder can thus be obtained in 12 hrs.

W. J. BOYD.

Kneading machines (B.P. 302,152).—See I. Alcohol from vapours (B.P. 310,400 and 310,403).—See XVIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Decomposition of acetylsalicylic acid in alkaline solution. D. B. DORT (Pharm. J., 1929, 122, 302).—Contrary to the U.S. and British pharmacopœias, no decomposition into acetic and salicylic acids occurs when the drug is dissolved in sodium carbonate or bicarbonate solutions.

S. I. LEVY.

Bacterial content of certain medicaments. J. P. TODD and (Miss) H. M. SMITH (Pharm. J., 1929, 122, 301—302).—A number of chemicals commonly employed in pharmaceutical preparations were examined; all were found to be sterile except glucose and gum arabic.

S. I. LEVY.

Evaluation of materials in the production of essential oils. V. VARENZOV (Trans. Sci. Chem.-pharm. Inst., Moscow, 1928, 203—208; Chem. Zentr., 1928, ii, 2516).—Evaluation of the total surface of fruits in various size-groups gives results in accord with the relative yields of products.

A. A. ELDRIDGE.

Separation of citronellal from essential oils. J. ZIMMERMANN (Pharm. Tijdschr. Ned.-Indie, 1928, 5, 293—295; Chem. Zentr., 1928, ii, 2601—2602).—Separation by sodium hydrogen sulphite is described in detail.

A. A. ELDRIDGE.

West Australian sandalwood oil. I. A. R. PENFOLD (J. Proc. Roy. Soc. N. S. Wales, 1928, 62, 60—71).—An examination of commercial samples of West Australian and East Indian sandalwood oils shows that a considerable difference exists between the alcoholic constituents of each class of oil. Each contains about 70% of alcohols. The West Australian oil contains 40—45% of santalol (allophanate, m.p. 162—163°) and a mixture of isomeric sesquiterpene alcohols ("fusanols") having b.p. 160—161°/4—5 mm., d_{20}^{20} 0.942—0.943, α_D^{20} $\pm 5^\circ$, and n_D^{20} 1.5030, together with a small quantity (less than 10%) of a dextrorotatory alcohol. The characteristics of 9 oils are tabulated. Oil from the wood of *Santalum lanceolatum* gave 70% of an alcohol having b.p. 163—165°/5 mm., d_{20}^{20} 0.9474, α_D^{20} -66.7° , n_D^{20} 1.5074 (allophanate, m.p. 114°), and a formula approximating to $C_{15}H_{24}O$. Australian oils may be distinguished from East Indian oils by the violet-red colour produced when an acetic acid solution of the oil is treated with bromine vapour. A modified potassium permanganate oxidation process which gives increased yields of santalenic acid and freedom from tarry products is described (cf. May, B., 1928, 426).

E. H. SHARPLES.

Hungarian essential oils. M. FÖLSCH (Riechstoffind., 1928, 3, 123—125, 141—142, 162—163, 180—181; Chem. Zentr., 1928, ii, 2295).—The production of the following oils is discussed: acacia blossom, dill, fennel, camomile, coriander, spearmint, and lavender.

A. A. ELDRIDGE.

Norwegian juniper oil. I. A. JERMSTAD (Riechstoffind., 1928, 3, 159—160, 178—179; Chem. Zentr., 1928, ii, 2296).—The juniper oil had d_{20}^{20} 0.8684—0.8775, n_D^{20} 1.4729—1.4752, α_D^{20} (200 mm.) $+34.05$ — 41.0° , acid value 0.33—0.60, ester value 2.50—8.56, saponif. value 3.0—8.88, acetyl value 20.43—30.40, n_D^{20} of acetylated oil 1.4724—1.4747. Juniper-berry oil had d_{20}^{20} 0.860—0.882, n_D^{20} 1.479—1.484, α_D^{20} (100 mm.) -1° to -15° , acid value 0—3, ester value 1—8. The juniper oil contained 11% of Δ^1 -menthen-4-ol.

A. A. ELDRIDGE.

Composition of pine-needle oil from Crimean *Pinus halepensis*, Mill. B. RUTOVSKI and I. VINOGRADOVA (Trans. Sci. Chem.-pharm. Inst., Moscow, 1928, 109—114; Chem. Zentr., 1928, ii, 2516).—The

oil had d_{20}^{20} 0.8960, α_D -49.44° , n_D^{20} 1.4940, acid value 1.13, ester value 18.8 (after acetylation, 54.83); it contained α -pinene (*l*- and *r*-) 10%, camphene 6%, tertiary alcohols 15%, and sesquiterpenes (apparently a mixture of isomeric caryophyllenes) 8%, and a less volatile compound (50%) which had a high optical activity. Borneol and phenylethyl alcohol were absent.

A. A. ELDRIDGE.

Caucasian *Thuja* oils. B. RUTOVSKI and K. GUSSEVA (Trans. Sci. Chem.-pharm. Inst., Moscow, 1928, 123—143; Chem. Zentr., 1928, ii, 2413—2414).—Oil of *Thuja occidentalis* had d_{20}^{20} 0.9203, α_D -7.58° , n_D^{20} 1.4605, acid value 1.05, ester value 25.46 (after acetylation 39.43); α -pinene, α -thujone, fenchone, and probably borneol and its esters were present. Oil of *Thuja varreana* had d_{20}^{20} 0.9078, α_D -1.23° , n_D^{20} 1.4550, acid value 1.5, ester value 16.36 (after acetylation 30.36); sabinene, thujone, and thujyl alcohol were present. Oil of *Thuja gigantea*, var. *semperaurea*, had d_{20}^{20} 0.9145, α_D -1.21° , n_D^{20} 1.4552, acid value 2.34, ester value 26 (after acetylation 47.15); α -pinene, thujone, and thujyl alcohol were detected, the presence of sabinene being assumed.

A. A. ELDRIDGE.

Essential oils of wild plants of Voronezh Government. A. TSCHERNUCHIN (Trans. Sci. Chem.-pharm. Inst., Moscow, 1928, 196—202; Chem. Zentr., 1928, ii, 2413).—The following values refer, respectively, to the oils of *Thymus odoratissimus*, *Achillea millefolium*, *Mentha aquatica*, *Acorus calamus*, and *Hyssopus cretaceus*: d_{15}^{15} 0.8682, 0.904, 0.9625, 0.960, 0.9122; α_D $+15.22^\circ$, $-$, $+28.2^\circ$, $+12.0^\circ$, -24.29° ; n 1.4755, $-$, 1.4865, 1.5033, 1.4743; acid value 0, 2.2, 6.8, $-$, 4.1; saponif. value 23.0, 26.1, 54.2, $-$, 48.2; ester value 23.0, 23.9, 47.4, $-$, 44.1; ester value after acetylation 50.2, 81.0, 91.5, $-$, 88.0.

A. A. ELDRIDGE.

Preparation of menthol from Ukrainian peppermint oil. M. EIDERMAN (Pharm. J. Russia, 1928, 35—37; Chem. Zentr., 1928, ii, 2355).—The fraction of b.p. 88—90°/8—10 mm. contains 80% of menthol, which is purified by cooling, separation from oil, and recrystallisation from petroleum.

A. A. ELDRIDGE.

Essential oil from the leaves of *Rhus cotinus*. B. RUTOVSKI and N. PROKOPTSCHUK (Riechstoffind., 1928, 3, 172—173; Chem. Zentr., 1928, ii, 2295—2296).—The leaves of *Rhus cotinus* yielded an oil (fresh 0.07—0.19%, dried leaves 0.134—0.207%) having d_{20}^{20} 0.843, α_D $+5.25^\circ$, n_D^{20} 1.4825, acid value 0.66, ester value 20.1, ester value after acetylation 27.2. Aldehydes and other compounds containing oxygen were not present. The fraction of b.p. 155—184°/13—15 mm. contained α -pinene (10—11%), camphene (8—9%), and a hydrocarbon (50%), $C_{10}H_{16}$, d 0.8209—0.824, probably myrcene, having three double linkings and polymerising readily.

A. A. ELDRIDGE.

Essential oil from a *Boronia* in the *pinnata* section, from Frazer Island, Queensland. A. R. PENFOLD (J. Proc. Roy. Soc. N.S. Wales, 1929, 62, 225—234).—Essential oils obtained by steam-distillation of leaves and terminal branchlets of the following plants are described. *Boronia thujona*, var. "A," from Frazer Island: yield 0.53—0.62%, d_{15}^{15} 1.0563—1.0565, α_D^{20}

-8.6° to -11.2° , n_D^{20} 1.5255—1.5260, ester value 6.3—10.8, ester value after acetylation 20.5—22.1, and solubility in 80% alcohol (by wt.) 1 in 5½—9 vols. The principal constituents are safrole (75—80%) and *l*-limonene, with small quantities of phenolic substances, sesquiterpene, and a paraffin, m.p. 65—66°. *B. thujona* (Penfold and Welch): yield 0.5—0.8%, d_{15}^{15} 0.9121—0.9152, α_D^{20} $+12^\circ$ to -56.54° , n_D^{20} 1.4526—1.4543. The oil contains α - and β -thujone (80—90%), sesquiterpene, and paraffin. *B. Muellieri* (Cheel) yields 0.38—0.57% of oil having d_{15}^{15} 1.0197—1.0265, α_D^{20} $+1.5$ — 3.8° , n_D^{20} 1.5125—1.5150, and containing elemicin (70—90%), *d*- α -pinene, geraniol, and geranyl acetate. *B. pinnata* (Smith) yields 0.02—0.1% of oil having d_{15}^{15} 0.8784—0.8917, α_D^{20} -4.7° to -15.25° , n_D^{20} 1.4762—1.4825, and containing limonene, *d*- α -pinene, sesquiterpene, and a paraffin.

E. H. SHARPLES.

Essential oil of a new species of anemone leaf *Boronia* rich in ocimene. A. R. PENFOLD (J. Proc. Roy. Soc. N.S. Wales, 1929, 62, 263—272).—Steam-distillation of the leaves and terminal branchlets of *Boronia dentigeroides* yielded 1.3—2% of pale, mobile oil with a pleasant characteristic odour. The oil has d_{15}^{15} 0.8421—0.8466, α_D^{20} $+10.2^\circ$ to $+16.22^\circ$, n_D^{20} 1.4778—1.4793, ester value 14.9—34.2, ester value after acetylation 33.2—88.7, solubility in 80% alcohol (by wt.) 1 in 7.5—10 vols., and contains ocimene (75—80%), *d*- α -pinene (less than 30%), *d*-limonene (total terpenes 90%), darwinol, and the corresponding decoate, *iso*-valerate, and acetate, ethyl formate (?) and *iso*-valerate, together with small quantities of sesquiterpenes, phenolic substances, and a paraffin, m.p. 64—66°. The following values for the oil from *B. anemonifolia* are also given: yield 0.6—1%, ester value 54—128, pinene 75%, and ocimene (trace only).

E. H. SHARPLES.

Concrete otto of *Boronia metastigma* (Nees). A. R. PENFOLD (J. Roy. Soc. W. Australia, 1927, 14, 1—5).—Four commercial samples of concrete otto of *B. metastigma* had the following characteristics: d_{15}^{15} 0.8989, d_{15}^{40} 0.9091—0.9189, n_D 1.4752—1.4852, acid value 22.8—30.5, acid value plus ester value 95.9—109.3, ester value after acetylation 136.5—147.8, and m.p. from 35—36° to 40—41°. A large amount of triacontane, m.p. 64°, was present together with a glyceride of palmitic and stearic acids, phytosterols, $[\alpha]_D^{20}$ $+60^\circ$, m.p. 162°, free octoic and palmitic acids, ethyl alcohol, and ethyl formate, unidentified phenolic substances, and the odoriferous constituents consisting of an unidentified alcohol and probably β -ionone.

E. H. SHARPLES.

Occurrence of a number of varieties of *Eucalyptus dives* as determined by chemical analyses of the essential oils. II. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. N.S. Wales, 1928, 62, 72—78; cf. B., 1927, 858).—The following varieties of *E. dives* from Tumbarumba, N.S.W., are described: Type, var. "B" (Mannus Hill), var. "B" (School Hill), and var. "C." Essential oils from them (yield 1.62%, 3.1%, 2.32%, 4.2%) have the following respective constants: d_{15}^{15} 0.9099, 0.9077, 0.8904, 0.9214; α_D^{20} -42.6° , -21.4° , -36.8° , $+4^\circ$; n_D^{20} 1.4817, 1.4686, 1.4721, 1.4627; solubility in 70% alcohol

1.4 vols., 1.4 vols., insol. in 10 vols., 1.1 vols.; piperitone content 52%, 8%, 5%, —; cineole —, 17%, 11%, 58% (all by *o*-cresol method); phellandrene about 40%, abundance, abundance, absent; ester value —, 52.2, 12.7, 12.1; ester value after acetylation —, 100.1, 77.1, 58.8. In using the *o*-cresol method, abnormal percentages of cineole were found with oils of the *E. Australiana* type due to the presence of α -terpineol, and it is suggested that the determination be made on the portion distilling below 190° when applied to oils of this type.

E. H. SHARPLES.

Determination of cineole in eucalyptus oil, with special reference to the crude oil obtained from *Eucalyptus cneorifolia*. P. A. BERRY (Australasian J. Pharm., 1929, 203—206).—The *o*-cresol method gives satisfactory results in the determination of cineole in *E. cneorifolia*, whilst the phosphoric acid method gives very inaccurate results due to the solvent action of the aldehydes on the cineole phosphate. A detailed description of the *o*-cresol method, together with f.p. curve and a review of other analytical methods, are given.

E. H. SHARPLES.

Exudation from the wood of *Pentaspodon Motleyi*. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. N.S. Wales, 1929, 62, 218—224).—The dark brown, viscous exudation from *P. Motleyi* occurring in New Guinea has an odour like that of boiled linseed oil and gives the constants: d 1.01—1.011, n_D^{20} 1.5280—1.5295, acid value 138.24—139.08, saponif. value 142.09—146.64, acid value after acetylation 102.07—102.53, iodine value (Wijs) 192.1, solubility in 70% alcohol (by wt.) 1 in 2.5—2.6 vols. It consists of 90—95% of acid substances and gives a deep violet colour with alcoholic ferric chloride solution. The principal constituent is a dihydroxymonocarboxylic acid, $C_{24}H_{36}O_4$, having d 1.0132, n_D^{20} 1.5270, acid value 145.52, acid value after acetylation 106.5, saponif. value 152.90, saponif. value after acetylation 203.24, iodine value (Wijs) 188.3, and solubility in 70% alcohol 1 in 3.3 vols. The alkaline salts have good emulsifying properties.

E. H. SHARPLES.

Essential oil of hops. A. C. CHAPMAN (J. Inst. Brew., 1929, 35, 247—253; cf. B., 1928, 501).—Myrcene, linalool, geraniol, linalyl isononoate, humulene, luparone, luparenol, and luparol were isolated from hop oil by repeated fractional distillation. Of these constituents the last three were isolated from the higher-boiling fractions of the oil, details of which have already been published (*loc. cit.*)

C. RANKEN.

PATENTS.

Production of codeine from opium. J. BYLINKIN (Russ. P. 3381, 15.9.24).—The solution obtained by adding ammoniacal alcohol or a mixture of acetone and alcohol to an aqueous opium extract and removing the precipitated morphine is distilled to remove the alcohol, treated successively with ammonia to precipitate tarry impurities, with calcium hydroxide to remove meconic acid, and with acidified alcohol to remove final traces of tar, after which the alcohol is removed by distillation and the codeine salt recovered by crystallisation.

L. A. COLES.

Ester of salicylic acid and pyruvic acid [for elimination of uric acid]. S. L. SUMMERS (U.S.P. 1,689,696, 30.10.28. Appl., 30.6.25).—Salicylic acid and 2 mols. of pyruvic acid heated at 160° yield *pyruvylsalicylic acid*, $C_6H_4(O \cdot CO \cdot COMe) \cdot CO_2H$.

R. BRIGHTMAN.

Production of esters of di-iodobehenolic acid. A. WINGLER, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,688,100 and 1,688,169, 16.10.28. Appl., [A, B] 11.5.26. Ger., [A, B] 12.5.25).—(A) *isoAmyl di-iodobehenolate*, m.p. 5—6°, and (B) *isobutyl di-iodobehenolate*, m.p. 14°, are obtained by heating di-iodobehenolic acid with excess of the alcohol and 4% of sulphuric acid at 100°.

R. BRIGHTMAN.

Basic ether of resorcinol. H. HAHN, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,711,020, 30.4.29. Appl., 13.8.27. Ger., 20.9.26).—See B.P. 300,695; B., 1929, 149.

Preparation of arylazodiaminopyridines. A. K. CROAD. From PYRIDIUM CORP. (B.P. 311,349, 10.11.27).—See U.S.P. 1,680,108—1,680,111; B., 1928, 837.

Metallmercapto-acid esters and their manufacture. W. SCHOELLER and H. G. ALLARDT, Assrs. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,689,366, 30.10.28. Appl., 16.12.27. Ger., 16.12.26).—See B.P. 282,427; B., 1929, 302.

Production of unsymmetrical arseno-compounds. J. PFLEGER and A. ALBERT (U.S.P. 1,688,351, 23.10.28. Appl., 29.11.24. Austr., 12.4.23).—B.P. 249,584; B., 1926, 462.

Complex antimony compound. H. HAHN, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,688,964, 23.10.28. Appl., 8.10.26. Ger., 8.6.25).—See B.P. 271,940; B., 1927, 573.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitivity. III. Sensitising action of previous exposure to a dim light. IV. Desensitising action of previous fogging by X-rays. O. MASAKI (Mem. Coll. Sci., Kyoto, 1929, 12, 107—116, 117—126).—III. Faint spectral lines can be successfully developed if the plate is previously exposed to a dim light. It is now shown that with certain plates the density of the image is greater than would result from the sum of the two exposures, and that the fogging therefore increases the sensitivity. Fogging is more effective when made before the main exposure than when made after it. The sensitising action diminishes with increase in the time between the two exposures. The sensitising action is greatest when fogging is produced by red light, and least by violet light. The effect is more marked in panchromatic than in ordinary plates.

IV. The effect of fogging by X-rays on the sensitivity of commercial photographic plates is examined. A fog impression produced by X-rays is not reversed by light, but the inertia of the plate is increased. The decrease in sensitivity is greater when the fogging precedes the main exposure than when it follows it, and is equal for all wave-lengths. The effect is not modified by lapse

of time between the exposures. The change in sensitivity is greatest for very rapid plates. The decrease in sensitivity produced by X-rays is restored by a second fogging by a dim light, and the increase in sensitivity produced by light-fogging (cf. *supra*) is destroyed by subsequent fogging by X-rays. C. J. SMITHELLS.

PATENTS.

Manufacture of photographic silver-salt emulsions. I. G. FARBENIND. A.-G. (B.P. 283,222, 7.1.28. Ger., 7.1.27).—The active sulphur content of gelatins or other proteins to be used for photographic purposes is increased by treatment during manufacture with traces (1 pt. to 500,000 pts. of protein solution) of colloidal sulphur, inorganic sulphides, particularly alkali and alkaline-earth sulphides, or carbon disulphide.

J. W. GLASSETT.

Multicolour screens for colour photography. J. H. CHRISTENSEN (B.P. 309,113, 5.1.28).—Acid dyes are added to a mixture of tannic acid and a basic dye and are thereby strongly held by the insoluble compound formed on drying. Tungstic acid, molybdic acid, and casein may be used to replace tannic acid as the mordant. Suitable acid dyes are brilliant-croceine, sea-blue, patent-blue, erioglaucine, and naphthalene-green.

J. W. GLASSETT.

Production of photo-prints and photo-copies. I. G. FARBENIND. A.-G. (B.P. 289,895, 4.5.28. Ger., 6.5.27. Addn. to B.P. 286,233; B., 1928, 466).—In the process of the prior patent (cf. also B.P. 286,736 and 289,386; B., 1928, 549, 944), a salt of a sulphonic acid derivative of 1:2-naphthaquinone or its halogen, hydroxy-, or other substitution product may be used as the light-sensitive material, but 1:2-naphthaquinone-4-sulphonic acid or its substitution products are excepted.

J. W. GLASSETT.

Production of relief images upon sensitised, continuous film-strips which have been exposed or printed. J. E. THORNTON (B.P. 303,262, 6.12.27).—The exposed films are passed continuously through a series of tanks or sprays in which the unhardened colloid is first removed by hot water or other suitable solvent, leaving a relief image which is then hardened by a solution of chrome alum, formalin, etc., and finally rendered flexible by immersion in dilute solutions of glycerol or sugar. The last two operations may be carried out simultaneously by combining the solutions.

J. W. GLASSETT.

Printing or reproducing photographic films bearing colour records. SOC. CIVILE POUR L'ETUDE DE LA PHOTOGRAPHIE ET DE LA CINÉMATOGRAPHIE EN COULEURS (B.P. 295,313, 26.9.27. Fr., 9.8.27).

XXII.—EXPLOSIVES; MATCHES.

Vapour pressure of nitroglycerin and nitroglycol. A. MARSHALL (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 177). P. NAOUM and K. P. MEYER (*Ibid.*, 177).—The higher values obtained by Naoum and Meyer (B., 1929, 379) as compared with those of Marshall and Peace are explainable by the differences in technique. The former workers determined the amount of nitroglycerin that was evaporated by weighing the condensed nitro-

glycerin, whilst the latter determined it by finding the loss of weight of the mixture of nitroglycerin and guhr when air was drawn over it. The loss of weight would include that due to moisture and volatile matter present in the nitroglycerin. Contrary to Naoum and Meyer's statement, different velocities of air gave the same values for the vapour pressure of nitroglycerin.

Marshall's belief that the higher values obtained by Naoum and Meyer were due to moisture and volatile matter in the materials is unfounded as the nitroglycerin was pure and the guhr was ignited before the determinations were made. His statement that the values for the vapour pressure are independent of the velocity of the air is contrary to the experience of other workers on the determination of vapour pressures by the aspiration method. S. BINNING.

Determination of moisture in smokeless powder. W. TIBELL and G. AHLFELDT (Chem.-Ztg., 1929, 53, 356).—Benesch's method (B., 1927, 716) cannot be employed for the determination of powder made with ether-alcohol instead of acetone-alcohol as solvent; in this case the indirect method yields equations for the three unknowns in which the denominator is zero, and no solution is therefore possible. A. R. POWELL.

PATENTS.

Preparation of explosives. H. C. BUGBIRD (U.S.P. 1,706,871, 26.3.29. Appl., 2.5.25. Renewed 7.8.28).—Granular carbonised lignin residues are leached to extract water-soluble mineral substances and then ground to such a degree that the apparent sp. gr. is at least 0.2 and so that the amount of liquid oxygen that can be absorbed is at least 2.6 times the weight of the carbon.

A. R. POWELL.

Blasting explosive. L. N. BENT, Assr. to HERCULES POWDER CO. (U.S.P. 1,706,517, 26.3.29. Appl., 26.7.27).—A mixture of 25—80% of ammonium nitrate, 0—25% of sodium nitrate, 10% of nitroglycerol, and 1—18% of wood which has been disintegrated by saturation with high-pressure steam at a high temperature followed by sudden release of the pressure is claimed.

A. R. POWELL.

Explosive composition. E. SORENSON (U.S.P. 1,709,498, 16.4.29. Appl., 17.9.27).—A combination of ammonium and potassium nitrates, sulphur, manganese dioxide, paraffin wax, petroleum naphtha, rosin, potato flour, aluminium, and nitroglycerin is claimed.

H. ROYAL-DAWSON.

Tracer compositions. S. ST. P. MEEK (U.S.P. 1,708,186—7, 9.4.29. Appl., [A] 21.5.24, [B] 18.1.26).—(A) The composition includes an oxygen-containing compound of an alkaline-earth metal, salts of a rare-earth metal, a fuel, and a binder. (B) The mixture contains magnesium, a metal resinate, a zinc compound, and a suitable oxygen carrier. H. ROYAL-DAWSON.

[Pyrotechnic] tracer compositions. H. C. PRITTHAM (U.S.P. 1,708,151, 9.4.29. Appl., 23.5.24).—A non-explosive composition contains salts of thorium and cerium together with a binder.

H. ROYAL-DAWSON.

Igniting mixture for [pyrotechnic] tracer compositions. L. HENDLER, Assr. to U.S. SECRETARY OF

WAR (U.S.P. 1,708,174, 9.4.29. Appl., 22.10.23).—The mixture includes an alkaline-earth peroxide, red lead, and a metallic fuel. H. ROYAL-DAWSON.

Manufacture of propellant powders. A. S. O'NEIL, Assr. to WESTERN CARTRIDGE CO. (U.S.P. 1,709,868—1,709,870, 23.4.29. Appl., [A] 20.6.24, [B, C] 12.10.25).—(A) The characteristics, (B) densities, and (c) compositions of blended grains are chosen so as to give a progressive-burning powder. S. BINNING.

Smokeless powder and its treatment. W. T. INGRAHAM (U.S.P. 1,710,024, 23.4.29. Appl., 24.7.25).—The powder is gelatinised and grained, the grains after drying being dipped in a solvent for nitrocellulose. S. BINNING.

Diethylene glycol dinitrate and its preparation. W. H. RINKENBACH, Assr. to W. O. SNEILING (U.S.P. 1,686,344, 2.10.28. Appl., 31.5.27).—Diethylene glycol dinitrate, m.p. -11.3° , is obtained in 60–70% yield by nitrating pure diethylene glycol below 15° with mixed acid, sufficient of the ether being added to exhaust completely the nitric acid taken (cf. Rinkenbach, B., 1927, 763). R. BRIGHTMAN.

XXIII.—SANITATION; WATER PURIFICATION.

Disinfectant action. III. Unsaturated compounds as germicides. H. D. CHEESEWORTH and E. A. COOPER (J. Physical Chem., 1929, 33, 720–728; cf. Cooper and Mason, A., 1928, 702).—The bactericidal and protozoicidal action of various aliphatic unsaturated compounds and their corresponding saturated derivatives, and of phenols and cyclohexanols have been compared. In general, unsaturated compounds are the more effective germicides; thus, in respect to various bacteria, allyl alcohol, and crotonic, fumaric, and maleic acids are more effective than the corresponding saturated compounds, and the phenols are more effective than the cyclohexanols. Exceptions to this rule, however, are indicated. The same general relation holds for unsaturated and saturated compounds with respect to paramecium. Benzyl alcohol is a much weaker germicide than are the isomeric cresols and even than the methylcyclohexanols, indicating that the presence of a hydroxyl group in a cyclic structure is more favourable to germicidal power than its presence in a side-chain. Fumaric acid shows a stronger action than maleic acid, an indication that configuration is a more important factor than ionisation. The precipitating power of hydroxy-compounds runs parallel with germicidal action, the unsaturated compounds having a greater effect on egg-albumin and lecitho-protein than the corresponding saturated compounds; it is suggested that greater germicidal power is connected with an increased precipitating or denaturing power of colloidal suspensions. L. S. THEOBALD.

PATENTS.

Extraction of halogens and precious metals from sea-water. H. BARDT (B.P. 294,655, 26.7.28. Ger., 29.7.27).—Iodine, bromine, and precious metals may be recovered from the precipitate obtained by treating sea-water simultaneously or successively with a

reducing agent, e.g., sulphur dioxide, and a mixture of finely-divided, activated carbon and a powdered metal, e.g., copper. C. JEPSON.

Apparatus for purifying waste water with quickened sludge. M. KUSCH (B.P. 310,749, 2.11.28).—In this modification of the activated-sludge process the floor of the aeration tank consists of a series of inverted pyramids, and the circulation and aeration of the contents are effected by means of air-lifts reaching from the bottom of each funnel to a point just below the surface and having an outlet intended to impart an undulating motion to the discharge. The air is applied through a series of small holes from an annular chamber encircling the pipe. C. JEPSON.

Sterilisation of water and other liquids. G. A. KRAUSE (B.P. 279,085, 12.10.27. Ger., 14.10.26).—A vessel, lined with or made of any "oligo-dynamic" metal, e.g., silver, copper, bismuth, antimony, manganese, nickel, aluminium, or their alloys, is so filled with pieces of such metal that the liquid to be sterilised is brought into intimate contact with the maximum amount of metallic surface, thus causing an increased rate of sterilisation. The spaces between the pieces of metal used for filling are made too large to retain micro-organisms by mechanical filtration. C. JEPSON.

Purification of industrial [wool] washing water and the like. C. VAN OVERSTRAETEN (B.P. 300,386, 5.11.27).—To mud and suspended fatty matter, separated or decanted from the water from wool washing etc., is added the precipitate formed by treating the separated water with acid, bleaching powder, or aluminium sulphate. The magma is washed with the least possible amount of water, with or without alkali, and is passed through a mud separator. The mud is discarded after washing again, if necessary, and all the wash waters from the muds are boiled, with or without alkali, and treated to separate the neutral grease. The water from the hydro-extractor etc. is used again as washing agent for the magma. (Cf. B.P. 275,627; B., 1928, 636.) W. G. CAREY.

Antiseptic, antifouling, germicidal, waterproof coatings. A. BIDDLE, Assr. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,689,008, 23.10.28. Appl., 3.7.26).—A water-repelling material, e.g., rubber latex, is incorporated with poisons and/or antiseptic or growth-preventive materials. Thus a sanitary paint is obtained by mixing 10 pts. of calcium rosolate and/or hydroxide with 10 pts. of water and 2 pts. of latex. Methods are given for preparing a plant spray and an antifouling composition. Vulcanising and accelerating agents may also be incorporated. R. BRIGHTMAN.

Vermin-killer. F. FLURY, Assr. to DEUTS. GES. F. SCHÄDLINGSBEKÄMPFUNG M.B.H. (U.S.P. 1,712,917, 14.5.29. Appl., 29.8.21. Ger., 8.4.20).—See B.P. 196,524; B., 1923, 796 A.

[Pendant] apparatus for filtering and treating air [of rooms]. W. JENKS (B.P. 310,351, 24.1.28).

Protective gas-masks, respirators, etc. E. DRÄGER (B.P. 292,531, 4.5.28. Ger., 21.6.27).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 19, 1929.

I.—GENERAL; PLANT; MACHINERY.

Works' control and laboratory equipment. A. SCHRODER (Ind. Eng. Chem., 1929, 21, 481—485).—The considerable reduction in the number of types of physical and chemical apparatus and the more rigid standardisation of the surviving types is discussed and illustrated in the fields of colorimetry, pyrometry, gas analysis, laboratory glassware, porcelain, and chemicals.

C. A. KING.

Automatic control in chemical industries. I. GINSBERG (Ind. Eng. Chem., 1929, 21, 410—414).—Automatic pressure and temperature controllers may be self-operated, in which case they are not very precise and only available through a limited range, or worked by compressed air. These can be made to control temperature to 1° and the best are mercury-actuated. Automatic temperature controllers are successfully used on alcohol or benzol stills, oil-cracking units, nitration kettles, dryers, etc., and in the rubber, paper, and textile industries.

C. IRWIN.

Conversion of batch into continuous processes. J. V. N. DORR (Ind. Eng. Chem., 1929, 21, 465—471).—The development of the Dorr classifier has afforded a continuous process applicable to the separation of sands and slimes in oil treatment and to the size regulation of pigments. It is also used for the removal of grit from sewage. The Dorr thickener performs continuous countercurrent decantation of slimes, and has also been applied to the manufacture of phosphoric acid, aluminium sulphate, caustic soda, and lithopone. A similar device, the "clarifier," is used for the sedimentation of water and of sewage. The Dorr agitator, combining a revolving mechanism with central air agitation, has made continuous agitation processes possible.

C. IRWIN.

Mechanical handling of materials in and about the chemical plant. I. A. K. BURDITT and W. F. SCHAPHORST (Ind. Eng. Chem., 1929, 21, 489—493).—An illustrated account of gravity, belt, screw, and pneumatic conveyors.

C. A. KING.

Progress of industrial heating by oil circulation. A. B. McKECHNIE (Ind. Eng. Chem., 1929, 21, 496—498).—The heating and circulating of oil at high temperatures (316°) for industrial heating requires plant of careful design and robust construction, properly designed for expansional stresses. A rapid circulation of oil should be maintained to prevent local overheating and carbonisation, and the physical properties of the oil are important, particularly the cold viscosity, the proportion of lighter oils, and the quantity of carbon after distillation to dryness. An oil known as "Meprolene" is recommended for this purpose.

C. A. KING.

Calcination. W. S. DICKIE (Ind. Eng. Chem., 1929, 21, 461—464).—The rotary kiln was developed in the cement and lime industries, but has more recently been applied to the undermentioned processes: the reduction of sulphur content of some iron ores, the dehydration of bauxite, the preparation of barium sulphide and lithopone, the revivification of bone black and barium silicate in sugar refining, the calcination of lime sludge and of gypsum, and the dehydration of fuller's earth.

C. IRWIN.

Renaissance of the absorption refrigeration cycle. F. G. KEYES (Ind. Eng. Chem., 1929, 21, 477—480).—The typical refrigerating machine functions on the general principle embodying the difference in solubility of gases in cold and hot absorbing liquids of low vapour pressure, the cooling medium usually being water. Increased efficiency has been attempted by the use of a solid salt, *e.g.*, ammonium nitrate or thiocyanate, which is soluble in ammonia liquor, and recent development in adsorption materials has led to machines in which charcoal or silica gel is used as the adsorbent, or even compounds capable of forming associated compounds, *e.g.*, $\text{BaCl}_2 \cdot 8\text{NH}_3$, with ammonia. Accelerated evaporation has been induced in a constant-pressure system by circulating air over the liquid ammonia, the same result being attempted without mechanical pumping by the introduction of hydrogen, which has a small density as compared with ammonia.

C. A. KING.

Mechanical dispersion by means of the colloid mill. P. M. TRAVIS (Ind. Eng. Chem., 1929, 21, 421—425).—The colloid mill, as distinct from pressure homogenisers, is defined as obtaining dispersion by hydraulic shearing forces. Colloid mills may be of beater type, as the original Plauson mill, or may depend on shearing forces only between either smooth surfaces, or rough surfaces (when turbulence is added), or a combination of the two. It is probable that colloid mills do not actually disintegrate solid particles, but deflocculate aggregates, as in the preparation of paints. In the preparation of emulsions the control of p_H values and temperature and the use of protective colloids are very important. Colloid mills should usually be operated with a clearance of 0.006—0.008 in., and no individual particle of greater diameter should be fed to them.

C. IRWIN.

Crushing and pulverisation. L. T. WORK (Ind. Eng. Chem., 1929, 21, 498—502).—The overall energy of crushing quartz is proportional to the speed of crushing and probably to the new surface developed, though it is much greater than is theoretically necessary. Fundamental resistance to pulverisation of any material is important, but an unmeasured quantity, and practice

has formulated an empirical classification into direct pressure mills, roller mills, tube mills, beater mills, and revolving-plate mills. New developments are in the direction of more wear-resistant materials, simplification of design, and new applications for closed-circuit grinding.

C. A. KING.

Present trends in dust recovery. E. P. PARTRIDGE (Ind. Eng. Chem., 1929, 21, 446—451).—A summary is given of the comparative fields of usefulness of cyclone dust separators, Cottrell precipitators, and vacuum air-filters. It is suggested that air-filters may be applied to the cleaning of chimney gases previous to the use of these to minimise explosion risks in grinding as in powdering coal.

C. IRWIN.

Trend of filtration. A. WRIGHT (Ind. Eng. Chem., 1929, 21, 493—495).—Progress in construction of filter presses has developed chiefly in the direction of increased filter area per unit of floor space. The present trend is to introduce automatic filters to ensure continuity and uniformity of product with minimum attention. The plant must be designed to suit the objective of the particular process, the complete discharge of the cake without impediment to filter porosity being fundamental to every successful filter.

C. A. KING.

Evaporation. P. DE WOLF (Ind. Eng. Chem., 1929, 21, 451—456).—A discussion of the principles of the design of vacuum evaporators. Recent improvements have been mainly in the direction of more rapid circulation. Nickel tubes and tube sheets are increasingly used in the evaporation of caustic soda and of milk.

C. IRWIN.

Apparatus for technical gas analysis. O. BURKHARDT, A. FISCHER, and F. FRANK (Gas- u. Wasserfach, 1929, 72, 504—505).—A modified type of Orsat apparatus is described.

J. S. CARTER.

Errors and illusions in comparison of colours. R. TOUSSAINT (Chim. et Ind., 1929, 21, 924—930).—The difficulties in comparing colours which arise from the different effects produced in light from different sources, and from the imperfections of the eye itself, are discussed. These may be overcome by using the photo-electric colorimeter for the analysis of composite colours.

W. J. BOYD.

See also A., June, 642, **Ultrafiltration, dialysis, and osmometry** (SIGAUD). 673, **Fractionating columns** (MIDGLEY).

PATENTS.

Brine evaporation. [Tube] evaporators. Condensers, coolers, or like apparatus having tubular heat-exchanging surfaces. D. A. QUIGGIN (B.P. 309,104—6, 2.1.28).—(A) The brine discharge and blow-down valves are so arranged and screwed plugs so provided that a cleaning rod may be inserted into the brine-discharge pipe without disturbing the valves. (B) The joints between steam-conveying members are constructed with a conical male part and curvilinear female part. (C) In a tubular evaporator in which one tube plate is smaller than the other so that the whole bundle of tubes can be withdrawn through the shell, means are described for making the (detachable) joints between the shell, tube plates, and headers for inner fluid.

B. M. VENABLES.

Dryers for various materials. R. DE REYERET (B.P. 293,765, 10.7.28. Belg., 11.7.27).—Fire gases pass from a hearth through a first longitudinal flue of refractory material, then up to a second longitudinal flue of cast-iron plates above the first, then sideways (at the hearth end) to a pair of third longitudinal flues constructed between sheet-iron plates and the outer "stone" wall of the dryer. The material is distributed over the whole length of the furnace and slides over each side of the cast-iron and brick flues; between them and the sheet-iron flues the iron plates are suitably inclined and the material is drawn out laterally below the third flues. An air flue may be formed between the brick and cast-iron flues; the air heated in this is allowed to permeate the material from under a hood formed over the top of the cast-iron flue.

B. M. VENABLES.

Spray drying. W. S. BOWEN (U.S.P. 1,711,306, 30.4.29. Appl., 1.7.27).—The heated gas is admitted to a circumferential passage round the top of the desiccating chamber and descends between radial guides that extend from the circumference part way towards the axis; the liquid is sprayed at the top of the central passage thus left.

B. M. VENABLES.

Antifreezing composition. A. H. OSTERLUND (U.S.P. 1,711,324, 30.4.29. Appl., 24.8.25).—A mixture of glycerin with boiling sodium glycerophosphate is cooled, and a clarifying agent added, the latter being finally drawn off with the settled impurities. The mixture is adjusted to freeze below -37° .

B. M. VENABLES.

Grinding mill. D. COLE (U.S.P. 1,711,405, 30.4.29. Appl., 27.7.28).—A form of self-aligning roller bearing for rotary drums is described.

B. M. VENABLES.

Tubular grinding mills. F. KRUPP GRUSONWERK A.-G. (B.P. 288,259, 13.3.28. Ger., 7.4.27).—A tube mill is provided with a transverse chamber at one end for the collection and discharge, by known methods, of fine material; immediately preceding the transverse chamber is a screening chamber arranged after the manner of a Krupp ball mill, except that the perforated, stepped grinding plates may be inclined to the axis, and that the material passing the screen enters an annular collecting chamber, rotating with the mill, whence it passes to the transverse chamber above mentioned.

B. M. VENABLES.

Grinding machine. L. RUPRECHT and A. G. KOLLSTEDE (U.S.P. 1,711,464, 30.4.29. Appl., 10.2.27).—Beaters rotate in a casing in such a manner that they strike upwards the material which is fed in a compact mass by a substantially horizontal worm conveyor.

B. M. VENABLES.

Roller mills for grinding. H. E. COX and J. R. TORRANCE (B.P. 310,769, 27.1. and 26.10.28).—A mill with 4 rollers and 3 nips is arranged so that the total material passes downwards between an upper pair of slow rolls, then divides and passes outwards between the lower fast rolls and the upper rolls. The lower rolls may run from 6 to 30 times as fast as the upper. The upper rolls may rotate at equal speeds, or, better, one or both may have a continually varying speed so that the relative motion is continually changing. All the nips are adjustable.

B. M. VENABLES.

Crusher roll construction. G. W. JOHNSON. From AMER. ENGINEERING CO. (B.P. 311,534, 23.4.28).—A toothed roll is formed round a prismatic shaft upon the flat sides of which rest the bases of the teeth. The teeth are shouldered and are a loose fit in holes in sleeves which embrace the shaft and teeth; keys of special form are provided to tighten the teeth in their holes.

B. M. VENABLES.

Pulverising mill. R. S. RILEY, ASST. to SANFORD RILEY STOKER CO. (U.S.P. 1,711,063, 30.4.29. Appl., 3.1.24).—A bull ring and co-acting roll hammers are both driven positively in opposite directions.

B. M. VENABLES.

Pulverising apparatus. F. H. DANIELS, ASST. to RILEY STOKER CORP. (U.S.P. 1,711,044, 30.4.29. Appl., 12.4.27).—Two pulverising zones are provided within one disintegrator casing; the first reduction of the material is effected by hammers and the second by beaters, the space outside the hammer path forming direct peripheral communication with the beater zone.

B. M. VENABLES.

Mixing and grinding device. J. A. MICHAL, ASST. to TURBINATOR CO., INC. (U.S.P. 1,711,154, 30.4.29. Appl., 30.12.26).—A rotor having a diameter substantially greater than its axial length is provided with teeth or blades on the faces which are substantially transverse to the axis. The teeth intercalate with others on a fixed casing. The feed for material is near the axis and outlet at the periphery.

B. M. VENABLES.

Machines for kneading, crushing, mixing, etc. H. G. TORULF (B.P. 310,791, 30.1.28).—The machine comprises a drum rotated by sleeve shafts, or trunnions, surrounding a solid shaft which is cranked within the drum; the crank pin carries a pair of links between the free ends of which a kneading roller is journaled. The sleeve trunnions and solid shaft are rotated by power at different speeds, preferably in opposite directions, but the rotation of the roller about its own axis is effected by contact with the material in the interior of the drum. The crankshaft also carries a scraper to discharge the material through a door in the drum when the latter only is held stationary.

B. M. VENABLES.

Mixing machines. A. E. WHITE. From HOBART MANUF. CO. (B.P. 311,099, 10.5.29).—A mixing machine having a beater attached to a [vertical] shaft is provided with a conical sleeve at the junction of the beater and its shaft to arrest material creeping upwards. An upwardly inclined extension of the beater arm may also be provided to exert a downward thrust on the material to prevent it rising.

B. M. VENABLES.

Apparatus for use in separating, sorting, and/or grading materials. E. M. SAVAGE (B.P. 311,310, 9.2.28).—A tiltable shaking table suitable for the separation of rounded from angular pebbles (e.g., diamonds from rock) is described.

B. M. VENABLES.

Centrifugal machine. L. D. JONES, ASST. to SHARPLES SPECIALTY CO. (U.S.P. 1,711,468, 30.4.29. Appl., 9.4.26).—A centrifuge which is used to separate concentrated emulsion and pure continuous phase is provided with means to keep constant the ratio of the two products discharged, at any speed or output.

B. M. VENABLES.

Centrifugal extractor. T. A. BRYSON, ASST. to TOLHURST MACHINE WORKS, INC. (U.S.P. 1,710,447, 23.4.29. Appl., 25.10.21).—A centrifugal separator comprising an imperforate bowl in which the heavier constituent is retained is divided into a number of annular chambers by baffles extending inwards from the wall of the bowl to a greater extent than the overflow rim for separated lighter liquid. The radial flow is practically nil, and the axial flow to the outlet is made very slow compared with the circumferential flow by providing transfer ports in the baffles at points which are far removed circumferentially from their neighbours. If desired, before final outlet the lighter liquid may pass inwardly through a cylindrical screen within the bowl.

B. M. VENABLES.

[Centrifugal] separation of impurities from liquids. C. H. HAPGOOD, ASST. to DE LAVAL SEPARATOR CO. (U.S.P. 1,711,315, 30.4.29. Appl., 24.8.21).—A viscid substance is separated centrifugally, then heated by a medium not in contact with it to render it sufficiently fluid to flow away.

B. M. VENABLES.

Separators for treatment of mixtures of mutually insoluble liquids. H. J. HOLFORD, and HARVEY HOLFORD SEPARATORS, LTD. (B.P. 310,996, 4.2.28).—The mixture is admitted to the lower part of a vessel, and the light liquor travels upwards without much obstruction, the top level being held constant by a ball valve controlling the outlet for light liquid. The heavy liquid passes downwards through slots in an inclined baffle, then upwards in a sub-chamber charged with packing material, the outlet being in the upper part of the sub-chamber, but not so high as that for the lighter liquid.

B. M. VENABLES.

Rotary filters. BRIT. ROTARY FILTER CO., LTD., and A. G. E. JOYCE (B.P. 311,359, 10.2.28).—A construction of the cells and filtrate discharge pipes within the drum of a rotary filter is described; its object is to reduce the wetted surface and amount of undischarged liquor to a minimum.

B. M. VENABLES.

Effecting an intimate contact between two fluids. C. H. FOOT (B.P. 311,494, 8.3.28).—The fluids may be either a liquid and a gas or two liquids of different sp. gr. One fluid (for convenience assumed the lighter) is introduced in the form of drops or bubbles into the lower part of a comparatively narrow column of the heavier or carrier fluid, the bubbles or drops filling the whole cross-section of the tube and causing a rising broken column of mixed fluids. The actual introduction of the lighter fluid is through a fine jet situated in an enlarged space at the bottom of the column, and the separation of the two fluids is effected in an enlargement at the top, the carrier fluid returning to the bottom through a separate conduit forming a closed circuit; no other means of circulation is provided, and the circulation may be retarded to give any desired time of contact by an adjustable obstruction, such as a tap, in the return conduit. If the carrier fluid has the lower sp. gr. the apparatus must be arranged for reversed circulation.

B. M. VENABLES.

Apparatus for treating gases with liquids. CHEM. ENGINEERING & WILTON'S PATENT FURNACE CO., LTD., and N. and T. O. WILTON (B.P. 311,249, 8.11.27).—

The gas passes downwards through a number of chambers in succession, and liquid is sprayed into the top of each chamber and passes together with the gas over fixed contact surfaces comprising a number of loose vertical tubes inside which are twisted strips or like helical elements. The liquid is circulated from the bottom of each chamber to the spray of the same chamber, and there is a slower flow from chamber to chamber; this flow is arranged to be countercurrent to the gas flow.

B. M. VENABLES.

Method of reciprocal action between gases and finely subdivided materials. METALLGES. A.-G. (B.P. 285,038, 9.2.28. Ger., 9.2.27).—The gas to be treated with powder is caused to flow upwards through a vertical chamber, which preferably expands upwardly, at such a rate that the powder is held in suspension. When the powder is spent the gas current is increased and the powder carried over to a separate collector. If desired, the gas current may be pulsating with intermittent removal of the powder. Two towers may be used alternately, one being used for the reaction while the other is being used for discharge. B. M. VENABLES.

Simultaneous separation and thermic treatment of mixtures of fluids and granular or other substances. P. SCHMIDT (B.P. 285,046, 9.2.28. Ger., 9.2.27).—The mixture is caused to move between surfaces which are maintained at different temperatures and have relative parallel motion. One form of apparatus suitable for drying of dust in dust-laden air comprises an unheated disc rotating above an annular surface heated from below. The mixture is fed upwards through the centre of the heated surface and is discharged at the periphery; the dust then falls downwards and the air passes out upwards. In an apparatus suitable for extracting crystals from hot saturated solutions the rotating disc is cooled. B. M. VENABLES.

Separating gases and liquids under pressure. C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,985, 31.12.27).—The mixed fluid is admitted to a floating chamber within a pressure-resisting vessel of about the same shape; the floating chamber is supported by a spring or equivalent means and operates a sleeve valve through which liquid can leave through the lower part of the floating chamber and of the outer pressure-resisting wall. The gas leaves from the upper part of the vessel. The level of the liquid being automatically held constant by the floating chamber, the outflow of gas may be controlled by hand or other means, and will consequently control the inflow of mixture. B. M. VENABLES.

Separation of gaseous and liquid products [under pressure]. C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,193, 31.12.27).—The products from a high-pressure reaction (*e.g.*, the destructive hydrogenation of carbonaceous material) are transferred (without substantial reduction of pressure) to a catch pot which is maintained at a temperature high enough to prevent condensation of vapours. The liquid only is drawn off through a cooler (which may be a heat exchanger) and valve or engine such as a Pelton wheel. The gas and vapour pass through a separate cooler or heat exchanger to a second (cold) catch pot

where the liquefied vapour is separated from the permanent gas, the pressure energy of each product being recovered in an engine if desired. B. M. VENABLES.

Separation of mixtures of liquids, or of liquids and solids, or of liquids and gases. SOC. ANON. DES CHARBONS ACTIFS E. URBAIN (B.P. 283,192, 1.11.27. Fr., 8.1.27).—A capillary siphon is used to transfer the liquid only from one vessel to another. The capillary spaces may be larger than the particles of solids to be separated provided that the capillary material is so chosen as to have an electric charge of the same sign as the phase to be left in the first vessel; this charge may be developed by washing the capillary material with compounds that are used in the mordanting of webs. In some cases the material should be cleaned with detergents, and in all cases it is advisable previously to wet the material with the liquid that is to be transferred. B. M. VENABLES.

Removal of dust from air, gas, and other elastic fluids. C. H. SCHOL (B.P. 311,639, 29.10.28).—A number of inclined baffles are placed alternately along either side of a long chimney, causing repeated slight compression with subsequent eddying of the stream of gas. The dust is thrown out of the eddies and falls down channels left between the baffles and the wall of the chimney, or through separate passages leading from different vertical stages of the chimney.

B. M. VENABLES.

Conditioning of air for ventilation. HEENAN & FROUDE, LTD., and G. H. WALKER (B.P. 311,304, 8.2.28).—In cases, such as mines, where a supply of cooling water is available rather below the temperature of the untreated air which contains considerable moisture, the cooling water is passed through the annular spaces of a heat interchanger constructed of twin concentric tubes; an absorbing liquid such as calcium chloride solution is passed through the inner tubes and afterwards sprayed over the outside of the outer tubes (which may be gilled) in company with the air to be conditioned. The air is thus dried and its heat content reduced, while the consequent rise of temperature of the fluid is prevented. If desired, the device for effecting contact between liquid and air may be separate from the cooler, and, after drying, the air may be again humidified. The absorbing liquid is kept in circulation and part or all is subjected to a concentrating process and returned to the circuit. B. M. VENABLES.

Fluid-storing material. F. G. KEYES, Assr. to NAT. REFRIGERATING CO. (U.S.P. 1,705,482—4, 19.3.29. Appl., [A, B] 6.11.23, [C] 15.1.25).—A hard porous mass for storing gases and liquids comprises a mixture of (A) zinc oxychloride, calcium chloride, and sodium silicate, (B) magnesium oxychloride, calcium chloride, and active charcoal, and (C) a halide capable of forming additive compounds with fluids, an oxychloride cement, and alundum cement. A. R. POWELL.

Conveying and consuming without loss liquid gases such as liquid air, liquid oxygen, liquid nitrogen, etc., boiling at low temperatures. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 280,569, 10.11.27. Ger., 13.11.26).—The storage or transport vessel has no communication with the atmosphere, but

communicates with a receiver into which any gas evaporated from the liquid passes and causes a progressive rise of pressure, which, in turn, stifles evaporation. The capacity of the receiver is calculated to contain safely all the vapour produced in the longest period between successive intentional withdrawals of gas for use.

B. M. VENABLES.

Means for ascertaining the percentage composition of substances after a given dilution and vice versa. NESTLÉ & ANGLO-SWISS CONDENSED MILK CO. (AUSTRALASIA), LTD. (B.P. 285,383, 13.2.28. Austral., 14.2.27).—The apparatus comprises a chart of the percentage compositions of various concentrated substances (*e.g.*, foods) and a rotatable circle on the principle of the slide rule by which the composition after adding a known proportion of water, or the water to be added to give a desired composition, may be determined.

B. M. VENABLES.

Pulverising apparatus. O. CRAIG, ASST. to RILEY STOKER CORP. (U.S.P. 1,714,080, 21.5.29. Appl., 15.12.27).—See B.P. 302,330; B., 1929, 306.

Filtration. E. A. ALLIOTT, C. BOIS, and A. E. HATFIELD (U.S.P. 1,713,317, 14.5.27. Appl., 19.2.27. U.K., 20.3.26).—See B.P. 270,461; B., 1927, 511.

Centrifugal apparatus [for gases]. H. A. HUMPHREY, ASST. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,714,370, 21.5.29. Appl., 26.11.27. U.K., 15.1.27).—See B.P. 276,557; B., 1927, 842.

Absorption refrigerating apparatus. G. BEAUMONT and G. MANSIO (B.P. 290,585, 14.5.28. Fr., 13.5.27).

Refrigerating apparatus of absorption type. H. D. FITZPATRICK. From N. V. KODOWA REFRIGERATOR CO. (B.P. 311,891, 3.4.28).

Evaporators for refrigerating. H. R. VAN DEVENTER and J. A. GRIER (B.P. 299,728, 29.10.28. U.S., 27.10.27).

Thermostats [of float-valve type]. J. L. CATON (B.P. 311,901, 3.1.29).

Apparatus for filling solid articles in layers into receptacles. I. G. FARBERIND. A.-G. (B.P. 299,887, 1.11.28. Ger., 3.11.27).

Heat-treatment of pulverulent materials (B.P. 310,907).—See II. **Moisture-absorbent** (B.P. 311,578).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Determination of hygroscopic moisture in coal. H. LÖFFLER (Chem.-Ztg., 1929, 53, 411).—The coal is dried at 60–70° in a vacuum. A convenient form of apparatus, in which heating is effected by a vapour jacket, is described and illustrated. J. S. CARTER.

Waxes and boghead coals as parents of petroleum. IV. N. D. ZELINSKI and K. P. LAVROVSKI (Ber., 1929, 62, 1264–1266; cf. A., 1928, 731, 865).—Distillation of purified beeswax with aluminium chloride affords carbon dioxide and inflammable gases with a mixture of liquid and solid hydrocarbons of paraffinoid nature. Two substances, m.p. 59° and 70–71°, respectively, are characterised. Boghead coal when heated with alum-

inium chloride gives a liquid volatile with steam in which unsaturated cyclic hydrocarbons appear to predominate; the portion of the distillate not volatile with steam gives a crystalline paraffin, m.p. 63–64°.

H. WREN.

Chemical composition of peat. IV. Chemical studies of highmoor peat from Maine. S. A. WAKSMAN and K. R. STEVENS (Soil Sci., 1929, 27, 389–398; cf. B., 1929, 421).—Examination of some sphagnum peats is recorded. The sphagnum horizon was acid (p_H 4.0) and had low ash, nitrogen, and lignin contents and high cellulose, hemicellulose, fat, and wax contents. With the transition to the lower or sedimentary peat layers there is a rise in p_H value, an increase in ash, protein, and lignin values, and a decrease in cellulose and hemicellulose.

A. G. POLLARD.

Economic test of low-temperature coking. R. S. MCBRIDE (Chem. Met. Eng., 1929, 36, 288–291).—The K.S.G. process, which has been in large-scale operation at Essen for 5 years, has been adopted in a plant now starting work in New Jersey. It consists of 8 retorts consisting of two concentric drums 85 ft. long. These are externally heated by producer gas, the coal travelling first through the inner drum and back through the outer. The transference takes place as the coal is becoming plastic, and the rapid subsequent heating reduces the plastic period to a minimum. Soot deposits are minimised by tangential steam-jets. The gas is to be used for public supply. It is anticipated that the tar will yield 5 gals. per ton of light products and a high content of phenols. Pea-size semi-coke is to be used for generating producer gas. The steam used is generated by the products of combustion, and the total heat used in carbonisation is estimated at 800 B.Th.U. per lb. of coal.

C. IRWIN.

Phenol recovery [from coke-oven effluent] and treatment. Works of the Hamilton Coke and Iron Company. B. F. HATCH (Ind. Eng. Chem., 1929, 21, 431–433).—The first large-scale plant operating the Seaboard, N.J., process of phenol recovery is described. Ammonia liquor is withdrawn from the bottom of the free ammonia still before liming and sprayed down a high steel tower packed with tiles against a mixture of 90% of steam and 10% of air, the temperature being kept just below the b.p. of the liquor. The lower three fourths of the tower form dephenolising sections, being packed with steel turnings and fed with caustic soda. Both gas and caustic soda are recirculated. The heat consumption is low owing to good insulation, and the phenol recovery may reach 95%.

C. IRWIN.

Occurrence of pyridine bases in the tar oils from Russian bituminous schists. J. DODONOV and E. SOSCHESTWENSKAJA (Ber., 1929, 62, 1348–1352; cf. B., 1926, 1006).—Fractions of the oil, b.p. 140–250°/650 mm., are freed from phenols by treatment with alkali and shaken with sulphuric acid (d 1.3) whereby 1.1% of basic substances are isolated. The bases are further fractionated and the individual fractions are transformed into their hydrochlorides and thence into their additive compounds with mercuric chloride. The presence of pyridine, 2- and 3-methyl-, 2:4-, 2:5-, and 2:6-dimethyl-pyridine is established.

H. WREN.

Oil-testing apparatus. H. HERBST (Chem.-Ztg., 1929, 53, 344—345).—By means of the apparatus the surface tension of liquids (especially of lubricating oils), the lubricating power of oils in contact with metals, the angle of contact of these oils and metals, the viscosity of pitch, rosins, size, etc., and the thickness of bodies (e.g., the dimensions of metal blocks) may be measured. The apparatus consists of a rectangular table along two adjacent sides of which is fixed a vertical framework in which sliding carriages may be moved vertically up or down by means of micrometer screws. Each carriage carries a horizontal arm; to one of these is fixed a reading telescope or lens, and to the other a holder supporting a capillary tube in which the height of capillary rise etc. of the liquids under examination may be measured by means of the telescope and the scales on the micrometer screws. For measuring, e.g., the lubricating power of oils in contact with metals, standard rectangular blocks of the same or of different metals are fixed by means of clamps and distance pieces so that their inner faces are parallel and at a standard distance apart. The blocks are then placed centrally under a capillary tube containing the required oil, and the distance through which the surface of the oil in the tube falls is measured. In order to determine the dimensions of the metal blocks referred to, the capillary tube is replaced by a steel ruler suitably graduated. The apparatus can also be used for measurement of small pressure differences by substituting a manometer for the capillary tube. B. P. RIDGE.

Oxidation of motor fuels. E. BERL, K. HEISE, and K. WINNACKER (Z. physikal. Chem., 1929, 141, 223; cf. B., 1929, 158).—Acknowledgment is made that the "peroxide" theory, which explains the knocking of certain fuels in internal-combustion engines, was put forward by Moureu and his co-workers in 1925, and by Callendar (B., 1927, 272) only in 1927. A. J. MEE.

Determination of unsaturated and aromatic hydrocarbons in light oils and motor spirits. A. B. MANNING (J.C.S., 1929, 1014—1020).—0.2—0.5 g. of the oil is vaporised in a current of air and passed through a bubbler charged with a 10% mixture of concentrated nitric acid (or 16% of potassium nitrate) in concentrated sulphuric acid. The increase in weight of the bubbler gives the total unsaturated and aromatic hydrocarbons in the oil. This solution in the acid mixture is heated on the water-bath for 2—3 hrs. to complete both the nitration of the aromatic hydrocarbons and the oxidation of the unsaturated hydrocarbons. The cooled acid solution is poured into excess of water and the nitro-compounds are extracted with three 50-c.c. portions of benzene. The benzene extract is itself extracted with 10% sodium hydroxide, and is finally evaporated to dryness. The weight of aromatic hydrocarbons in the original oil is calculated by multiplying the weight of mixed nitro-compounds obtained by the factor 0.452; the result so obtained is liable to an error of about one unit on the percentage for every 20% of unsaturated hydrocarbons present.

R. J. W. LE FEVRE.

See also A., June, 655, **Low-temperature oxidation of hydrocarbons** (LEWIS). 658, **Activity of charcoal**

(ALEXSEEVSKI and AVGASTINIK). 673, **Fractionating columns** (MIDGLEY). 687, **Action of acetylene on benzene in presence of aluminium chloride** (BÖESEKEN and ADLER).

Oil for heating purposes. McKECHNIE.—See I. **Wet carbonisation of wood.** SCHWALBE.—See V. **Sulphuric acid.** SPANGLER.—See VII. **Cast-iron pipes.** BRADSHAW.—See X.

PATENTS.

Briquetting of fuels. CHEM.-TECHN. GES.M.B.H. (B.P. 300,195, 28.9.28. Ger., 8.11.27).—Briquettes are manufactured from a blend of a caking and a non-caking or poorly-caking fuel; the two fuels are first heated separately, then mixed and briquetted under pressure. The caking fuel is heated to a temperature below its softening point, whilst the non-caking fuel is raised to a higher temperature, so that on admixture the interchange of heat causes the former fuel to cake. The caked product may be subjected to further heat-treatment before being allowed to cool.

A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. H. G. WATTS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,197—8, [A] 28.1.28, [B] 30.1.28).—(A) The separation of the solid and liquid products of the destructive hydrogenation of coal etc. by filtration is facilitated by the preliminary addition thereto of finely-divided, insoluble, carbonaceous material, e.g., coal or coke. The product is preferably filtered hot, i.e., at about 100°, after the removal of the lower-boiling constituents. (B) The separation is further facilitated by dilution of the material with an oil capable of dissolving pitch, preferably an oil of b.p. 200—300° derived from the process itself. The solid matter may be washed with light oil and then briquetted.

A. B. MANNING.

Heat treatment of pulverulent [carbonaceous] materials. J. N. BAILEY, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 310,907, 1.2.28).—A retort for the fractional distillation of carbonaceous material is so shaped that the material falls through one elongated chamber or a succession of separate vortex chambers, the heating gas being admitted tangentially at different regions or to different chambers; portions of the contents of the chambers are withdrawn from points in different regions, but all adjacent to the axis of the vortices, by means of a number of concentric conduits.

B. M. VENABLES.

Liquefying and solubilising coals by extraction with solvents under pressure. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,031, 1.3.28).—Solid carbonaceous materials are heated under pressure with a suitable solvent, e.g., tetralin, high-boiling mineral oil fractions, in the presence of small quantities of halogens, halogen hydrides, or compounds which set free these substances under the conditions of the reaction.

A. B. MANNING.

Manufacture of valuable liquid products from varieties of coal, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 311,628, 3.7.28. Addn. to B.P. 282,384; B., 1929, 465).—The apparatus described

in the main patent is modified by constructing of silver or its alloys those parts which come into contact with sulphur-contaminated hydrocarbons at elevated temperatures.

A. B. MANNING.

Manufacture of valuable liquid hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,251, 5.12.27).—Coals, tars, mineral oils, etc. are subjected to destructive hydrogenation or cracking in the presence of catalysts, *e.g.*, tungstic acid, molybdic acid, either alone or mixed with chromium or other metallic oxides, cobalt oxide, etc. which have been subjected to a preliminary treatment at an elevated temperature with gases such as carbon dioxide, sulphur dioxide, nitric oxide, nitrogen, or oxygen, which have no reducing action on them.

A. B. MANNING.

Control of combustion. W. M. HEPBURN, ASSR. to SURFACE COMBUSTION CO. (U.S.P. 1,710,772, 30.4.29. Appl., 10.12.23).—A furnace is provided with a regulator for controlling the rate of supply of air, which is automatically increased or decreased in accordance with the presence in the flue gases of unconsumed fuel or unconsumed air respectively.

A. B. MANNING.

Production of activated carbon. W. M. WILLIAMS, R. S. CLAYTON, (SIR) J. P. FRY, and A. R. HARPER (B.P. 310,908, 1.2.28).—Powdered carbonaceous material, *e.g.*, coal dust, mixed with a limited supply of air, is injected into a retort maintained at 800–1000°, and the partially burnt material is kept in suspension in the gaseous combustion products until it is activated. The retort may take the form of a vertical cylindrical chamber into the upper end of which the material and air are injected tangentially, the retort having first been heated by the combustion of a gas-air mixture therein. An acid atmosphere is maintained within the retort either by addition of acid to the raw material or its introduction directly into the combustion chamber. A water spray may be introduced into the lower part of the chamber in order to quench the product as soon as activation is complete.

A. B. MANNING.

Manufacture of activated carbon. A. B. RAY, E. G. DOYING, and J. J. BUTKOVSKY, ASSRS. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,694,040, 4.12.28. Appl., 22.10.25).—Cellulose material, *e.g.*, nut shell, is impregnated with 50% of phosphoric acid or 100% of zinc chloride, or other dehydrating agent, and heated to 350°, leached, and reheated to 600° in steam or other oxidising atmosphere.

R. BRIGHTMAN.

Gas producers. F. L. BROUGHTON and D. HADLINGTON (B.P. 311,587, 3.7.28).—A producer with a rotatable base has attached thereto an ash box, a regular discharge of ashes from the producer to the box being caused by arms rotating on a shaft within the latter. A scraper on the underside of the base gathers together the ashes discharged from the box and delivers them to a truck or conveyor. Vertical rods attached to the base prevent the formation of masses of clinker within the producer. Air and steam are admitted through a central stationary pipe, above the open end of which is a cap, and attached to which are guards or scrapers to prevent blocking of the inlet. Additional

steam is preferably supplied through the sides of the producer at a slightly higher level than the main steam and air inlet.

A. B. MANNING.

Production of gas. SILAMIT-WERKE DR. STRASSMANN & CO., FABR. FEUER- & SÄUREFESTER PROD. M.B.H. (Swiss P. 122,317, 12.5.26).—Part of the distillation retort is heated directly by combustion gases during the whole process, while the remainder is heated directly until the gas consumption is at a maximum; it is then heated by the waste gases from the heating of the first part.

L. A. COLES.

Apparatus for production of oil gas. A. SCHILLING, R. SACHSE, D. LIAMIN, and T. CALLAERT (U.S.P. 1,710,900, 30.4.29. Appl., 23.11.26).—A generating chamber with a chequered hearth has a wall of refractory material rising therefrom and dividing the interior into a number of concentrically disposed chambers, which are connected by apertures in the lower part of the wall. A combustible mixture of oil, air, and steam is projected axially downwards into the inner chamber, while jets of oil and steam are projected against the outer surface of the refractory wall. The gases produced pass through the chequered hearth, and are then withdrawn from the apparatus.

A. B. MANNING.

Gas scrubber. G. FAST (U.S.P. 1,713,175, 14.5.29. Appl., 28.5.25).—The gas passes through a number of rotating sprays of liquid moving in opposite directions.

J. A. SUGDEN.

Extraction of acetylene from gases. I. G. FARBENIND. A.-G. (B.P. 303,068, 27.12.28. Ger., 27.12.27).—Gases poor in acetylene are treated with acetonitrile cooled to about –10°; the acetylene is recovered on boiling.

H. ROYAL-DAWSON.

Still for use in oil refining, and method of making same by electric arc welding. E. C. R. MARKS. From A. O. SMITH CORP. (B.P. 310,761, 24.12.27; cf. B., 1929, 385).—Two end heads in the form of hollow hemispheres and a number of thick, seamless, annular sections of cast steel of the same thickness and diameter, having the circumferential edges partially cut away are aligned end to end and fused together by an electric arc in the region of the grooves to constitute an integral tubular structure without longitudinal joints.

H. S. GARLICK.

Extraction of oils from materials containing the same. I. G. FARBENIND. A.-G. (B.P. 283,159, 4.1.28. Ger., 5.1.27).—Oils, mixed with solid coaly materials and inorganic matter, obtained as residues in the destructive hydrogenation of carbonaceous materials, are completely extracted by treatment with liquid sulphur dioxide.

H. S. GARLICK.

Manufacture of natural gasoline. H. E. THOMPSON, ASSR. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,695,162, 11.12.28. Appl., 12.4.24).—Liquid gasoline prepared from natural gas, after stabilisation by removal of undesirable volatile fractions, is cooled below its normal b.p. for example, first with water, and then to at least 0° with the cold vapours (–20°) from the rectifying column. Alternatively, vapour may be withdrawn

from the liquid gasoline, compressed, cooled, and expanded, and the cold expanded material returned.

R. BRIGHTMAN.

Cracking of hydrocarbons. E. C. HERTHEL and H. L. PELZER, Assrs. to SINCLAIR REFINING CO. (U.S.P. 1,703,528, 26.2.29. Appl., 17.1.25).—The bulk supply tank is provided with a filter bed containing metallic oxides, and oil circulates relatively slowly through the tank and bed and rapidly through an externally-heated cracking coil from which the hot oil is discharged to the supply tank by a submerged pipe above the bed. Vapours escape to a reflux tower, and thence to condenser and receiver. Crude oil is fed into the top of the reflux tower, and the reflux is returned to the tank or direct to the circulating pipe to the heating coil. Tar oil may be withdrawn from this circulating pipe before it discharges to the heating coil.

R. BRIGHTMAN.

Conversion [cracking] of hydrocarbon oils. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,703,100, 26.2.29. Appl., 31.10.21).—The crude oil passes downward through closed coils forming the condensing medium in a reflux tower up which the cracked vapours from the vaporising chamber pass to the water condenser. Pressure distillate may also be sprayed in at the top of the reflux tower in contact with the ascending vapours, and the phlegms collect in a well into which the crude oil from the coil discharges and from which the oil thus preheated is pumped to the heating tubes. Part of the oil may be by-passed from the well, through the cooler, to the crude oil feed to the tower.

R. BRIGHTMAN.

Cracking of hydrocarbon oil and apparatus therefor. C. ARNOLD. From STANDARD OIL DEVELOPMENT CO. (B.P. 311,362, 10.2.28).—A stream of oil is heated to cracking temperature in a single passage through a heating zone and passed to a digestion zone in which partial vaporisation occurs. The vaporised portion is partially condensed in a condensing zone under substantially the same pressure as that of the heating and digestion zones. The condensate is run wholly or partially under the action of gravity into the stream of hot oil passing from the heating to the digestion zone at a position anterior to that at which it enters the digestion zone and in sufficient quantity to control the temperature therein.

H. S. GARLICK.

Treatment [cracking] of petroleum oils. W. M. CROSS, Assr. to GASOLINE PRODUCTS CO., INC. (U.S.P. 1,696,030, 18.12.28. Appl., 14.10.22).—Petroleum oil under 400–750 lb./in.² pressure is heated above its flash point and passed into a reaction chamber, where it is maintained at 375–485° by the controlled introduction, through a perforated pipe, of a combustible gas. The converted oil is passed into a still, where the pressure is released, and the lighter fractions distil off. The reflux condensate is returned to the heating coil and the residuum in the still, containing the carbonaceous matter, is drawn off.

R. BRIGHTMAN.

Emulsifiable oil. D. R. MERRILL, Assr. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,695,197, 11.12.28. Appl., 14.6.23).—Emulsions for treating wools, spraying fruit trees, or for use as sheep dip, lubricants, or Turkey-red oil substitute, are obtained by mixing 70 pts. of

sodium naphthenate, of about 8% water content, with 17 pts. by vol. of butyl alcohol or other aliphatic alcohol containing more than two carbon atoms, and dissolving 30 pts. of this mixture in about 70 pts. by vol. of a mineral lubricating oil.

R. BRIGHTMAN.

Decolorisation of kerosene, gasoline, and similar light petroleum distillates. V. A. KALICHEVSKY, Assr. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,695,251, 11.12.28. Appl., 19.9.27).—When washed with sodium hydroxide, *e.g.*, with 5% of a 10% solution, before agitation with acid and clay or with clay alone, light petroleum distillate gives a product equal in colour to, but more stable than, that obtained by air oxidation.

R. BRIGHTMAN.

Manufacture of pure mineral oil sulphonates. C. FISCHER, JUN., and W. T. REDDISH, Assrs. to TWITCHELL PROCESS CO. (U.S.P. 1,703,838, 26.2.29. Appl., 11.10.27).—Mahogany soap, containing, *e.g.*, 20–25% of oil, is dried and mixed with an aqueous solvent, *e.g.*, alcohol above 75% concentration, saturated with sodium carbonate or other water-soluble salt to keep the sulphonates out of the lower layer. The upper layer on decantation and evaporation of the solvent affords pure mineral oil sulphonates.

R. BRIGHTMAN.

Manufacture of carbonaceous material. H. N. GILBERT, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,714,165, 21.5.29. Appl., 9.5.25).—See B.P. 251,982; B., 1927, 805.

Plant for utilising the sensible heat of hot coke. O. HELLER (U.S.P. 1,714,168, 21.5.29. Appl., 29.9.24. Ger., 3.10.23).—See B.P. 222,886; B., 1925, 580.

Apparatus for drying and distilling lignite, peat, non-coking coals, and other similar carbonaceous matter. H. DEBAUCHE (U.S.P. 1,713,032, 14.5.29. Appl., 6.7.27. U.K., 20.7.26).—See B.P. 270,921; B., 1927, 547.

Production of mixed coal gas and water-gas. F. D. MARSHALL (U.S.P. 1,713,189, 14.5.29. Appl., 10.4.26).—See B.P. 261,975; B., 1927, 244.

Conversion of hydrocarbon oils. J. F. DONNELLY (U.S.P. 1,712,789, 14.5.29. Appl., 21.11.24).—See B.P. 243,339; B., 1927, 356.

Manufacture of synthetic liquid fuels. E. A. PRUDHOMME, Assr. to SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (U.S.P. 1,711,856, 7.5.29. Appl., 7.4.26. Fr., 9.3.26).—See B.P. 267,512; B., 1928, 470.

Low-compression fuel. E. V. BERESLAVSKY, Assr. to ETHYL GASOLINE CORP. (U.S.P. 1,713,589, 21.5.29. Appl., 17.9.25).—See B.P. 258,550; B., 1927, 900.

Apparatus for treating [hydrocarbon] oils. G. W. WALLACE (U.S.P. 1,714,198, 21.5.29. Appl., 24.10.24. U.K., 1.8.24).—See B.P. 233,395; B., 1925, 662.

Apparatus for [oil]-separation of combustible material from its associated non-combustible material. W. L. REMICK (U.S.P. 1,711,326, 30.4.29. Appl., 18.2.26).

Quenching of coke. J. PINTSCH A.-G. (B.P. 299,315, 22.10.28. Ger., 21.10.27).

[Mixing device for] preparation of charges for

use in internal-combustion engines. A. S. JOHN (B.P. 311,847, 9.8.28).

Separation of gaseous and liquid products (B.P. 311,193).—See I. Hydrogen and gases containing it (B.P. 311,299). Sulphur from gases (U.S.P. 1,695,068).—See VII. Improvement of ferrous metals (U.S.P. 1,712,879).—See X. Sulphur product (U.S.P. 1,690,335).—See XIII.

III.—ORGANIC INTERMEDIATES.

Amination by ammonolysis [the substitution of the amino-group for other radicals by means of ammonia]. P. H. GROGGINS (Chem. Met. Eng., 1929, 36, 273—275).—The type of reaction referred to is illustrated by the preparation of aniline from chlorobenzene, β -naphthylamine from β -naphthol, etc. It is affected by possible variables as follows. Excess of ammonia of a given concentration minimises the formation of imino-compounds and phenols. Increase of ammonia concentration acts similarly. The reaction velocity is increased by rise in temperature within the working limits for the reaction considered. Stirring sufficiently to mix the materials thoroughly is necessary. In the case of water-insoluble aromatic halogen compounds the use of nitrobenzene, amyl alcohol, etc. as a common solvent is desirable. The use of a copper catalyst should be limited to the production of volatile amines or those that will remain in solution in the ammonia. The value of such catalysts has been much exaggerated.

C. IRWIN.

See also A., June, 693, Esters of aminoazobenzene-sulphonic acids (HANTZSCH). Aminoazobenzenes and their salts (HANTZSCH and VOIGT). 701, Polyoanthraquinones (ECKERT and KLINGER). 702, Reduction products of 1:1'-dianthraquinonyl (ECKERT). 712, Determination of carbon and carbon dioxide (FRIEDEMANN and KENDALL). 713, Determination of amino-acids (ROSENTHALER). 724, Acetaldehyde by fermentation (KISCH and LEIBOWITZ). Conversion of acetic acid into succinic and fumaric acids by *Mucor stolonifer* (BUTKEVITSCH and FEDOROV). 730, Purification of picric acid (BENEDICT).

Phenol from coke-oven effluent. HATCH.—See II. Detection of isopropyl alcohol in presence of acetone. MEYER.—See XVIII.

PATENTS.

Manufacture of methyl alcohol. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 308,181, 19.9.27).—Almost pure methyl alcohol is obtained by hydrogenation of oxides of carbon at ordinary or slightly increased pressure and at 100—250° by using a copper catalyst to which one or more oxides of metals of groups II to VII have been added; metals of group VIII are excluded. A catalyst, prepared by precipitating a solution of copper nitrate (2 mols.) and magnesium nitrate (1 mol.) with sodium hydroxide at 20°, followed by reduction with dilute (5%) hydrogen at 100—250°, converts a mixture of carbon monoxide and hydrogen at 150° and at ordinary pressure into colourless methyl alcohol, d_{15}^{20} 0.792, practically free from unsaturated compounds.

C. HOLLINS.

Manufacture of mono- and poly-hydric alcohols. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 309,200, 28.9.27).—The crude reaction product from aldolisation of aldehydes or ketones (or mixtures of either or both), without separation of the alkaline condensing agent, is hydrogenated in presence of diluents (water, alcohols) and of a catalyst (nickel, cobalt, copper, or mixtures). Aldol from acetaldehyde gives $\alpha\gamma$ -butylene glycol; from *n*-butaldehyde, γ -octanol. The alkaline condensation products from formaldehyde or from formaldehyde and aldol yield syrups on hydrogenation. [Stat. ref.]

C. HOLLINS.

Manufacture of esters and other valuable organic compounds. E. I. DU PONT DE NEMOURS & Co. (B.P. 287,846, 12.3.28. U.S., 26.3.27).—Vapour of ethyl (or higher) alcohol is led over a dehydrogenating catalyst, e.g., copper oxide containing oxides of manganese and magnesium, at 250—500° under high pressure (e.g., 275 atm.). Ethyl alcohol gives ethyl acetate, *n*-butyl alcohol, and acetaldehyde.

C. HOLLINS.

Manufacture of new compounds containing sulphur. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 307,728, 12.12.27).—Secondary alicyclic amines are converted by the usual methods into dithiocarbamates. Examples of amines used are: cyclohexylethylamine, 2-methylcyclohexylethylamine, and cyclohexylmethylamine.

C. HOLLINS.

Production of aldehyde-sulphoxylates. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 308,229, 19.9.27).—Solutions of aldehyde or ketone bisulphite compounds or aldehyde hyposulphite compounds are treated with hydrogen in presence of an activated or unactivated hydrogenation catalyst. To avoid hydrolysis the pH should be 7 or a little above. In the reduction of formaldehyde bisulphite an unactivated catalyst and a high pressure (40—140 atm.) must be used. The preparation of sulphoxylates of formaldehyde, benzaldehyde, *m*-tolualdehyde, acetone, and methyl ethyl ketone is described. The catalysts are nickel, nickel-cobalt, nickel-tungsten, nickel-vanadium, nickel-vanadium-tungsten, or nickel-chromium-tungsten, on kieselguhr.

C. HOLLINS.

Manufacture of methylol [hydroxymethyl] derivatives of urethanes. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 309,108, 3.1.28).—Paraformaldehyde is stirred into a molten urethane (alkyl carbamate) to which a little barium or calcium hydroxide or oxide has been added; nearly quantitative yields of *N*-hydroxymethylurethanes are obtained. Methyl *N*-hydroxymethylcarbamate, m.p. 61—62°, and the corresponding *n*-propyl (m.p. 63—64°), *n*-butyl (m.p. 62—63°), and β -ethoxyethyl, m.p. 59—60°, derivatives are described.

C. HOLLINS.

cycloHexyl compounds and their manufacture. R. ADAMS, Assr. to ABBOTT LABORATORIES (U.S.P. 1,703,186, 26.2.29. Appl., 8.8.27).—cycloHexyl alkyl halides are condensed with substituted malonic esters, or ω -cyclohexylalkylmalonic esters or cyclohexylmalonic esters with alkyl halides and the product is hydrolysed; the malonic acid derivative fused affords bactericidal compounds (cf. A., 1928, 62).

R. BRIGHTMAN.

Catalytic oxidation of organic compounds.

A. O. JAEGER, ASST. to SELDEN Co. (U.S.P. 1,694,122, 4.12.28. Appl., 24.11.26).—Zeolites containing catalytically active elements, such as vanadium, obtained by base exchange methods are used alone or diluted with silica as catalysts, *e.g.*, for vapour-phase organic oxidations. Alternatively, an inert artificial or natural zeolite may be impregnated with a catalyst. Thirty-one examples are given. R. BRIGHTMAN.

Purification of benzoic acid and its derivatives.

A. O. JAEGER, ASST. to SELDEN Co. (U.S.P. 1,694,124, 4.12.28. Appl., 17.10.27).—Benzoic acid or its derivatives are separated from the corresponding phthalic anhydride by treating the mixture with the vapours of an organic solvent for benzoic acid at temperatures at which the benzoic acids are volatile, but below those at which the phthalic acids are transformed into anhydrides. The operation is effected in presence of water or steam and may be continuous.

R. BRIGHTMAN.

Manufacture of benzoates. W. S. CALCOTT and H. W. DAUDT, ASSS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,692,927, 27.11.28. Appl., 13.9.24. Renewed 27.2.28).—Coloured impurities in benzoates made from benzoic acid from phthalic acid are removed by oxidation with permanganate. C. HOLLINS.

Benzoic acid salt of aminobenzoic esters. H. SEYDEL (U.S.P. 1,690,705, 6.11.28. Appl., 5.4.27).—Aminobenzoic esters in dilute hydrochloric acid are added to sodium benzoate solution, giving, *e.g.*, *ethyl p-aminobenzoate benzoate*, m.p. 70—71°. R. BRIGHTMAN.

Purification of nitroaniline. H. MERRILL, ASST. to TOWER MANUF. Co. (U.S.P. 1,692,308, 20.11.28. Appl., 1.7.25).—*p*-Nitroaniline made from chloronitrobenzene is stirred with hot 10% caustic alkali, filtered at 40—50°, and allowed to crystallise. C. HOLLINS.

Hydrogenation of pyridine and/or its homologues. TECHN. RES. WORKS, LTD., and E. J. LUSH (B.P. 309,300, 31.3.28).—Pyridine etc. is passed in the liquid state over active nickel at 180° in a hydrogen atmosphere at 300 lb./in.² The products are piperidine and a little tetrahydropyridine, with no amylamine.

C. HOLLINS.

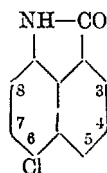
Manufacture of 8-amino-6-alkoxyquinolines. I. G. FARBENIND. A.-G., and A. CARPMAEL (B.P. 307,727, 12.12.27. Addn. to B.P. 267,457; B., 1927, 379).—The 8-amino-group is introduced by Hoffmann decomposition of 6-alkoxyquinoline-8-carboxylamides. *E.g.*, 6-methoxyquinoline-8-carboxylamide, m.p. 169—170°, obtained from the methyl ester, is treated with potassium hydroxide and bromine. C. HOLLINS.

Manufacture of alkali salts of nitrosoamines of primary aromatic amines. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 307,965, 15.12.27).—A diazo solution is run into cooled alkali and the resulting *syn*-diazotate is added to alkali at the appropriate temperature for conversion into *isodiazotate*. The optimum temperatures for this conversion are given for the following amines: *m*-chloroaniline (105°), 3:4-dichloroaniline (110°), 3:5-dichloroaniline (100°), 2:4:5- and 2:4:6-trichloroanilines (100°), 2-chloro-5-

bromoaniline (100°), 5-chloro-*o*-toluidine (115°), 4-chloro-*o*-toluidine (110°), 5:6-dichloro-*o*-toluidine (120°), 2:6-dichloro-*m*-4-xylydine (120°), 4- and 5-bromo-*o*-toluidines (110—115°), 2:5- and 5:2-chloro-bromo-*p*-toluidines (105°), 4-chloro-*m*-toluidine (110°), 4:5-dichloro-*o*-toluidine (120°), 2:5- and 2:3-dichloro-*p*-toluidines (105°), 6-chloro-*o*-toluidine (115°), 3-chloro-*p*-toluidine (105°), 3:6-dichloro-*o*-toluidine (105°), 4:6-dichloro-*m*-toluidine (105°), 3:5-dichloro- and -dibromo-*p*-toluidines (100°), 4-chloro-*m*-anisidine (115—120°), 5-chloro-*o*-anisidine (120°), 4:6-dichloro-*m*-anisidine (100°), 4:5-dichloro-*o*-anisidine (110°), 6-chloro-3-methoxy-*p*-toluidine (115°), α -aminoanthraquinone (120—125°), 2:2'-dichlorobenzidine (100°).

C. HOLLINS.

Manufacture of monohalogenated naphthastyril compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,107, 3.1.28. Cf. B.P. 278,100; B., 1927, 102).—Naphthastyril, boiled with sodium hypochlorite solution, yields the *N*-chloro-compound, m.p. 132°, which is converted by heat or by treatment with acid or in boiling solvents into 6-chloronaphthastyril (annexed formula), m.p. about 270°; this is also obtained



directly by the action of iodine at 15—20°, or from an acid suspension of naphthastyril hydrochloride and sodium chlorate, or by the action of sulphuryl chloride in tetrachloroethane at 50—60°. Bromine in tetrachloroethane at 50—60° in presence of iodine gives 6(?)-bromonaphthastyril, m.p. 256—257°. *N*-Chloro- (m.p. 117°) and 6-chloro- (m.p. 250—252) derivatives of 5-ethoxynaphthastyril are also described. C. HOLLINS.

Production of anthraquinone colouring matters. W. M. MURCH, ASST. to NAT. ANILINE & CHEM. Co. (U.S.P. 1,692,854, 27.11.28. Appl., 2.4.26).—The alkaline fusion of β -aminoanthraquinone for indanthrone is performed in a nickel-steel vessel.

C. HOLLINS.

Manufacture of highly-chlorinated perylenes. A. ZINKE and K. FUNKE, ASSS. to F. BENSA (U.S.P. 1,713,362, 14.5.29. Appl., 11.4.28. Austr., 2.4.27).—See B.P. 288,171; B., 1928, 921.

Preparation of phthaloyl-2:3-thionaphthens. F. MAYER, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,713,670, 21.5.29. Appl., 25.1.27. Ger., 29.1.26).—See B.P. 265,193; B., 1928, 635.

Purification of crude anthracene. SELDEN Co., ASSEES. of A. O. JAEGER (B.P. 304,179, 19.3.28. U.S., 16.1.28).—See U.S.P. 1,693,713; B., 1929, 350.

Manufacture of 1:4-diaryl-amino-5:8-dihydroxyanthraquinone. K. WEINAND, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,713,576, 21.5.29. Appl., 26.6.26. Ger., 3.7.25).—See B.P. 263,370; B., 1927, 136.

Manufacture of benzanthrone compounds. B. STEIN, W. TRAUTNER, and R. BERLINER, ASSS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,713,565, 21.5.29. Appl., 16.9.26. Ger., 8.10.25).—See B.P. 297,129; B., 1928, 847.

Manufacture of Bz-methylbenzanthrones. A. WOLFRAM and H. GREUNE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,695,626, 18.12.28. Appl., 28.11.25. Ger., 4.12.24).—See B.P. 244,120; B., 1927, 326.

1-Phenylbenzanthrone compounds. Cinnamylideneanthrones. 10-Phenylbenzanthrone and its derivatives. R. BERLINER, B. STEIN, and W. TRAUTNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,713,571 and 1,713,590—1, 21.5.29. Appl., [A—C] 16.9.26. Ger., [A, B] 8.10.25, [C] 20.10.25).—See B.P. 297,129; B., 1928, 847.

Preparation of flavanthrone. W. SMITH and J. THOMAS, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,714,249, 21.5.29. Appl., 16.5.27. U.K., 26.5.26).—See B.P. 287,039; B., 1927, 902.

Base-exchange bodies (U.S.P. 1,694,620).—See VII. **Reduction of sugars to alcohols** (U.S.P. 1,712,952).—See XI. **Pinacol** (U.S.P. 1,703,220).—See XX.

IV.—DYESTUFFS.

Analysis of dyestuffs. H. E. FIERZ-DAVID (J. Soc. Dyers and Col., 1929, 45, 133—142).—The dye is first purified and the group to which it belongs ascertained by means of Green's tables. The ultra-violet lamp is valuable for this purpose, and it has been found that dyes containing an -S- group or a thiazole ring invariably show a very strong fluorescence in ultra-violet light. Fluorescence is thus a general property of some classes of dyes. It is shown that a study of the literature, especially the patent literature, is of great assistance when dealing with unknown dyes. The above methods have been applied in the case of such dyes as Brilliant Wool Blue FFR, Alizarine Fast Grey 2BL, and Indocyanine BF. The value of the spectroscopy in dye analysis is also emphasised.

L. G. LAWRIE.

See also A., June, 671, **Device for identifying colours** (HOLMES). 694, **Absorption spectra and composition of azo dyes** (BRODE). 702, **Reactions for cantharidin** (VAN URK). 706, **Pyrimidine dyes** (JOHNSON). 712, **Eosinates** (HOLMES).

PATENTS.

Manufacture of [black benzanthrone] vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,723, 10.12.27).—Black vat dyes are obtained by alkaline fusion of 2-aminobenzanthrones carrying as *N*-substituent a 1-anthraquinonyl residue. Examples are 2-benzanthronyl derivatives of 1-amino-, 1-amino-4-methoxy-, 1:6- or 1:5-diamino-anthraquinone, and condensation products from 2:9-dichlorobenzanthrone and 1-aminoanthraquinone, or from 2:10-dichlorobenzanthrone and 1-amino-4-methoxyanthraquinone.

C. HOLLINS.

Manufacture of [black dibenzanthrone] vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,947, 12.9.27, 16.3. and 19.3.28; cf. B.P. 307,364 and 285,502; B., 1929, 388).—*N*-Substituted amino-dibenzanthrones or -isodibenzanthrones in which the *N*-substituent is an aromatic or heterocyclic residue and may be complex (e.g., residues of perylene, dibenz-

pyrene, anthanthrone, *ms*-anthradianthrone, etc.) and/or may link together two or more aminodibenzanthrone molecules, are valuable black vat dyes. They may be prepared, for example, by condensing the aminodibenzanthrone obtained by reduction of the nitro-compound (B.P. 1818 of 1905; B., 1905, 963) with 1- or 2-chloro-anthraquinone, dibromoanthanthrone, trichloroanthraquinoneacridone, dibromoisodibenzanthrone, dichloro-*allo-ms*-naphthadanthrone, tri- or tetra-bromopyranthrone, nitrobenzanthrone (cf. B.P. 220,212), α -nitro-naphthalene, 1:5-dinitroanthraquinone, chloranil, cyanuric chloride, or with cyanuric chloride and 1:4-amino-methoxyanthraquinone; or of aminodibenzanthrone from the nitro-compound of B.P. 220,212 (B., 1924, 862) with chloranil, or cyanuric chloride and 1 amino-anthraquinone; or of nitrodibenzanthrone with hydrazine, hydrazine and tetrabromopyranthrone, or hydrazine and 1:5-dichloroanthraquinone; or aminodibenzanthrone, from hydroxylamine and dibenzanthrone, with tribromopyranthrone. [Stat. ref.]

C. HOLLINS.

Manufacture of sulphur dye pastes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,061, 31.12.27).—Addition of a carbohydrate (starch, flour) which is swelled by alkali to sulphide dye pastes prevents separation of crystals or water even on long keeping or on addition of the sodium sulphide required for the dyeing operation. Bactericidal agents may be added to prevent mildew; if Chloramine T is used the starch is solubilised during the heating in the dye-bath. The amount of carbohydrate added should be less than 12½% of the weight of dye.

C. HOLLINS.

Manufacture of monoazo dyes [for wool and acetate silk]. I. G. FARBENIND. A.-G. (B.P. 282,683, 20.12.27. Ger., 23.12.26).—Violet to black wool dyes (which when they contain only one sulphonic group also dye acetate silk) are obtained by coupling *p*-hydroxyphenyl- β -naphthylamine (or derivatives) with diazotised *p*-nitroaniline-*o*-sulphonic acid, sulphonic acids of 4-nitro- α -naphthylamine, or 5-nitro-*o*-toluidine- ω -sulphonic acid, or derivatives of these. Examples of coupling components used are *p*-hydroxyphenyl- β -naphthylamine, its 7-sulphonic acid, 2-*p*-hydroxyphenylamino-3-naphthoic acid, and 4'-hydroxy-3'-sulphophenyl- β -naphthylamine. The diazo components are *p*-nitroaniline-*o*-sulphonic acid, 5-nitro-*o*-anisidine-3-sulphonic acid, *p*-nitroaniline-2:5-disulphonic acid, 4-nitro- α -naphthylamine-5- and -6-sulphonic acids, and 5-nitro-*o*-toluidine- ω -sulphonic acid.

C. HOLLINS.

Manufacture of [direct green] azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 308,958, 30.12.27).—An aminoazo compound is diazotised and coupled with 1:2-aminonaphthyl ethyl ether (or derivative) and the product is rediazotised and coupled with an acylated 1:8-aminonaphtholsulphonic acid. Examples are: sulphanilic acid \rightarrow *o*-toluidine \rightarrow 2-ethoxy-Cleve acid \rightarrow *N*-benzoyl-H-acid; *o*-toluidine-5-sulphonic acid \rightarrow *o*-toluidine \rightarrow 2-ethoxy-Cleve acid \rightarrow *N*-*o*-chlorobenzoyl-H-acid; 2:8-naphthylaminesulphonic acid \rightarrow aniline (as its methane- ω -sulphonic acid) or *m*-5-xylylidine \rightarrow 2-ethoxy-Cleve acid \rightarrow *N*-acetyl-H-acid.

C. HOLLINS.

Manufacture of fast azo dyes. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 309,216, 31.12.27).—Dyes of good fastness to light are obtained in substance or on the fibre by coupling diazotised 2 : 4 : 5-trichloroaniline with a 2 : 3-hydroxynaphthoic arylamide. [Stat. ref.] C. HOLLINS.

Manufacture of insoluble azo dyes. I. G. FARBERIND. A.-G. (B.P. 282,682, 20.12.27. Ger., 22.12.26).—2 : 3-Hydroxynaphthoic arylamides are coupled in substance or on the fibre with a diazotised unsulphonated aminocarbazole to give violet pigments or dyeings. Examples are: the β -naphthylamide with 2-aminocarbazole, and the dianisidide with 3 : 6-dichloro-2-aminocarbazole. C. HOLLINS.

Manufacture of [solubilised o-hydroxy] azo dyes. I. G. FARBERIND. A.-G. (B.P. 282,107, 12.12.27. Ger., 13.12.26).—o-Hydroxyazo dyes are treated with chlorosulphonic acid or esters, especially in presence of pyridine or dimethylaniline, to give soluble sulphuric esters. The product from 4-chloro-o-aminophenol \rightarrow β -naphthol dyes wool orange, becoming reddish-brown on chroming. The sulphuric ester of 5-nitro-o-aminophenol \rightarrow 2 : 3-hydroxynaphthoic acid or phenylmethylpyrazolone may be reduced and phosgenated to give a direct cotton dye (blue-violet or red, respectively, after coppering). C. HOLLINS.

Manufacture of [violet] indigoid vat dyes. I. G. FARBERIND. A.-G. (B.P. 283,118, 3.1.28. Ger., 3.1.27. Addn. to B.P. 282,805; B., 1928, 849).— α -Arylamides of 7-methylisatins, carrying a 5-methyl group or, in positions 4 and 5, two methyl, two chlorine, or chlorine and methyl substituents, are condensed with thioindoxyls to give violet vat dyes. The isatin anils are made by Sandmeyer's method from *m*-4-xyldine, ψ -cumidine, 6-chloro-*m*-4-xyldine, 4 : 5-dichloro-o-tolidine, 5-chloro-*p*-2-xyldine. C. HOLLINS.

Manufacture of brown dyes suitable for dyeing wool and leather. R. SCHMIDLIN, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,711,860, 7.5.29. Appl., 13.7.24. Ger., 23.7.23).—See B.P. 219,673; B., 1925, 538.

Manufacture of azo dyes. A. L. LASKA and A. ZITSCHER, Assrs. to I. G. FARBERIND. A.-G. (Re-issue 17,306, 28.5.29, of U.S.P. 1,667,667, 24.4.28).—See B., 1928, 400.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Composition of jute fibre with special reference to the use of chlorine dioxide as an analytical reagent. J. K. CHOWDHURY and P. C. MAJUMDAR (J. Indian Chem. Soc., 1929, 6, 239—251).—Successive treatment of jute fibre with a 2.5% chlorine dioxide solution (Schmidt and Graumann, A., 1921, i, 912) removes almost the whole of the lignone (15%) but does not affect the raw cellulose (about 85%). The value for the lignone content agrees with that obtained using Willstätter and Zechmeister's method, whilst the cellulose content is about 10% higher than is indicated by Cross and Bevan's method. The delignified fibre is of a good tensile strength, but is disrupted by boiling with sodium sulphite or alkalis; with Schweitzer's

reagent swelling first occurs with ultimate dissolution. Removal of the hemicelluloses from the raw cellulose by washing with 17.5% sodium hydroxide solution gives about 71.5% of α -cellulose (60.6% of dry jute), which contains only small amounts of ash and furfuraldehyde. Determinations of the pentosans in raw jute and raw cellulose give values which are almost identical (about 10%), showing that they are unaffected by the chlorine dioxide treatment. Washing the raw cellulose with 5% sodium hydroxide solution removes the gummy material (13.56%), which is isolated by diluting the washings with much alcohol and subsequently acidifying with a slight excess of hydrochloric acid. Partially delignified jute (lignone content about 5%) is less susceptible to washing with alkali than the completely delignified fibre. H. BURTON.

Wood chemistry. V. Mechanical and chemical pulps from "Ezomatsu" (*Picea jezoensis*). Y. UYEDA and I. MITSUHASHI (J. Cellulose Inst., Tokyo, 1929, 5, 98—99).—The results of analyses of the original wood and of the mechanical and sulphite pulps prepared therefrom are given. D. J. NORMAN.

Wet carbonisation of wood and sulphite waste liquor. C. G. SCHWALBE (Papier-Fabr., 1929, 27, 309—311).—The production by Germany alone of about a million tons of cellulose is accompanied by the loss of roughly an equal weight of organic matter in the waste liquors. If this were used on a large scale (e.g., as a source of fuel) considerable saving would be effected. Methods are discussed whereby such recovery may be undertaken which depend on precipitation of the organic matter on absorbent material produced by swelling bark chips with magnesium chloride solution. The concentrated waste liquor is heated under pressures of about 10 atm. with water, or at lower pressure if salt solution is used. Work carried out during the war on the production of sugar and fodder from cellulose showed that on treatment of the latter with sulphuric acid at 70—80° carbonisation occurred readily. Magnesium chloride solutions may be used instead, thereby causing simultaneous swelling and hydrolysis of the material, whilst the wet distillation obtained by use of these solutions under pressure is much more rapid than the ordinary dry distillation. Carbon, acetic acid, and wood spirit are obtained as in the latter process, but no tar or acetone. From coniferous woods the yields (%) obtained by wet and dry distillation, respectively, are: carbon 55, 30 (of calorific values 6800, 8000 kg.-cal.); acetic acid 6, 2; wood spirit 1.2, 0.6; and wood oil —, 2%. Deciduous woods may also be used. The lower calorific value of the carbon from the wet process is of minor importance since the yields of wood spirit and acetic acid are higher. The carbon obtained may be used in the carbonisation of sulphite waste liquors in the proportion of 1 pt. of carbon to 3 pts. of concentrated liquor. Carbonisation of the waste liquor is less satisfactory if the material has previously been submitted to a process of removal of sugars, but proceeds better if the basic material present is first removed by the addition of sulphuric acid, whilst the ash content of the resulting carbon is reduced from 20—22% to 9—12%. Fuel obtained by the methods described may

be converted into briquettes which do not powder on burning. The adaptation of the methods for use on a technical scale is discussed. B. P. RIDGE.

Sclerenchema in straw cellulose. KORN (Papier-Fabr., 1929, 27, 311—314).—In an attempt to find whether the cellulose or the paper manufacturer is responsible for the appearance of sclerenchema particles in paper, experiments were carried out on samples of straw cellulose made by different processes at 12 different works. The particles were collected under standardised conditions, identified under the microscope, and counted. Out of all the samples, which had been prepared under the ordinary conditions of commercial manufacture, only one showed absence of the sclerenchema. The remainder showed numbers ranging from 9 to 93 particles per m.² of test paper; the greater the number, the greater, in general, was their size. Of the test samples, 2 were of high content, 5 of medium, and 4 of small. The presence of large or small amounts cannot be correlated with any particular process of preparation of the cellulose. Cleaning the mincers and the manner of preparation of the cooked straw must be of importance. B. P. RIDGE.

Catalytic action of selenium on calcium bisulphite solution in cellulose production. N. F. JERMOLENKO (Chem.-Ztg., 1929, 53, 343—344).—Under the conditions obtaining during sulphite cooking, free sulphur, which is always present in the cooking liquor, combines with the sulphurous acid to form thiosulphuric and polythionic acids which are themselves decomposed, yielding sulphuric acid and sulphur. Sulphuric acid is especially dangerous in this process since it causes hydrolysis and charring of the cellulose and renders it useless. Pyrites (the starting material for the preparation of the calcium bisulphite used) always contains selenium, which behaves in a similar manner to sulphur but has a catalytic action about 500 times as great. Experiments carried out in an autoclave under normal conditions of cooking, except that increasing amounts of selenium were added, show that a concentration of 0.7 mg. of selenium per litre of cooking liquid renders the cellulose unfit for papermaking after 10 hrs.' cooking at about 140°. At about 130° this concentration causes darkening of the liquor but no charring of the wood shavings used, whereas 1.4 mg./litre causes charring. The higher the concentration of selenium present the greater is the amount of free acid formed, and the greater is the calcium sulphate content of the precipitate formed and the less its calcium sulphite content, thus illustrating the catalytic action of the selenium. B. P. RIDGE.

Viscose. XXIII. Surface tension of viscose during ripening. G. KITA, S. IWASAKI, and S. MASUDA (J. Cellulose Inst., Tokyo, 1929, 5, 122—124).—According to Fukushima and Atsuki the surface tension changes during ripening in a manner similar to the viscosity but to a smaller extent, whilst Mukoyama states (B., 1927, 810) that no change occurs. The authors have examined viscose solutions prepared from three different kinds of cellulose in both 6% and 0.5% cellulose concentration by means of du Noüy's apparatus as used by Fukushima and by the stalagmometer method as used by Mukoyama. In both cases the drop number does not change—as

asserted by the latter worker—but with du Noüy's apparatus a slight fluctuation was observed except in one case. If the difficulty of measuring the surface tension of a 6% solution by means of this apparatus, and that of preparing a homogeneous 0.5% viscose solution, are taken into account, however, it is rational to assume that no change of surface tension takes place.

B. P. RIDGE.

Transverse sections of artificial silk. III. The coagulating bath. I. Y. KAMI and M. NOZAKI (J. Cellulose Inst., Tokyo, 1929, 5, 117—121; cf. B., 1928, 809).—Viscose prepared by the usual process, of cellulose content 8.1—8.15%, sodium content 7—7.05%, and ripeness (ammonium chloride) 8.7—9, was spun under standard conditions into coagulating baths of different concentrations of various acids at 36.5—40°. The following properties of the threads: denier, extensibility (%), strength per denier, cross-sectional area, fullness (%), etc. were measured and correlated with the kind and concentration of acid used. Generally speaking, viscose spun from a bath containing acid alone has a circular cross-section. Concentrated acid coagulates strongly and rapidly, hence the thread is white but matt; its cross-section has many boundary fissures and a non-uniform, uneven circumference. These cracks are the smaller the more dilute is the acid, until finally they disappear and the silk has the normal lustre and good tearing strength and extensibility like the commercial material. The percentage "fullness" of the sections increases with increasing dilution of the acid. Threads spun in solutions of acids which react with the sodium in the xanthate have a greater percentage fullness than is obtained for solutions of other acids; thus the percentage fullness given by the use of acetic acid is greater than that for sulphuric, hydrochloric, or nitric acid. Relatively concentrated nitric acid baths give lustrous threads of high strength with a somewhat different cross-section. Extensibility increases, and tearing strength diminishes very slowly, with increasing dilution of the acid in the bath. B. P. RIDGE.

Analytical detection of the metal-corrosive capacity of papers. L. KALB and F. FRH. VON FALKENHAUSEN (Papier-Fabr., 1929, 27, 330—333).—The behaviour of papers placed in contact with metals depends on the content of acidic ingredients of the paper; the latter can be determined more quickly by titration or p_H measurement than by measurement of metal-corrosive capacity. The latter has been determined for various parchment and parchment-substitute papers, and the results are correlated with those obtained by titration and p_H experiments. Titration was carried out with standard sodium hydroxide solution on the aqueous extract obtained after steeping a sample of the paper for 24 hrs., and the number of c.c. of 0.1N-solution used per 100 g. of paper is called its "acid coefficient." For the determination of p_H the spotting method of Schleicher and Rössler (B., 1924, 553) was found less satisfactory than the Wulff foil colorimeter method. In the metal contact tests copper was used, since it has the advantage of showing characteristic temper colours according to the severity of attack. Strips of the paper were clamped between strips of clean copper foil and exposed in a glass vessel to a

controlled atmospheric humidity of 90–95% until the first appearance of a colour patch corresponding with a certain stage of attack. Four such stages are identified and described which vary in intensity with the acidity of the paper and the time of treatment. The test is generally completed in 7–10 weeks. All papers showing a p_H less than 5 by Wulff's test are injurious to metal and all of 5 or over are non-injurious. (One exception is quoted in which a paper of p_H 5.4 showed a very slight corrosive effect.) Whilst low acidity coefficients correspond, in general, with no corrosion, high coefficients do not necessarily indicate excessive corrosion effects. The corrosive capacity of sulphide sulphur has been investigated by the use of zinc sulphide. This substance alone is corrosive and its action is increased by the presence of alum; addition of zinc oxide, however, prevents such attack. Papers which contain filling material of a basic nature, in spite of having a high acidity coefficient, may on account of their small hydrogen-ion concentration be non-corrosive to metals. In these cases the titration indications are valueless and a p_H method gives the best results. B. P. RIDGE.

See also A., June, 684, **Lignin and cellulose** (HESS). **Cellulose benzyl ethers** (NAKASHIMA). **Cellulose-amine and celluloseaniline** (SAKURADA).

Dyeing of tissue paper. LANDOLT.—See VI. **Sugar factory filter-cloths.** ŠANDERA.—See XVII.

PATENTS.

Artificial wool. G. M. ROSSATI and G. DE BLASIO (U.S.P. 1,703,693, 26.2.29. Appl., 18.1.28).—Vegetable fibres, preferably jute or other fibre of the *Tiliaceae* family, are immersed for 2 hrs. in clear 1% lime water and treated with sulphur dioxide for 1 hr. After 12 hrs. rest the fibre is washed, dehydrated, treated with sodium hydroxide solution (d 1.075), and dried. The dried fibre is washed, bleached, fixed in 0.25% sodium bisulphite solution, and, after washing in 0.5% hydrochloric acid, is immersed in a sulphonated fatty acid bath. R. BRIGHTMAN.

Aircraft covering. Gas-retaining fabric. I. M. JACOBSON and S. TRUSCOTT (U.S.P. 1,706,294–5, 19.3.28. Appl., [A] 11.4.25, [B] 30.9.25).—(A) A fabric treated with a mixture of regenerated cellulose and rubber dispersed in an organic solvent is claimed. (B) The fabric is treated with an aqueous solution of a soluble compound of cellulose glycolate. A. R. POWELL.

Manufacture of chloroform-soluble cellulose acetate. H. LE B. GRAY and C. J. STAUD, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,690,632, 6.11.28. Appl., 6.6.27).—Cellulose is treated below 30° with less than 2.5 pts. of acetic anhydride, but more than the theoretical quantity, in the presence of a catalyst, preferably sulphuric acid and phosphoric acid. The anhydride is of at least 85% concentration. R. BRIGHTMAN.

Cellulose esters containing nitro-groups and halogen-substituted acyl groups. H. T. CLARKE and C. J. MALM, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,690,621, 6.11.28. Appl., 14.5.27).—Nitrocellulose is treated with an unsaturated acid, and the mixed cellulose is halogenated. As example, nitrocellulose (11% N) in

chloroacetic anhydride is treated at 60–65° with crotonic acid in presence of magnesium perchlorate, and the nitrocellulose crotonate is treated with bromine in acetic acid giving a product containing 17.8% Br and 6.8% N. R. BRIGHTMAN.

Composition containing cellulose derivatives. J. G. DAVIDSON, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,693,746, 4.12.28. Appl., 29.4.26).—Polyglycols, e.g., diethylene glycol, b.p. 250°, and triethylene glycol, b.p. 290°, are used as solvents for cellulose esters, e.g., nitrocellulose. R. BRIGHTMAN.

Treatment of fibres. A. E. MILLINGTON (U.S.P. 1,708,586, 9.4.29. Appl., 18.8.26).—Waste wood products which have been disintegrated and screened are softened by cooking in a solution of salt and soda ash with steam under pressure; the mass is refined by shredding and made into building boards. F. R. ENNOS.

Recovery of valuable products from spent sulphite liquors. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,710,272, 23.4.29. Appl., 6.1.27).—Spent liquor from the digestion of cellulose material in a sulphurous acid solution of ammonium salts is neutralised with basic calcium phosphate and, after addition, if desired, of potash in a suitable form, is evaporated, and the dry residue containing all the organic matter removed from the cellulose material is used as a fertiliser. F. R. ENNOS.

Wood fibre for high-grade paper and derivative manufacture. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,709,322, 16.4.29. Appl., 7.11.24).—By suitable treatment of unbleached sulphite pulp, a cellulose wood pulp of high whiteness is produced containing 93.5–96% of α -cellulose, 2–4% of β -cellulose, 2–3% of γ -cellulose, not more than 2.4% of pentosans, and giving a copper number of 1.0–2.3, a reddish-purple colour when subjected to Herzberg fibre stain, and a solubility of not over 9% in 3% boiling sodium hydroxide. F. R. ENNOS.

Sizing of paper. CONTINENTALE A.-G. F. CHEMIE, Asses. of A. ILLOCH (G.P. 454,005, 28.1.26).—An emulsion obtained by the saponification of pinabietic acid is used. E.g., the acid is saponified by means of sodium hydroxide or carbonate solution, and the mixture, after being boiled for about $\frac{1}{2}$ hr. until free from acid crystals, is poured into water and forms a rosin milk. B. P. RIDGE.

Liquidation of natural silk threads. T. MUTO and S. HIDA, Assrs. to KANEGA-FUCHI BOSEKI KABUSHIKI KWAISHA (U.S.P. 1,714,039, 21.5.29. Appl., 21.2.28. Japan, 15.12.27).—See B.P. 306,699; B., 1928, 352.

Production of textile fibres of high quality from skins of fish such as, in particular, those of the *Chondropterygii selachii*. A. EHRENREICH (U.S.P. 1,713,036, 14.5.29. Appl., 24.1.28. Belg., 27.1.27).—See B.P. 284,297; B., 1928, 668.

Industrial treatment of coconuts and their constituents, particularly coconut fibres. B. G. H. VAN DER JAGT, Assr. of Z. VAN DER BERGH and F. A. J. VAN KUYK (U.S.P. 1,713,681, 21.5.29. Appl., 29.1.26. Holl., 2.2.25).—See B.P. 246,837; B., 1927, 472.

[Mechanical] preparation of flax, hemp, and like fibrous materials for spinning. LINEN INDUSTRY RES. ASSOC., and J. A. MATTHEW (B.P. 311,883, 30.3.28).

[Pumping] apparatus for use in manufacture of artificial silk or other operations in which liquids are supplied under pressure. BRIT. CELANESE, LTD., and E. KINSELLA (B.P. 311,670, 9.2.28).

Drying paper or the like in the form of continuous webs, and papermaking machinery therefor. E. A. ÖHLIN (B.P. 285,914, 23.2.28. Swed., 26.2.27).

Emulsifiable oil (U.S.P. 1,695,197).—See II. Wood fibre-asbestos product (Swiss P. 122,820).—See IX. Oils for fabrics, paper, etc. (B.P. 311,786).—See XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effect threads. J. G. GRUNDY (J. Soc. Dyers and Col., 1929, 45, 142—148).—A tabular synopsis is given classifying the properties of the main groups of dyes suitable for the production of effect threads on mixed fibres. These tables include lists of acid, Neolan, and chrome colours dyed on wool with resists on cotton, viscose, passivated and immunised cotton, acetate silk, and silk, together with direct colours dyed on cotton with resists of wool, silk, acetate silk, and passivated and immunised cotton. Formulæ are also given for dyeing the various classes of dyes in order to obtain the best resist with various effect threads.

L. G. LAWRIE.

Dyeing of tissue paper with acid dyes fast to water. A. LANDOLT (Papier-Fabr., 1929, 27, 357—362).—Direct dyes on tissue paper give, as a rule, dull shades, whilst acid dyes, although bright and fast to light, in many instances give dyeings which are not fast to water. Many acid dyes, however, can be made fast to water by dyeing the paper by the dipping method and after-treating the dyed paper with aluminium acetate. The amount of the acetate to be added should be sufficient to ensure fixation of the dye, but insufficient to cause precipitation of dyestuff in the dyebath; suitable proportions are: 10 g. of dyestuff, 400 c.c. of 5% aluminium acetate solution, and 1 litre of water. Lake formation can be prevented with many dyes by increasing the hydrogen-ion concentration by the addition of 20—50 c.c. of acetic acid per litre of dyebath. Suitable dyes are Kiton Yellow S, Kiton Yellow 3GN, Paper Red PSN, Benzyl Red B, Brilliant Benzyl Violet 3B, Cloth Fast Brilliant Red 4B, Pure Blue BSI, Benzyl Green B, and Acid Black HA. The fastness to water is tested by soaking the dyed paper in distilled water for 10 min. and comparing the result with an untreated portion. A dyeing fast to water should neither change in shade nor stain the water. The handle of paper treated with aluminium acetate is somewhat firmer.

L. G. LAWRIE.

See also A., June, 641, Adsorption of soap solutions (NONAKA; MIKUMO). 645, Diffusibility and dispersibility of dyes (PISCHINGER). 671, Device for identifying colours (HOLMES).

Comparison of colours. TOUSSAINT.—See I. Dye-wood extracts. BRECHT.—See XV.

PATENTS.

Weighting of natural silk. I. G. FARBERIND. A.-G. (F.P. 631,082, 17.3.27).—In the usual tin phosphate-silicate process, phosphate solutions produced from pure phosphoric acid are used. B. P. RIDGE.

Manufacture of softener for silk. R. E. ROSE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,691,994, 20.11.28. Appl., 17.11.25).—Diarylguanidine salts of sulphonated castor oil are used as softeners or lubricants for weighted silks. C. HOLLINS.

Cross-dyeing of cellulose fabric. J. C. WATSON, Assr. to B. B. & R. KNIGHT CORP. (Re-issue 17,295, 14.5.29, of U.S.P. 1,629,769, 24.5.27).—See B., 1927, 553.

Fluid treatment of artificial silk threads or filaments. J. BRANDWOOD (B.P. 311,803, 17.2.28).

Bleaching of fatty acids (G.P. 454,308). Bleaching of fish oils (B.P. 311,665).—See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Recent developments in the manufacture of sulphuric acid. S. F. SPANGLER (Ind. Eng. Chem., 1929, 21, 417—421).—The following developments and tendencies are noted. (1) The introduction of a mixing nozzle whereby anhydrous ammonia can be discharged into water, thus allowing ammonia for oxidation units to be transported anhydrous and used as liquor. (2) The development of the two-stage submerged-pipe method of concentrating sludge acid from oil refineries. Cottrell precipitators are used with silicon-iron tubes. (3) The introduction of the vanadium mass in contact plants: eight plants to use the improved "Selden mass" are now under construction in America. They incorporate a new type of sulphur burner burning molten sulphur and having a centrifugal air-blower which provides all the draught required. The sulphur dioxide heats a steam boiler before entering the converter; the sulphur trioxide is cooled and passed through three absorbing towers, through the first two of which acid is circulated, whilst the last is a filter. This plant is considered likely to produce 95% acid at a lower cost than any other type. C. IRWIN.

Determination of the purity of potassium and sodium ferrocyanides by titration with zinc sulphate solution. FARBSALZ-GES.M.B.H. (Chem.-Ztg., 1929, 53, 399).—A solution of 1 g. of the salt in 150 c.c. of water and 10 c.c. of 0.1N-sulphuric acid is titrated at 15—20° with 0.2N-zinc sulphate solution, the end-point being determined by means of a spot test on filter paper impregnated with ferric ammonium sulphate solution; no blue colour should develop in 2—3 min. The solution should be standardised against the corresponding pure salt as the titre with potassium ferrocyanide is not the same as that with the sodium salt.

A. R. POWELL

Recovery of bromine from sea water. C. M. A. STINE (Ind. Eng. Chem., 1929, 21, 434—442).—In the recovery of bromine from sea water (which contains 60—70 p.p.m. of bromine) as tribromoaniline the reaction is $3\text{Br}^- + 3\text{Cl}_2 + \text{PhNH}_2 = \text{C}_6\text{H}_2\text{Br}_3\text{NH}_2 + 3\text{H}^+ + 6\text{Cl}^-$. Chlorine in sea water at this concentration

is converted into hypochlorous acid to the extent of 73%, but by the addition of 200 p.p.m. of sulphuric acid this hydrolysis is reduced to 15% at 25°. The aniline is used in the form of the sulphate. The mixing of the acidulated chlorine solution with the aniline salt solution must be as rapid as possible in order to avoid oxidation losses. An experimental shore plant treating 25 gals./min. in a continuous pipe-line plant gave yields of 40–50%, the tribromoaniline being largely contaminated with sand. This latter point determined the further development of the process on board ship. The s.s. *Ethyl* was fitted up to treat 7000 gals. of sea water per min. Sulphuric acid and aniline were stored in steel tanks and handled by means of compressed air, liquid chlorine in 1-ton drums. The mixing devices used included large injection rings and turbulence chambers. The chlorinated water was handled by rubber-lined pumps and tanks. Aniline was converted into aniline sulphate solution (15%) in a lead-lined tank daily, and further diluted by the recirculation of one third of the treated water. The total time of passage of the sea water through the three mixing systems was 10 sec. It was then filter-pressed. The process depends on accurate feed control, and certain difficulties were experienced on the first and only voyage through interference with this by corrosion. It is, however, believed that these could be readily remedied.

C. IRWIN.

See also A., June, 626, Phosphorescent calcium tungstate (SCHLEEDER and TSAO). 650, System potassium perchlorate-sodium nitrate or chloride-water. System sodium nitrate-sodium chloride-potassium perchlorate-water (CORNEC and NEUMEISTER). 662, Phosphorescent alkaline-earth compounds (VANINO and SCHMID). 666, Ultra-violet colorimetry and fluorescent substances (EISENBRAND). 667, Determination of phosphoric acids (SOMEYA; STOLLENWERK and BÄURLE; DWORZAK and REICH-ROHRWIG). 668, Determination of arsenic (VILLECZ; GNESIN). Test for boric acid and borates (DODD). Detection of alkali metals in salts (TANANAEV). Determination of metals as sulphates (GUNTZ and BARBIER). Detection of potassium (REED and WITHROW). Determination of beryllium in rocks (DIXON). 669, Volumetric determination of thallous salts (JILEK and LUKAS). 672, Containers for caustic solutions (SOULE). 713, Determination of halogens in organic substances (ROMAN).

Stainless iron nitric acid equipment. MITCHELL.—See X. Analysis of three-component systems. TÄUFEL and RUSCH.—See XII. "Kalkammon." NEHRING. Acidimetric titration of silica. KÜHN.—See XVI. Germicidal washing solutions. MYERS.

PATENTS.

Manufacture of sulphur trioxide and sulphuric acid. R. TERN (B.P. 311,862, 8.3.28).—Nitrogen oxides produced from air and formed on non-conductors at the end of current conductors, by the action of high-tension currents or fields, are employed to effect the catalytic oxidation of sulphur dioxide to sulphur trioxide under the action of similar currents or fields. Steam and warm air may be admitted to the oxidation chamber, and the

treated gas mixture may be sucked into an electric filter.

J. S. G. THOMAS.

Production of nitric acid. C. C. SMITH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,553, 10.5.28).—Nitrogen oxides, preferably under pressure, from the oxidation of ammonia are absorbed in dilute nitric acid, the resulting acid is concentrated by indirect contact with the hot burner gases, and the condensate (2–3% nitric acid) is used for further absorption, which may be brought about by injecting the acid into the hot burner gases before or after their passage through the heat-exchanger of the evaporator.

W. G. CAREY.

Synthetic production of hydrochloric acid. W. HIRSCHKIND and C. W. SCHEDLER, Assrs. to GREAT WESTERN ELECTRO CHEM. CO. (U.S.P. 1,695,552, 18.12.28. Appl., 14.9.25).—Chlorine and steam are passed through hot carbon in approximately the proportion required by the equation: $C + 2H_2O + 2Cl_2 = 4HCl + CO_2$, a ratio of 7–9 cub. ft. of carbon to 100 lb. of chlorine entering being maintained in the reaction zone.

R. BRIGHTMAN.

Calcination of alkaline-earth materials. W. CROW, Assr. to DITTLINGER CROW CO. (U.S.P. 1,710,967, 30.4.29. Appl., 5.3.23).—Alkaline-earth carbonates are treated with superheated steam (100 lb./in.²) at 320°, then subjected to the action of air sufficiently heated to decompose the hydroxide formed.

H. ROYAL-DAWSON.

Manufacture of beryllium oxide. R. C. PRICE and H. S. COOPER, Assrs. to BERYLLIUM CORP. OF AMERICA (U.S.P. 1,710,840, 30.4.29. Appl., 13.12.23).—Beryllium mineral is heated with a flux which includes a calcium compound, sulphuric acid is added, and the product is dehydrated, dissolved in water, evaporated to $d\ 1.31$, and the precipitated calcium sulphate separated.

W. G. CAREY.

Production of titanite oxide. L. E. BARTON and C. J. KINZIE, Assrs. to TITANIUM PIGMENT CO., INC. (U.S.P. 1,695,270, 18.12.28. Appl., 16.2.25).—Titaniferous ores are heated with sulphuric acid and nitre cake (or other alkali sulphate) at 200–350°, the titanite oxide and sulphates are dissolved out, and the former is separated in known manner.

R. BRIGHTMAN.

Production of lead arsenate. A. F. MEYERHOFER (Swiss P. 122,353, 22.7.24. Ger., 20.12.23).—The compound is formed by the interaction of lead fluosilicate and, e.g., calcium arsenate in accordance with the equation: $3PbSiF_6 + 2CaHAsO_4 = Pb_3(AsO_4)_2 + 2CaSiF_6 + H_2SiF_6$.

L. A. COLES.

Treatment of phosphorites with a low phosphoric acid content. E. ORLOV (Russ. P. 3464, 31.8.27).—A suspension of the phosphorites in water or dilute ammonia is treated with gases containing sulphur dioxide and, after filtration, the solution obtained is heated to expel excess sulphur dioxide and to cause separation of calcium and ammonium phosphates, sulphates, and sulphites.

L. A. COLES.

Base-exchange body. A. O. JAEGER, Assr. to SELDEN RES. & ENG. CORP. (U.S.P. 1,694,620, 11.12.28. Appl., 28.2.27).—Substances, obtained by base-exchange methods, which are free from combined silicon and contain catalytically active elements in a non-exchange-

able form and at least one exchangeable base other than alkali metal are used as catalysts, *e.g.*, for organic oxidations, hydrogenations, etc. Examples are given of potassium vanadate, tungstate, chromate, molybdate, or tantalate, treated with, *e.g.*, vanadyl sulphate, in presence or absence of diluent siliceous material, infusorial earth, or of ferric, silver, or copper oxides, base-exchange reaction taking place in neutral, acid, or alkaline solution. Many other examples are given. R. BRIGHTMAN.

Catalytic agent and its manufacture. W. A. PATRICK, Assr. to SILICA GEL CORP. (U.S.P. 1,695,740, 18.12.28. Appl., 5.5.21).—Silica gel which has not been completely dried is impregnated with a suitable solution (*e.g.*, ammonium chloroplatinate, ferric acetate) and dried at high temperature. C. HOLLINS.

Preparation of moisture-absorbing material. W. R. ORMANDY, T. J. I. CRAIG, and P. SPENCE & SONS, LTD. (B.P. 311,578, 15.6.28).—An absorbent mass which can be many times revived at comparatively low temperatures is formed from aluminous silicates in lump form, which are treated with mineral acid to remove most of the alumina, a hard porous mass being left after extraction and washing. This mass is impregnated with a solution of an absorbent (*e.g.*, calcium chloride) and heated till practically all the solvents used are evaporated. Calcination may be employed before or after treatment with mineral acid. B. M. VENABLES.

Production of hydrogen. J. S. BEEKLEY, Assr. to LAZOTE, INC. (U.S.P. 1,711,036, 30.4.29. Appl., 10.9.26).—Oxygen and a reducing gas are added to a mixture of steam and a gaseous hydrocarbon, and the mixture is passed over a heated catalyst. W. G. CAREY.

Production of hydrogen and gases containing hydrogen. K. GORDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,299, 31.1.28).—A process is described for removing carbon dioxide from mixtures with hydrogen, carbon monoxide, etc. The gases are scrubbed with water under pressure (*e.g.*, 200 atm.). Partial release of the pressure (to 20 atm.) separates gases richer in hydrogen, leaving in solution gases richer in carbon dioxide. The process may be repeated to effect further separation. J. A. SUGDEN.

Liquefying and separating the constituents of gaseous mixtures at low temperatures. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 287,558, 15.3.28. Fr., 25.3.27. Addn. to B.P. 281,675; B., 1928, 603).—Oxides of nitrogen are removed from gas mixtures prior to liquefaction by hydrogenating them. The process is carried out at a temperature between 100° and 200° (or at any rate below a moderate upper temperature) in the presence of a catalyst such as reduced copper or reduced iron, and is effected when the proportion of oxides of nitrogen is very small relatively to the hydrogenating constituents present. Acetylene will also become hydrogenated under similar conditions. Another method of removing the oxides of nitrogen is to wash the gas mixture with a hot solution of ammonium sulphate, with the liberation of nitrogen and formation of sulphuric acid. B. M. VENABLES.

Production of sulphur. A. P. THOMPSON, Assr. to GEN. CHEM. CO. (U.S.P. 1,695,068, 11.12.28. Appl., 16.12.26).—Sulphur is recovered from gases containing sulphur dioxide by reducing the latter with carbonaceous fuel in presence of a controlled amount of carbon dioxide at temperatures sufficiently high for the endothermic reduction of carbon dioxide to carbon monoxide by the fuel to take place. The endothermic reduction prevents the temperature rising high enough to fuse the ash and cinder. The exit gas may be used in part as source of carbon dioxide. R. BRIGHTMAN.

Manufacture of aluminium oxide from aluminium sulphide. C. VON GIRSEWALD (U.S.P. 1,713,411, 14.5.29. Appl., 28.2.28. Ger., 4.3.26).—See B.P. 294,079; B., 1928, 710.

Brine evaporation (B.P. 309,104—6). Anti-freezing composition (U.S.P. 1,711,324). Conveyance etc. of liquid gases (B.P. 280,569).—See I. Spent sulphite liquors (U.S.P. 1,710,272).—See V. Fertilisers (Norw. P. 42,724 and Swiss P. 122,821).—See XVI.

VIII.—GLASS; CERAMICS.

Clays. XIII. Flocculative action of hydrophilic sols on suspensions of clays and its practical uses. T. OKAZAWA and T. SANO (Bull. Inst. Phys. Chem. Res., Tokyo, 1929, 8, 356—368).—Suspensions of clay are readily flocculated by the addition of small quantities of hydrophilic colloids. The flocculation values for casein, albumin, gum arabic, and starch are small, gelatin has a high value, but the most effective agent is a sol of "Konnyaku-ko" (a powder obtained from the tuberous root of the devil's tongue), which precipitates clay suspensions when present at a concentration of only 2 p.p.m. An acid suspension of clay is more susceptible to flocculation than an alkaline suspension. Suspensions of charcoal in water are difficult to flocculate by hydrophilic colloids, but are readily flocculated thereby after addition of a small amount of clay. Clay particles in non-aqueous media also are flocculated by hydrophilic colloids. The precipitated particles are much coarser than those flocculated by electrolytes, and the method is therefore applicable to the acceleration of filtration in industrial processes. E. S. HEDGES.

See also A., June, 637, **System silica and its stability regions** (VAN NIEUWENBURG). 650, **Ceramics of highly refractory substances** (RUFF and others). 668, **Detection of alkali metals in silicates** (TANANAEV). 669, **Detection of magnesium in silicates** (LEITMEIER and FEIGL). 671, **Calibration of Lovibond glasses** (PRIEST and others).

PATENTS.

Continuous-tunnel kiln. T. G. McDUGAL, Assr. to AC SPARK PLUG CO. (U.S.P. 1,710,995, 30.4.29. Appl., 11.3.25).—The ware passes through a zone of steady graduated temperature directly heated by the counter-flow of combustion gases. J. A. SUGDEN.

Continuous kiln. H. R. STRAIGHT, Assr. to E. H. STRAIGHT (U.S.P. 1,711,910, 7.5.29. Appl., 28.9.26).—The upper corners of the stack of ware on the truck fit into longitudinal grooves in the arch of the kiln. Adjustment for the track is provided. J. A. SUGDEN.

Refractory. J. M. LAMBIE and D. W. ROSS (U.S.P. 1,712,005, 7.5.29. Appl., 1.2.24).—Sufficient finely-divided cyanite is added to counteract the firing shrinkage of the other ingredients. J. A. SUGDEN.

Manufacture of dense and solid articles or appliances of zirconia or other highly-refractory oxides. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 292,529, 5.4.28. Ger., 21.6.27).—To the refractory oxide is added 5% of magnesia and a solution of a salt of the refractory oxide to the extent of the equivalent of 5% of the magnesia. J. A. SUGDEN.

Manufacture of translucent, hollow glass articles, particularly of milk-glass bulbs, for electric incandescence lamps. M. PIRANI, ASSR. to PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN (U.S.P. 1,713,394, 14.5.29. Appl., 8.3.28. Ger., 24.3.27).—See B.P. 287,545; B., 1929, 55.

IX.—BUILDING MATERIALS.

Effect of temperature on the setting times of cements, and on the strength of cements, mortars, and concretes. W. W. THOMAS and N. DAVEY (Dept. Sci. Ind. Res., Bldg. Res., Spec. Rept. No. 13, 1929, 38 pp.).—The setting of Portland cement is invariably accelerated by rise in temperature, but there is no direct connexion between the actual times and the variations. Rapid-hardening cements were found to behave similarly. One exception may be due to consistency changes in the wet mix rather than to setting. Aluminous cements which evolve heat on setting have given conflicting results. A slight reduction of the strength of concrete with rise of temperature during setting is perhaps due to increased internal stresses. The strength obtained by curing for a given time at 1.5° was found to be only half that with the same treatment at 16°. Rapid-hardening cements behave similarly. Laboratory tests with aluminous cements again show a less strength at low temperatures, though thermal effects make comparison with large-scale work difficult. If only the earlier part of the treatment of any cement is at low temperature, full strength may or may not be attained. Different results have been recorded as to whether any hardening occurs below 0°. In most cases a period of freezing during curing slows down the rate of increase of strength when the temperature has again become normal. This applies also to aluminous cement. There is some evidence to show that the effect of freezing is more severe the earlier after mixing it occurs. Experimental data on alternate freezing and thawing are uncertain. Increased cement or water content of mix increases the damage to Portland cement concrete, and probably to aluminous cement concrete. There is some evidence to show that unduly high temperatures may cause failure with aluminous cement. C. IRWIN.

Hydraulic moduli and resistance limits of cement. S. GIERTZ-HEDSTRÖM (Svensk Kem. Tidskr., 1929, 41, 119—124).—A review of the various formulæ which have been employed to obtain a "hydraulic modulus" for Portland cement. In the case of hydraulic slags a new modulus is proposed, calculated from

the amounts of the components of the slag which are attacked and unaffected by water respectively. If the slag is to be hydraulic, the ratio between the two groups must be greater than 1, i.e., $(\text{CaO} + \text{MgO} + \text{CaS} + \text{SO}_3 + \dots)/(\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{FeO} + \text{MnO} + \dots) > 1$. It is claimed that the above formula affords a better measure of the hydraulic value of the slag than the one usually employed, i.e., $(\text{CaO} + \text{MgO})/(\text{SiO}_2 + \text{Al}_2\text{O}_3)$.

H. F. HARWOOD.

PATENTS.

Manufacture of light-weight concrete. R. CROSS and W. A. COLLINGS, ASSRS. to SILICA PRODUCTS CO. (U.S.P. 1,710,921, 30.4.29. Appl., 25.3.25).—A hydraulic cement and mineral filler are mixed with gelatinising clay, the last-named serving to hold the aggregate in suspension. J. A. SUGDEN.

Treatment of wood. J. MAXIMOFF and M. S. DE COSTA (F.P. 629,558, 6.5.26).—Gases and liquids for drying, extracting, or impregnating wood are charged into the wood in the direction of the natural channels in it, so that the processes are effected rapidly. The apparatus is so devised that each piece of wood can be treated at a desired temperature and pressure.

L. A. COLES.

Construction of road and like surfaces. C. W. SHARROCK (B.P. 311,751, 16.2.28).—Suitably-graded aggregate mixed with powdered bitumen, with or without the addition of an oil, is laid cold on the surface of the road, and then hot-rolled at 120—180°.

H. ROYAL-DAWSON.

Product containing wood fibre and asbestos. J. FAHRNI (Swiss P. 122,820, 5.6.26).—Purified asbestos fibres are treated in a bath containing powdered metallic oxide, tannin, water-glass, formaldehyde, and ammonium chloride, and wood fibre in one containing ammonium chloride, magnesium chloride, boric acid, and sulphite-cellulose waste liquor. After squeezing or filtration, the fibres are mixed and pressed into sheets. The product is useful as insulating or building material.

B. P. RIDGE.

Production of hydraulic cement composition. L. FORSÉN (U.S.P. 1,712,818, 14.5.29. Appl., 3.6.26. Finland, 24.10.24).—See B.P. 272,163; B., 1927, 909.

Preservation of timber etc. A. M. KOBIOŁKE (U.S.P. 1,713,388, 14.5.29. Appl., 27.7.27. Austral., 10.8.26).—See B.P. 295,126; B., 1928, 713.

Brick kilns. A. HABLA (B.P. 311,884, 30.3.28).

[Fixing of] roof, wall, and other surface coverings of corrosion-proof metals or alloys, especially copper. METALLGES. A.-G. (B.P. 292,621, 23.6.28. Ger., 23.6.27).

Fluid-storing material (U.S.P. 1,705,482—4).—See I. Treatment of fibres (U.S.P. 1,708,586).—See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Corrosion in cast-iron main pipes. J. R. BRADSHAW (Gas J., 1929, 186, 593—596).—Eight samples of cast-iron pipes from water and gas mains, under varying conditions of time and soil, were examined inside

and out for characteristics of fracture, deposit, corrosion, pitting, wear, etc. Analyses of inside and outside deposits showed essentially brown hydrated ferric oxide with varying amounts of ferrous oxide, and for gas mains carbonaceous and organic matter. Outside corrosion was typical of electrolytic soil action and the product was ferrous oxide. Microstructure examination indicated corrosion emanating from graphite nuclei, whilst in several cases polishing started an etching attack round the graphite areas. The varying amounts of corrosion in the samples show the necessity of obtaining a standard quality of high-density iron which will give an almost indefinite life when buried underground. For this purpose spun pipe may be found preferable. The influence of cold weather on tensile strength and the stress and strain on cast-iron pipes due to modern traffic are subjects which should not be ignored.

C. B. MARSON.

Corrosion—a problem in protective coatings.

F. N. SPELLER (Ind. Eng. Chem., 1929, 21, 506—510).—Observations on failures in large pipe systems, *e.g.*, gas services, indicate that there is little difference in the rate of corrosion of steel and iron in soil, and that the method of manufacture or the quantity of foreign inclusions within wide limits has little influence. Copper in excess of 0.15% in steel appears to exert a protective effect towards exposure to air not evident with any other additional elements. For the protection of underground pipe paints have little value, but linings of Portland cement or bitumen reinforced with sand are used extensively, often being applied by centrifugal action. Where cost is not of primary importance, enamelled or rubber-lined pipes are available, or more resistant metals may be used, particularly an alloy containing 18% Cr and 8% Ni.

C. A. KING.

Metallic materials of construction for chemical engineering equipment. E. P. PARTRIDGE (Ind. Eng. Chem., 1929, 21, 471—476).—Alloys of iron with high chromium content have proved resistant at high temperatures to the influence of sulphur gases and to scaling; in the artificial silk industry the action of mixed acids has been uneven. Chromium-nickel-iron alloys (*e.g.*, Staybrite steel) are used increasingly in high-pressure oil-cracking stills and dairy and preserved food equipment, as are also aluminium and nickel containers. For resistance to acids a group of alloys which might be classed as bronzes is available, and a high-zinc alloy, Zilloy, has recently been developed for roofing the construction of industrial plants. Coated metals possess advantages in certain fields, but suffer from the fundamental disadvantage that even a minute fracture in the coating may cause the ruin of a whole equipment.

C. A. KING.

Stainless iron nitric acid equipment.

W. M. MITCHELL (Ind. Eng. Chem., 1929, 21, 442—445).—Stainless irons resistant to nitric acid contain 16—20% Cr and under 0.10% C. They are suitable for all synthetic nitric acid plant, but are attacked by the halide acids present in Chili saltpetre. Riveting presented difficulties at first owing to the brittleness of the alloy if overheated, but the technique is now understood. Riveting is, however, only done in the shop, which

limits the size of absorption towers etc. Welding is usually excluded owing to the risk of local corrosion following recrystallisation. Forgings and castings (using metal with 0.3—0.4% C) are quite satisfactory. If welding is unavoidable, 7—10% of nickel should be added. The construction of drums in such alloys for nitric acid transport is still experimental. C. IRWIN.

Welding in the chemical and process industries.

W. SPRARAGEN (Ind. Eng. Chem., 1929, 21, 425—431).—Methods of welding using thermite or oxyacetylene and various methods of electrical welding are described. The steel used should be selected with reference to the requirements of the particular case, but that with carbon content below 0.25% is the most easily handled. Greater strength is obtained by welding parts designed for welding than by welding a lap joint intended for riveting. The need for adequate supervision of welding work is referred to, and developments in the welding of stills, pipelines, etc. are mentioned. C. IRWIN.

Hydraulic classification of minerals.

G. BOZZA (Giorn. Chim. Ind. Appl., 1929, 11, 151—154).—The conditions to be maintained in an ore classifier with an ascending water current so as to obtain perfect classification are deduced, these conditions corresponding with those on which Fahrenwald's controlled-density classifier (B., 1928, 695) is based. Experimental values obtained show that neither the theory advanced by Fahrenwald nor that usually assumed concerning the relation between fall in mass and free fall represents the actual facts of the case.

T. H. POPE.

Oxygen in iron and steel. II.

P. OBERHOFFER, H. HOCKSTEIN, and W. HESSENBRUCH (Arch. Eisenhüttenw., 1928—9, 2, 725—738; Stahl u. Eisen, 1929, 49, 799—800; cf. B., 1927, 966).—The properties of certain alloy steels prepared in the normal way have been compared with similar steels prepared from iron which has been subjected for 1 min. to an oxidising blast. Oxidised vanadium and chromium steels are very viscous when molten, whereas nickel and tungsten steels are quite mobile. All the oxidised steels have a coarse-grained structure and the nickel steel is distinctly hot-short due to the presence of oxide in solid solution. Addition of chromium offsets slightly the presence of oxygen. Vanadium and aluminium are such strong deoxidising agents that they remove most of the oxygen from the steel, and their oxides, if retained in the metal, are so pliable that they do not affect the hot-working properties. The presence of oxygen in alloy steels hinders greatly the diffusion of carbon during cementation and the resulting structure is fine and hackly; it also narrows the hardening range and intensifies the effect of overheating.

A. R. POWELL.

Use of nickel in grey iron castings.

G. BRODSKY (J. Sci. Instr., 1929, 6, 168—169).—Addition of 2.5% of nickel to iron gives castings free from blow-holes and sponginess, especially suitable for hydraulic parts.

C. W. GIBBY.

Self-deforming zinc alloys.

L. LOSANA (Notiz. chim.-ind., 1927, 2, 616—622; Chem. Zentr., 1928, I, 1092—1093).—The changes that occur in the structure during ageing of zinc alloys containing aluminium and copper or aluminium and tin have been investigated.

Alloys with more than 3—4% Al undergo appreciable deformation on keeping at the ordinary temperature, especially when the castings are rapidly cooled. Copper accelerates the change and tin retards it. The deformation appears to be due to decomposition of the β -zinc-aluminium solid solution and to an allotropic change in the γ -zinc-copper constituent. These changes may continue for several years before reaching equilibrium. The completely stabilised alloys exhibit different anomalies on the dilatometric curve from those of the freshly cast alloys. A. R. POWELL.

Risk of poisoning when working with mercury. O. STELLING (*Svensk Kem. Tidskr.*, 1929, 41, 80—85).—The risk of contracting mercurial poisoning when work is carried on for long periods in rooms where this metal is exposed to the air has been pointed out by Stock (A., 1926, 707). In its earlier stages the disease is difficult to diagnose, as the symptoms closely resemble those met with in neurasthenia, but recourse should be had to examination of the urine and faeces, which contain small quantities of mercury if poisoning by that metal is in question; the air of the room should also be tested, using the colorimetric method with diphenylcarbazine, which permits of the detection of 7×10^{-6} mg. of mercury. The presence of only a few thousandths of a mg. of mercury per m.² of air can give rise to chronic mercurial poisoning. The question as to how far the amalgams used in dentistry can give rise to mercurial poisoning is still undecided, but there seems little doubt that the copper amalgam formerly employed for fillings is definitely injurious to health.

H. F. HARWOOD.

Electrolytic recovery of metals. G. EGER (*Z. angew. Chem.*, 1929, 42, 518—522).—A lecture before the Rhenish-Westphalian local section of the German Chemical Society describing the principles and methods used in the recovery of metals by electrolysis.

A. R. POWELL.

Electroplating of cadmium from cyanide baths.

L. R. WESTBROOK (*Amer. Electrochem. Soc.*, May, 1929. Advance copy. 14 pp.).—In a commercial type of bath containing the double cyanide, $\text{NaCd}(\text{CN})_3$, free sodium cyanide, sodium hydroxide and sulphate in the equivalent proportions 1:2:1.25:1.25, increasing the total concentration increases the conductivity and stability of the bath, raises the cathodic current efficiency, throwing power, and maximum current density for satisfactory plates, and gives a smoother and finer-grained deposit. Free cyanide keeps anodic polarisation low and anodic current efficiency high by preventing film formation on the anode, and also increases the smoothness and brightness of the deposit. Sodium hydroxide opposes the tendency of free cyanide to increase cathodic polarisation, whilst the tendency of the hydroxide to increase anodic polarisation is balanced by the opposite tendency of the cyanide. Increasing the sodium hydroxide concentration increases the cathodic current efficiency, the conductivity, and the brightness and ductility of the deposit. Sodium sulphate hardly affects the electrical properties of the bath, but seems to render it more stable. The cadmium content of the bath is maintained at a suitably high value

(generally about 40 g./litre) by using cadmium anodes of greater surface area than the cathodes and by maintaining sufficient free cyanide in the bath. Sodium cyanide and hydroxide are added at intervals to make up for losses due to hydrolysis and absorption of carbon dioxide, but the sodium carbonate so formed has practically no effect on the electrical properties of the bath nor on the character of the cathodic deposit if the cyanide and hydroxide concentrations are kept up. Of metals which act as "brighteners," nickel is most effective and 0.2—0.3 g./litre is commonly used. This addition of nickel (as sulphate) increases the brightness, ductility, and hardness of the deposit and greatly increases the maximum cathodic current density at which satisfactory plates can be obtained. Also impurities such as arsenic and antimony, which have a very bad effect on the deposit, are much less harmful in the presence of nickel. The effects of organic addition agents are discussed and the composition and conditions of operations of two typical cadmium-plating baths are given. H. J. T. ELLINGHAM.

Electrical resistance method of measuring corrosion of lead by acid vapours. R. M. BURNS and W. E. CAMPBELL (*Amer. Electrochem. Soc.*, May, 1929. Advance copy. 15 pp.).—A method is developed for measuring the rate of corrosion of a metal in terms of the rate of increase in resistance of a wire as it is reduced in cross-section by the progress of corrosion (cf. Hudson, B., 1928, 488). The accuracy and reproducibility of the method were determined by experiments with lead wires exposed to the vapour in equilibrium with dilute acetic acid of various concentrations. The percentage increase in resistance is a linear function of the time, and duplicate tests are reproducible to an accuracy of 15%. In order to compare the corrosive properties of green Douglas fir with those of other woods which might be used to replace it in the construction of conduits to carry lead-sheathed cables, the variation in the resistance of lead wires exposed to the vapour in equilibrium with damp sawdust from various woods was examined. Raw Douglas fir is found to be more corrosive than yellow pine or hemlock, and acidity determinations on the woods are in agreement with these results. Kiln drying does not reduce the corrosive action of the fir, but creosoting causes a notable reduction. The rate of corrosion of lead in acetic acid vapour falls off rapidly when the specimen is removed into the vapour of distilled water, and still more rapidly when put into the vapour from 1% ammonia solution. The rates of corrosion of two cable-sheath alloys and of pure lead in the vapour of 0.02*M*-acetic acid were compared, and the method was also applied to the examination of the corrosion of lead in nitric acid vapours.

H. J. T. ELLINGHAM.

See also A., June, 631, **Internal stress in carbon steels** (SEKITO). 668, **Determinations of metals as sulphates** (GUNTZ and BARBIER). 669, **Electrolytic determination of lead** (TÖPELMANN). 671, **Determination of tin by electrolysis** (ŠVĚDA and UZEL). **Separation of niobium and tantalum** (YNTEMA). **Separation of ruthenium and osmium, and titration of osmium** (SAITO).

Metal-corrosive papers. KALB and VON FALKENHAUSEN.—See V.

PATENTS.

Salt-bath furnace. A. G. E. HULTGREN (U.S.P. 1,713,472, 14.5.29. Appl., 17.5.28).—A non-volatile acid oxide, preferably silica, is introduced into a fused salt bath for hardening steel to prevent decarburisation.

C. A. KING.

Smelting furnace. A. JONES, Assr. to U.S. SMELTING FURNACE Co. (U.S.P. 1,712,769, 14.5.29. Appl., 29.10.25).—In a rotatable tilting furnace a fuel supply pipe extends along the furnace with a lateral branch carrying the burner, so that when the pipe is rotated the nozzle may be in or out of alinement with the furnace.

C. A. KING.

Ore-dust treatment. C. V. MCINTIRE, Assr. to CONSOLIDATION COAL PRODUCTS Co. (U.S.P. 1,711,153, 30.4.29. Appl., 6.7.25).—The dust is mixed with coal and the mixture heated and agitated in a retort to form agglomerates of semi-coke suitable for blast-furnace smelting.

A. R. POWELL.

Recovery of volatilisable metals [zinc] from iron-containing materials. F. KRUPP GRUSONWERK A.-G. (B.P. 299,300, 20.8.28. Ger., 22.10.27).—Iron ore containing zinc is mixed with at least 45% of coke and the mixture heated in a rotary furnace to reduce the iron to metal sponge and volatilise the zinc; the large excess of fuel prevents sintering of the charge and segregation of the sponge iron.

A. R. POWELL.

Direct reduction of iron from its ores. S. L. MADORSKY, Assr. to GATHMYS RES. CORP. (U.S.P. 1,711,738, 7.5.29. Appl., 1.2.26).—Iron oxide ore containing titanium is fractionally reduced by passing hydrogen through the molten ore substantially free from carbon and nitrogen.

C. A. KING.

Manufacture of cast iron. INTERNAT. NICKEL CO., P. D. MERICA, J. S. VANICK, and T. H. WICKENDEN (B.P. 311,798, 15.2.28).—A cast iron which does not exhibit the phenomenon of growth contains 3% C, less than 1.5% Si, and up to 5% Ni, 2–4% Ni being added for every 1% Si below the normal amount.

A. R. POWELL.

Carburisation of iron and steel. R. G. GUTHRIE and O. J. WOZASEK, Assrs. to PEOPLE'S GAS BY-PRODUCTS CORP. (U.S.P. 1,706,356, 19.3.29. Appl., 2.1.29).—Iron or steel is case-hardened in a retort filled with a carburising gas, a minute quantity of oxygen being admitted at the beginning of the heating to form an oxide catalyst which increases the depth of penetration of the carbon.

A. R. POWELL.

Improvement of ferrous metals and products thereof. E. S. FISHER, Assr. to UTAH METALS FLUX Co. (U.S.P. 1,712,879, 14.5.29. Appl., 20.8.26).—Molten iron is brought into contact with material similar to the carbonaceous shale occurring in districts of Emery County, Utah.

C. A. KING.

Annealing of carbon-containing iron and steel. VEREIN. STAHLWERKE A.-G., Assees. of E. H. SCHULZ and W. HULSBRUCH (B.P. 283,200, 12.12.27. Ger., 8.1.27).—To prevent decarburisation of the surface layers of iron and steel during annealing, a neutral atmosphere

comprising a mixture of a carburising and of a decarburising gas is maintained in the furnace. The most suitable mixtures are 10–40 vol.-% of methane and 90–60 vol.-% of hydrogen. [Stat. ref.]

A. R. POWELL.

Production of protective coatings on ferrous metals. F. B. RINCK, Assr. to WESTERN ELECTRIC Co., Inc. (U.S.P. 1,690,378, 6.11.28. Appl., 17.10.24).—Ferrous metal is subjected to an oxidising atmosphere, e.g., steam, at constant temperature (about 760°), the admission of steam being periodically increased, the hydrogen concentration reduced by having the hydrogen and the excess of uncombined oxygen minimised, e.g., by the introduction of hydrocarbon vapour.

R. BRIGHTMAN.

Production of diffused layers of metals [on iron]. E. LAY (U.S.P. 1,711,603, 7.5.29. Appl., 31.10.27. Ger., 15.6.26).—Iron is heated with a powder containing 8–30% of free aluminium.

H. ROYAL-DAWSON.

Hardening of molybdenum irons or steels. J. L. F. VOGEL (B.P. 311,588, 4.7.28).—The case-hardening of molybdenum steels by nitrogenisation is effected by heating them at 500–600° with calcium cyanamide and passing a controlled current of steam through the vessel for several hours. The following nitrogenising compounds may be used in place of the cyanamide: cyanides, urea, sodamide, naphthylenediamine, dicyanodiamide, phenylhydrazine, acetamide, or guanidine.

A. R. POWELL.

Iron-nickel alloy. F. KRUPP A.-G. (B.P. 302,994, 4.8.28. Ger., 24.12.27).—Alloys having high sp. resistance and high initial permeability, and containing 33–48% Ni and 0.3–5% Si, are claimed.

F. G. CROSSE.

Magnetic alloy. O. E. BUCKLEY, Assr. to WESTERN ELECTRIC Co., Inc. (U.S.P. 1,695,038, 11.12.28. Appl., 6.3.25).—A nickel-iron alloy, containing 80–83% Ni, with a higher permeability than iron at magnetising forces less than a few tenths of a c.g.s. unit, and independent of tension up to 5000 lb./in.², is used as loading material on signalling conductors for submarine cables.

R. BRIGHTMAN.

Stable-surface alloy steel resistant to acids. R. P. and H. A. DE FRIES, Assrs. to LUDLUM STEEL Co. (U.S.P. 1,712,030, 7.5.29. Appl., 26.11.26).—The alloy consists of 14–30% Ni, 6–20% Cr, 1–3% Si, 1–3% Ta, 0–1% C, the remainder being iron.

H. ROYAL-DAWSON.

Rust-, acid-, and heat-resisting ferrous alloy containing chromium and tantalum. P. A. E. ARMSTRONG (U.S.P. 1,711,484, 7.5.29. Appl., 8.8.25).—The alloy contains 7–25% Cr, less than 1.25% C, and less than 10% Ta.

H. ROYAL-DAWSON.

[Manufacture of chromium or manganese] iron or steel alloys. T. R. HAGLUND (B.P. 311,256, 7.2.28).—High-grade iron ore with a low content of silica is smelted with 60–70% ferrochromium, or with 80% ferromanganese, and with carbon so that an iron alloy with the desired chromium or manganese content is obtained together with a slag rich in chromic or

manganous oxide and containing less than 20% of silica. Before tapping, the slag is heated to such a temperature that the carbon content of the alloy is reduced to the correct amount by interaction with the slag, and subsequently other alloying elements, *e.g.*, nickel, are added as required. A. R. POWELL.

Steel alloy. J. L. COX (U.S.P. 1,711,519, 7.5.29. Appl., 12.2.27).—A forgeable steel alloy contains 0.35–1.5% C, 4–20% Cr, 6–18% W, and 1–6% Al. The chromium content is at most four times that of tungsten, and that of tungsten at most six times that of aluminium. C. A. KING.

Alloy steel. G. J. COMSTOCK, Assr. to FIRTH-STERLING STEEL CO. (U.S.P. 1,695,916, 18.12.28. Appl., 30.6.27).—Alloy steel containing 1.2–1.8% C, 10–14% Cr, 0.75–1.25% V, and 0.5–1.25% Mo has a high resistance to abrasion and can be air-hardened or annealed soft. R. BRIGHTMAN.

Reconditioning iron and steel drums. R. C. HEINZMAN (U.S.P. 1,696,036, 18.12.28. Appl., 15.10.27).—Internally-rusted drums are reconditioned by applying a solution of oxalic acid evenly over the interior surface, admitting live steam to the drum, and finally applying a solution of aluminium acetate. A rust-resisting, water-proof coating is produced over the inner surface. R. BRIGHTMAN.

Agent for cleaning and imparting rust-inhibitive properties to steel. J. D. KLINGER (U.S.P. 1,695,430, 18.12.28. Appl., 27.6.24).—A mixture of alcohol, citric acid, and sulphuric acid is used. C. HOLLINS.

Concentration of copper ores. J. A. HOLLADAY, Assr. to UNION CARBIDE SALES CO. (U.S.P. 1,706,293, 19.3.28. Appl., 11.8.26).—Oxidised copper ores are prepared for flotation by treating the finely-divided ore suspended in water with acetylene in the presence of an inert, non-combustible gas, *e.g.*, nitrogen, whereby the action of the acetylene on the particles of copper mineral is intensified. A. R. POWELL.

Refining of copper. H. H. ALEXANDER (B.P. 311,812, 16.1.28. Cf. B.P. 309,848; B., 1929, 479).—After refining, the surface of the molten copper is protected from oxidation and contamination by a molten layer of a sodium silicate or silicates to which may be added calcium oxide or fluoride; this layer covers the molten metal bath entirely, and is maintained during casting. M. E. NOTTAGE.

Heat treatment of aluminium castings. R. J. ROSHIRT, Assr. to BOHN ALUMINUM & BRASS CORP. (U.S.P. 1,713,093, 14.5.29. Appl., 18.1.26).—Aluminium alloys are quenched from as high a temperature as possible without distortion and reheated at 218–246°. C. A. KING.

Surface-treatment [colouring] of aluminium articles. A. PACZ (U.S.P. 1,710,743, 30.4.29. Appl., 16.4.26).—The articles are dipped in a solution containing a soluble fluorine compound and a salt of silver, nickel, tin, cobalt, zinc, cadmium, iron, manganese, lead, antimony, or copper. H. ROYAL-DAWSON.

Solder for aluminium. A. YERSIN (Swiss P. 122,845, 21.1.27).—An alloy of 60% Sn, 35% Zn, and 5% Al is claimed. A. R. POWELL.

Increasing the sp. gr. of molten electrolytes [for refining aluminium]. R. MÜLLER (G.P. 454,719, 13.1.26).—An addition of barium or strontium oxide, peroxide, or carbonate is made. A. R. POWELL.

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 312,555, 11.9.28. Addn. to B.P. 300,078; B., 1929, 24).—In the alloy described in the prior patent, the silicon content is increased up to 1.75% at the expense of the aluminium. F. G. CROSSE.

[Nickel] alloys. INTERNAT. NICKEL CO., and (A) E. J. BOTHWELL, (B) N. B. PILLING (B.P. 311,749 and 311,799, 15.2.28).—(A) An alloy containing 55–80% Ni, 43.5–6.5% Cu, 5–1% Si, 0.5–5% Fe, 0–0.5% C, and up to 3% Mn is claimed. The alloy is annealed at 830–1150°, quenched or air-cooled, and tempered at 500–890°. (B) The working properties of alloys with a high nickel content are improved by the addition of calcium just before pouring, preferably in the form of nickel or copper-nickel alloy containing 5–95% Ca and introduced below a layer of silicious material covering the molten metal. A. R. POWELL.

Protection of magnesium and its alloys. G. MICHEL (B.P. 287,046, 16.2.28. Fr., 12.3.27. Addn. to B.P. 249,484; B., 1927, 785).—The metal is coated with a varnish mixture having a base of synthetic resin and containing free phenol. The coating is subsequently baked. F. G. CROSSE.

Treatment of magnesium and its high-percentage alloys in the molten state. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 311,998, 9.8.28).—In the production of sound castings from magnesium and its high-percentage alloys, a coherent film in the shape of a hose of reaction products of the metal is caused to be formed on the surface of the flowing metal; this may be assisted by adding substances such as finely-divided sulphur to the atmosphere surrounding the molten metal. The mouth of the ladle is kept in a fixed position to the gate of the mould, and the rate of flow is regulated by tipping the ladle or crucible, the mouth of which acts as the pivot-point. M. E. NOTTAGE.

Manufacture of tungsten alloys low in tin and arsenic. F. M. BECKET, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,706,344, 19.3.29. Appl., 11.12.26).—Tungsten ores containing tin and arsenic are reduced in an electric furnace to produce an alloy containing at least 10% Si. This alloy is then fused with a metal oxide in quantity sufficient to oxidise and slag off the silicon. A. R. POWELL.

Recovery of zinc oxide. C. A. H. DE SAULLES, Assr. to AMER. SMELTING & REFINING CO. (U.S.P. 1,712,553, 14.5.29. Appl., 31.12.21).—Zinc ore is reduced continuously on the slag bath in a furnace having a slightly oxidising atmosphere, and the volatilised zinc products are condensed in a cooler chamber having an essentially oxidising atmosphere. C. A. KING.

Treatment of nickeliferous material [South African copper-nickel-platinum concentrates]. S. C. SMITH (B.P. 311,909, 18.4.28).—The ore is smelted to matte, which is roasted and reduced to a metal sponge by means of coal gas or other reducing agent. The metal sponge so obtained is leached with hot dilute

sulphuric acid, then air is drawn through the mass, whereby oxidation ensues with rise of temperature. A second charge of acid is passed through the mass and the air oxidation is repeated. Alternate leaching and oxidation are continued until no further metal is extracted, the residue is again roasted and reduced, and the leaching and oxidation are repeated until a product with a high content of platinum metals is obtained which can be refined by known methods.

A. R. POWELL.

Concentration of ores containing platinum and metals of the platinum group. E. C. R. MARKS. From CAYZER TIN SMELTING Co. (PROPRIETARY), LTD. (B.P. 311,991, 24.7.28).—Ores containing sulphides of nickel, copper, and iron, together with the platinum metals, are roasted sweet, mixed with 10% of bituminous coal or semi-coke, and heated at 900–1100° in a reducing atmosphere in a furnace with rotating hearth. The product is cooled out of contact with the air, and the sponge of iron, nickel, and copper containing all the platinum metals is separated by known means and treated for the recovery of the platinum metals.

A. R. POWELL.

Coating for metals. C. CHAFFER and H. HARGREAVES (B.P. 312,478, 12.5.28).—The metals are coated with an aqueous solution of gelatin. Hardening of the coating is effected with alcohol.

F. G. CROSSE.

Producing points of adhesion for galvanic deposits on light-metal bodies. LEIPZIGER SCHNELLPRESSENFABR. A.-G. (B.P. 305,453, 29.5.28. Ger., 4.2.28).—Pieces of metal having a better adhesion for the galvanic deposit than the light metal are placed in the casting mould of the light-metal body to be cast so that they are enclosed and appear on the surface of the finished casting.

J. S. G. THOMAS.

Working of blast furnaces. P. HESKAMP, Assr. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,713,435—6, 14.5.29. Appl., 5.11.26. Ger., [A] 6.4.26, [B] 8.9.26).—See B.P. 269,071 and 272,751; B., 1927, 575, 659.

Production of iron sponge S. E. SIEURIN (U.S.P. 1,714,280, 21.5.29. Appl., 17.12.27. Swed., 26.1.26).—See B.P. 296,235; B., 1928, 820.

Heat treatment of steel. N. D. CHOPRA and F. J. BULLEN (U.S.P. 1,712,253, 7.5.29. Appl., 18.3.27. U.K., 1.4.26).—See B.P. 271,606; B., 1927, 560.

Ascertaining the resistance to rusting shown by iron, steel, and iron alloys. C. MAULER (U.S.P. 1,714,035, 21.5.29. Appl., 11.7.27. Austr., 22.7.26).—See B.P. 292,794; B., 1928, 608.

Extraction of metals [zinc] from their [oxide] compounds. C. J. G. AARTS (U.S.P. 1,712,641, 14.5.29. Appl., 17.7.24. Belg., 28.7.23).—See B.P. 219,667; B., 1925, 676.

Reduction of zinciferous materials. F. G. BREYER and E. H. BUNCE, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,712,132 and 1,712,134, 7.5.29. Appl., [A] 27.1.27, [B] 20.4.27).—See B.P. 298,921 and 300,519; B., 1928, 931; 1929, 100.

Production of finished metal articles. E. G. BEK and E. THOMA (U.S.P. 1,712,244, 7.5.29. Appl.,

23.9.25. Ger., 11.12.24).—See B.P. 244,487; B., 1927, 633.

Electrolytic separation of metallic chromium for production of chromium coatings on other metals. R. APPEL (U.S.P. 1,713,514, 21.5.29. Appl., 3.8.26).—See B.P. 274,882; B., 1928, 97.

Steel etching plates (U.S.P. 1,705,155).—See XXI.

XL—ELECTROTECHNICS.

Insulating masses for transformers. W. HAMMERSCHMIDT (Z. angew. Chem., 1929, 42, 523–525).—The value of mixtures of rosin, rosin oil, and American asphalt as insulated sealing masses for transformers has been determined by measuring the voltage at which the insulation breaks down using electrodes 0.5 mm. apart. The results show that addition of rosin oil to rosin-asphalt mixtures rapidly decreases the insulating power, the decrease corresponding with the increase in fluidity.

A. R. POWELL.

See also A., June, 632, **Magnetic properties of iron** (VON AUWERS). 654, **Electrolytic polarisation: complex cyanides of silver and of copper** (GLASTONE). 671, **X-Ray tube for spectroscopy** (BAND and MADDOCK). 673, **Glass electrode** (MACINNES and DOLE).

Comparison of colours. TOUSSAINT.—See I. **Recovery of metals.** EGER. **Cadmium plating.** WESTBROOK. **Corrosion of lead.** BURNS and CAMPBELL.—See X. **Sterilisation of liquids.** LAKHOVSKY.—See XXIII.

PATENTS.

Electric discharge tube. F. SKAUPY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,712,407, 7.5.29. Appl., 30.4.21. Ger., 4.7.19).—Non-alkaline electrodes of appropriate area and consisting of fine wire mesh free from occluded non-inert gases are arranged in a sealed container filled with inert gas under reduced pressure, so that a glow discharge may be continuously produced between the electrodes at operating voltages of about 100 volts.

J. S. G. THOMAS.

[Cathode] filament. F. HOLBORN, Assr. to DE FOREST RADIO TELEPHONE & TELEGRAPH Co. (U.S.P. 1,695,687, 18.12.28. Appl., 9.1.24).—An alloy of iron or nickel and lithium, obtained by mixing iron with lithium amalgam, is claimed.

R. BRIGHTMAN.

Electrolysis reduction cell. Apparatus for carrying out electrolytic reduction of sugars to alcohols. H. J. CREIGHTON, Assr. to ATLAS POWDER Co. (U.S.P. 1,712,951—2, 14.5.29. Appl., [A, B] 26.3.26. Cf. U.S.P. 1,612,361; B., 1927, 234).—(A) An agitator projects into a mercury cathode placed within a catholyte container surrounding a porous cylinder containing an anode and anolyte. The porosity of the cylinder wall is such that hydrogen ions can pass from the anodic to the cathodic compartment, and hydroxyl ions in the reverse direction, whilst catholyte is prevented from passing into the anodic compartment. (B) Sugar-reducing apparatus comprises an electrolysis cell, from which an amalgam is discharged into a reduction cell provided with an agitator, and a mercury reservoir into which mercury is delivered from the reduction cell and

thence into the electrolysis cell, a given amount of mercury being maintained in each cell.

J. S. G. THOMAS.

Electrodeposition. L. C. TURNOCK (U.S.P. 1,712,284, 7.5.29. Appl., 17.12.25).—In an electroplating system electrically-operated means are provided for interrupting the electrical circuit, including the bath, after a predetermined amount of metal has been deposited.

J. S. G. THOMAS.

Electroplating apparatus. J. L. MUJICA (U.S.P. 1,712,674, 14.5.29. Appl., 30.7.26).—A rotatable permeable container, having a throat piece at one end, is supported on a carrier suspended on trunnions arranged at one side of a stationary tank, so that the carrier with the container and throat piece may be tipped from a receiving position in the tank to a position outside the tank to discharge its contents clear of the rotating mechanism.

J. S. G. THOMAS.

[Mounting for heating elements of] electric resistance furnaces. HEVI DUTY ELECTRIC Co., Assees. of E. L. SMALLEY (B.P. 312,006, 23.8.28. U.S., 22.5.28).

Sulphur trioxide and sulphuric acid (B.P. 311,862).—See VII. **Iron-nickel alloys** (B.P. 302,994). **Magnetic alloy** (U.S.P. 1,695,038). **Electrolytes for aluminium** (G.P. 454,719). **Deposits on light metal bodies** (B.P. 305,453).—See X.

XII.—FATS; OILS; WAXES.

Wizöff report: meeting of the German Commission for development of standard analytical methods in the fat industry (Chem. Umschau, 1929, 36, 137—164).—The suggested standard methods of fat analysis applicable to sulphonated oils, waxes, soaps, etc., and glycerin, circulated previously by the Wizöff (Central Association for Scientific Research in the Oil and Fat Industries) were discussed and amended.

E. LEWKOWITSCH.

Wizöff communication. **Colorimetry of oils and fats.** G. GREITEMANN (Chem. Umschau, 1929, 36, 167—168).—Iodine solutions are recommended as standards for the colorimetry of oils and fats where measurement of depth of colour is the primary requirement: the solutions keep well if protected from oxidation, and the apparatus required is cheap and simple. The "colour number" is defined as the number of mg. of free iodine contained in 10 c.c. of an aqueous iodine-potassium iodide solution which appears of an equal depth of colour in a layer 25 mm. thick examined by transmitted light (daylight lamp). (Cf. preceding abstract.)

E. LEWKOWITSCH.

Unification of methods for determination of iodine value. J. GIRAL (Anal. Fis. Quím. [Teen.], 1929, 2, 259—264).—A review. Suggestions are made for the definition of iodine value and for statements of the conditions used when the results of the determination of iodine values are recorded. The definitions and methods of determination of moisture and cellulose in foodstuffs are also discussed.

R. K. CALLOW.

Turbidity and solidification times of solid fats, with especial reference to cacao butter. W. SPRINGER (Chem. Umschau, 1929, 36, 165—167).—The times taken

by a melted fat cooling from 60° to reach (a) a definite degree of turbidity, and (b) the solidification point are found to be characteristic for different fats, and may be used for discrimination, particularly in the examination of cacao butter. Thus, the turbidity and solidification times, respectively, are: for cacao butter (of the order of) 500—1000 sec., 65—100 min.; for hardened coconut oil and other vegetable fats 300 sec., and 6—9 min.; and for tallow 75 sec., and 1.2 min. It is preferable to deacidify the fats before testing. E. LEWKOWITSCH.

Effect of clay on rancid fats. G. W. FIERO (J. Amer. Pharm. Assoc., 1929, 18, 491—493).—Colloidal clay removed rancidity from coconut oil either by simple agitation, by boiling with water and clay, or by treatment with clay, superheated steam, and reduced pressure. The first method gave the best product, and colour, odour, and taste were all improved. Under the conditions employed lard could not be completely renovated by any of the methods. E. H. SHARPLES.

Indirect analysis of three-component systems, especially of mixtures of fatty acids. K. TÄUFEL and M. RUSCH (Z. Unters. Lebensm., 1929, 57, 216—224).—The use of triangular co-ordinates for the indirect analysis of three-component systems is illustrated, using as examples: (a) a mixture of oleic, linoleic, and erucic acids, of known iodine and neutralisation values, and (b) a mixture of ammonium carbonate, bicarbonate, and carbamate, of which the contents of ammonia and carbon dioxide are known. The method is inapplicable to a mixture of oleic, linoleic, and linolenic acids, since the lines for the iso-iodine and iso-neutralisation values are parallel.

W. J. BOYD.

Glycerides of fats and oils. XIII. Glycerides of chaulmoogra oil. A. BÖMER and H. ENGEL (Z. Unters. Lebensm., 1929, 57, 113—147).—Chaulmoogra oil is rapidly oxidised in the air, but can be protected by hydrogenation. By repeated fractional crystallisation of the deacidified hardened oil from acetone-ether and acetone, a *dihydrochaulmoogra-didihydrohydnocarpin*, m.p. 30.7° (corr.), was isolated in yield amounting to 79% of the deacidified hardened oil. A *dihydrohydnocarpo-didihydrochaulmoogrin*, m.p. 42.2° (corr.), in 13% yield was also isolated, and a small quantity of difficultly soluble glyceride, probably tripalmitin or a stearodipalmitin. It is deduced that in the unhardened oil the corresponding unsaturated glycerides occur in similar proportions and that the fatty acids of the hardened oil before deacidification consisted approximately of 40% of dihydrochaulmoogric acid and 59% of dihydrohydnocarpic acid. The unusually low m.p., high refraction, and high solubility of hardened chaulmoogra oil as compared with other fats is due to the cyclic nature of the fatty acids. *Tridihydrochaulmoogrin* (m.p. 51.0°, corr.), *tridihydrohydnocarpin* (m.p. 39.2°, corr.), and *didihydrochaulmoogrin* (m.p. 60.7° corr.) were prepared synthetically from the lead soaps and appropriate halogenohydrins.

W. J. BOYD.

Drying of linseed oil and boiled oils. H. WOLFF and W. TOELDT (Farben-Ztg., 1929, 34, 2069—2072).—The rate of increase in weight of linseed and manganese-boiled oil films corresponds approximately with the rate

of drying as measured by the authors' dye-spreading test (B., 1929, 255); with cobalt-boiled oil change in weight begins after drying is well advanced. Increasing the thickness of the film scarcely affects the course of drying (physical change), but influences considerably the shape of the weight/time curves (chemical change): with thicker films a decrease in weight of the film was observed at the beginning of the drying process.

E. LEWKOWITSCH.

Economic utilisation of residues from oil manufacture: production of comestible flours rich in aleurone. E. ANDRÉ (Bull. Soc. d'Encour., 1929, 128, 347—359).—Oil seeds, *e.g.*, sunflower, sesamé, linseed, are rich in aleurone grains: by extracting the oil with light petroleum and applying to the residual meal grading methods similar to those used in milling, a nutritive nitrogenous product valuable for human consumption is obtained. Especially suitable is sunflower-seed meal, which is rich in aleurone grains containing 8.85% N and 3.2% P_2O_5 , equivalent to a 55% content of proteins.

E. LEWKOWITSCH.

See also A., June, 641, **Adsorption of soap solutions** (NONAKA; MIKUMO). 646, **Formation of soap gels** (HOLMES and MAXSON). 649, **Alkalinity of soap solutions** (MCBAIN and HAY). 671, **Calibration of Lovibond glasses** (PRIEST and others). 712, **Determination of carbon and carbon dioxide** (FRIEDEMANN and KENDALL). 714, **Fats of Japanese birds** (KOYAMA). **Silkworm chrysalis oil** (DE'CONNO and QUARTO). **Action of bromine on insect oils** (DAVID). 725, **Germicidal and hæmolytic action of α -bromo-soaps** (EGGERTH). 726, **Detection of vitamin-A** (STEUDEL). **Colour tests of vitamin-A in cod-liver oil** (TOWLE and MERRILL). **Fat-soluble vitamins in cod-liver oil** (BACHARACH and SMITH). **Vitamin-A in the liver** (LAQUEUR and others). **Vitamin colour reactions** (BEZSSONOFF). 727, **Irradiation of ergosterol** (DEPLAPLACE and REBIÈRE). 729, **Oil from *Datura stramonium*** (VERKADE and COOPS). **Seeds of *Monarda punctata*** (HARWOOD).

Analysis of butter. CHARLIERS. **Oil content of paprika.** DÖMÖTÖR.—See XIX.

PATENTS.

Manufacture of soft soap. L. G. LEFFER, Assr. to H. BACHSTEIN (U.S.P. 1,710,799, 30.4.29. Appl., 25.3.26, Ger., 4.4.25).—Coconut oil, palm-kernel oil, and rape-seed oil are saponified with potash lye under pressure, hydrocarbons being added during the process to regulate the setting point. E. LEWKOWITSCH.

Apparatus for separating fat or other material from gases. J. LYONS & Co., LTD., and G. W. CATLIN (B.P. 311,204, 3.2.28).—The apparatus comprises the chamber in which the mixture is formed, from which the mixture is drawn off laterally on opposite sides and conducted to an auxiliary chamber provided with baffles to retain all or a desired quantity of solid material.

E. LEWKOWITSCH.

Detergent. J. TENGLER (F.P. 629,691, 23.2.27. Spain, 18.3.26).—Benzine and carbon tetrachloride are added to soap liquefied by heating, and the product, when cold, is coated with wax, fat, varnish, etc. to prevent loss of the solvent by evaporation.

L. A. COLES.

Production of a detergent powder. A. LINDAHL (Swed. P. 58,788, 5.4.21).—A mixture of oil or fatty acids, water, and anhydrous sodium carbonate is treated with a blast of compressed air, or, alternatively, the water is replaced by a current of steam. The product settles out as a powder.

L. A. COLES.

Bleaching of fatty acids. A. WELTER (G.P. 454,308, 3.9.25).—The fatty acids (or oils) are saponified with soda (or caustic soda); the soaps are bleached with chlorine or sodium hypochlorite, treated with sulphuric acid, and vigorously agitated with air.

E. LEWKOWITSCH.

Refining and bleaching of fish oils. J. C. KERNOT (B.P. 311,665, 10.12.27).—The oily emulsion formed by subjecting fish oils to the action of aqueous caustic alkalis below 100° is separated, washed and agitated with a concentrated aqueous solution of sulphur dioxide or like sulphurous bleaching agent, and the separated oil is washed, dried, and filtered. Treatment with sulphurous acid may be repeated or the bleaching carried further by selective hydrogenation in the presence of mixed catalysts (*e.g.*, hydrated oxides of nickel and aluminium).

E. LEWKOWITSCH.

Prevention of rancidity in vegetable oils used in plastic compositions for coating fabric, paper, and the like. G. E. SCHARFF, and NOBEL'S EXPLOSIVES Co., LTD. (B.P. 311,786, 17.11.27. Addn. to B.P. 256,654; B., 1926, 887).—About 8% of zinc (or magnesium) oxide may be advantageously added to the antimony sulphide (or other metallic sulphide of the second group in wet analysis) to be incorporated in the plastic coating, a total of 2% (calc. on the vegetable oil content) of the mixture being suitable.

E. LEWKOWITSCH.

Apparatus for distillation of fatty acids, glycerin, etc. W. GENSECKE (U.S.P. 1,713,431, 14.5.29. Appl., 12.11.24. Ger., 28.11.23).—See B.P. 225,552; B., 1925, 556.

Removal of boiler-scale (B.P. 311,550).—See XXIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Action of the substratum on coloured paints. H. WAGNER (Farben-Ztg., 1929, 34, 2011—2014).—The unsatisfactory behaviour of chalk as a white base in coloured paints has been investigated by comparing the properties of paints made from it with those of paints made from barium or calcium sulphate. The inferiority of the chalk paints is not due to their impermeability to ultra-violet light, which is about equal to that of paints with barytes and other white bases, but appears to be due to the flocculated condition of the chalk particles in the prepared paint, to the fineness of the particles, to their soft texture, to the high oil content of the paint, and to its great tendency to swell.

A. R. POWELL.

Sipalin: a new softening reagent for cellulose lacquers. W. SCHRAUTH (Farben-Ztg., 1929, 34, 2018—2019).—The cyclohexyl and methylcyclohexyl esters of adipic and methyladipic acids are manufactured under the general name of "sipalin" for use in the preparation of cellulose lacquers. They are all viscous liquids with very low m.p. and high b.p., are practically

odourless, non-hygroscopic, and non-volatile, have a flash point above 180°, and are readily miscible with cellulose acetate and nitrate, resins, fatty oils, and rubber-producing plastic masses to give tenuous, pliable, and non-cracking films. Solutions of cellulose esters in sipalin may be coloured with inorganic pigments or with organic dyes.

A. R. POWELL.

See also A., June, 696, Resin acids (ROUIN).

Comparison of colours. TOUSSAINT.—See I. **Drying of linseed oil and boiled oils.** WOLFF and TOELDT.—See XII.

PATENTS.

Production of oiled pigments. H. GROSSMAN, Assr. to W. S. PRITCHARD (U.S.P. 1,696,034, 18.12.28. Appl., 25.2.27).—An emulsion of oil in water is added to a suspension of the pigment, the protective colloid (casein) is coagulated, and the oil in separating carries the pigment with it, giving an oiled pigment which retains its fine state of division.

C. HOLLINS.

Brushing lacquers. S. D. SHIPLEY, Assr. to ATLAS POWDER CO. (U.S.P. 1,692,584, 20.11.28. Appl., 28.9.25).—A nitrocellulose lacquer is employed which contains the minimum of glycol monoethyl ether as solvent, diluted with petrol; *e.g.*, glycol monoethyl ether (40 pts.), gum solution (25 pts.), petrol (35 pts.), nitrocellulose (12 oz. per gal.), and pigment (3.25 lb./gal.).

C. HOLLINS.

Lacquer solvent. J. P. TRICKEY, Assr. to QUAKER OATS CO. (U.S.P. 1,703,697, 26.2.29. Appl., 15.1.27).—Tetrahydrofurfuryl alcohol, alone or mixed with, *e.g.*, butyl alcohol (5–15%), butyl acetate (5–15%), ethyl acetate (20–30%), or toluene (30–45%), is used as solvent for cellulose nitrate, gums, etc. for lacquers.

R. BRIGHTMAN.

Production of synthetic resins. LA FIBRE DIAMOND (B.P. 282,435, 16.12.27. U.S., 16.12.26).—A mixture of a phenol, *e.g.*, cresol, with a larger quantity of an amine, *e.g.*, aniline, is heated with an unsubstituted aldehyde, *e.g.*, formaldehyde, and the product is distilled under reduced pressure to remove uncondensed constituents etc., leaving as residue a soluble, readily fusible condensation product which may be used direct for the manufacture of varnishes etc., or may be heated under pressure and in the presence of hardening agents, *e.g.*, hexamethylenetetramine, for the production of hard, infusible resins.

L. A. COLES.

Resinous material and its manufacture. C. ELLIS (U.S.P. 1,691,271, 13.11.28. Appl., 28.11.24).—A low-grade phenolic oil is condensed at 15° with a mixture of magnesia and formaldehyde; plaster of Paris is added (to take up the 4–5% of water in the separated resin) together with other fillers etc. to give a moulding-powder.

C. HOLLINS.

Production of plastic composition materials. C. A. NASH, Assr. to CUTLER-HAMMER MANUF. CO. (U.S.P. 1,692,856, 27.11.28. Appl., 12.3.21).—Phenol and formaldehyde (or hexamethylenetetramine) are caused to react below 80° in presence of a filler, *e.g.*, asbestos, reaction being stopped when about half the materials have combined.

C. HOLLINS.

Sulphur product and its manufacture. C. ELLIS (U.S.P. 1,690,335, 6.11.28. Appl., 7.4.28).—Crude cresylic acid, *e.g.*, a tar-acid fraction 50% of which distils below 212°, is treated with an equal weight of sulphur monochloride in presence or absence of a diluent, *e.g.*, toluene, and the product is deodorised by steaming at 140–150°. The sulphurised product, m.p. 91.5°, is incorporated with sulphur, a mixture of 25% of resin and 75% of sulphur having m.p. 105°. Sulphuryl chloride or sulphur dichloride may also be used.

R. BRIGHTMAN.

Mill for fine grinding [for paints etc.]. J. R. TORRANCE (U.S.P. 1,713,487, 14.5.29. Appl., 1.10.23. U.K., 17.10.22).—See B.P. 209,829; B., 1924, 238.

Production of titanium-containing pigments. F. G. C. STEPHENS, L. J. ANDERSON, and W. A. CASH, Assrs. to NAT. METAL & CHEM. BANK, LTD. (U.S.P. 1,714,408, 21.5.29. Appl., 24.3.27. U.K., 26.3.26).—See B.P. 273,017; B., 1927, 661.

Protection of magnesium etc. (B.P. 287,046).—See X.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

See A., June, 644, Viscosity of caoutchouc solutions (KROEPELIN). 674 and 702, Natural and synthetic rubber (MIDGLEY and HENNE).

PATENTS.

Manufacture of rubber articles. H. REITZ (G.P. 453,899, 2.11.23).—Latex, to which colloidal sulphur or soluble sulphides may have been added, is treated with aldehydes in the form of solution or vapour, which renders it viscous. The latex is then shaped or spread as desired and vulcanised.

D. F. TWISS.

Vulcanisation of rubber. I. OSTRONISLENSKY, Assr. to REVERE RUBBER CO. (U.S.P. 1,696,409, 25.12.28. Appl., 24.3.25. Cf. U.S.P. 1,249,180; B., 1918, 66 A).—Rubber is vulcanised with a nitro-compound (trinitrobenzene) in presence of amines (aniline). As accelerators elements of the carbon group (lamp black, graphite, silicon) are used, and as activators metal oxides, *e.g.*, litharge.

C. HOLLINS.

Vulcanisation [of rubber]. GIBBONS BROS., LTD. From C. DRESSLER (B.P. 311,507, 21.3.28).—Rubber articles, in moulds or otherwise, are passed slowly and continuously through an elongated chamber so that they are first gradually heated, then subjected to vulcanising heat, and finally cooled, the atmosphere, if desired, being of carbon dioxide. Arrangements may be provided for any moulds to be closed after the warming operation and before vulcanisation.

D. F. TWISS.

XV.—LEATHER; GLUE.

Moisture in leather. II. H. BRADLEY, A. T. MCKAY, and B. WORSWICK (J. Soc. Leather Trades' Chem., 1929, 13, 87–106; cf. B., 1929, 446).—The permeabilities of various leathers to water vapour have been determined. "Waterproofed" chrome upper leathers were most highly resistant to the passage of water vapour through them. Vegetable-tanned sole and insole leathers were least resistant. Diffusion constants have been deter-

mined for water into various leathers by immersing a rectangular piece of the leather in distilled water and noting the increase in weight at frequent intervals. The diffusion constant is a characteristic of the leather. The greater it is, the more absorbent or less waterproof is the leather. The smallest diffusion constant was shown by waterproofed chrome-tanned upper leathers. Curried vegetable-tanned splits had a diffusion constant three times, and waxed chrome-tanned sole leather five times, that of the above leathers. Sole leathers are only one fifth as waterproof as the corresponding uppers.

D. WOODROFFE.

Double refraction of tanned collagen fibres.

A. KÜNTZEL (Collegium, 1929, 207—214).—Collagen fibres were treated for 24 hrs. with solutions of gallo-tannic acid, sumac, pine bark, sulphite-cellulose, and chromium chloride, respectively, dried, immersed in different liquids, and examined. The double refraction of fibres treated with sumac and gallotannic acid was found to be reversed when they were examined in aniline, eugenol, and nitrobenzene, but the other treated fibres showed positive double refraction. No reversal occurred with tanned fibres when similarly treated. The tannins are not taken up between the micelles but by them, with consequent change of structure and optical properties. Tannage is a "permutoid reaction" and not a micellar surface reaction.

D. WOODROFFE.

Action of water on vegetable-tanned leather.

I. Rate of removal of uncombined soluble matter.

H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1929, 24, 235—242).—Leathers loaded with salt, glucose, and gallic acid were washed in Wilson-Kern extractors with distilled water and samples of the leather analysed at intervals. The whole of the loading material was removed after 2 hrs.' treatment. The removal of all the soluble, natural non-tannin took 4 days, but 5% of the water-soluble organic matter was removed in 2 hrs. Natural and added non-tannins cannot be differentiated by selective extraction.

D. WOODROFFE.

Tanning properties of dyewood extracts.

H. A. BRECHT (Collegium, 1929, 186—191).—Dyewood extracts contain flavonols or closely allied products, which precipitate gelatin solutions and salts of heavy metals, reduce Fehling's solution, and give all the reactions of the pyrocatechol tannins. They resemble tannins in the insolubility of the pure flavonols and the solubility of the dyewood extract. Analyses of extracts of the following by the filter-bell method gave (% tannin): fiset wood, d 1.180, 22.2; liquid fustic, d 1.241, 45.6; and dried logwood, 34.1. The non-tan filtrates were colourless. By the shake method of analysis lower results were obtained, and logwood and hæmatin extracts gave pale yellow non-tan filtrates. The hæmatein was absorbed by the hide powder in the tannin analysis, but the hæmatoxylin remained in the liquor. Dyewood extracts are too expensive to use for tanning purposes, but they are often used unknowingly for dyeing on account of their tanning properties, giving weight and a superior feel to the leather, which cannot be obtained by coal-tar dyes, hence their non-replacement by the latter for certain purposes. The close

relationship between the structure of the pyrocatechol tannins and the natural dyes is described.

D. WOODROFFE.

Evaluation of tannin solutions by interferometry.

F. ENGLISH (Collegium, 1929, 109—119).—It is shown that the measurement of interference effects of tannin solutions can be used for their evaluation and for distinguishing tans and non-tans. An exact determination of the interference factor has hitherto been impossible owing to the effect of concentration of the solution, temperature, and the method of detannisation. It is suggested that interference measurements could replace gravimetric analysis on the same tannins in extract factories or special laboratories.

D. WOODROFFE.

Determination of insoluble matter in tannin solutions.

D. McCANDLISH and W. R. ATKIN (J. Soc. Leather Trades' Chem., 1929, 13, 69—72, J. Amer. Leather Chem. Assoc., 1929, 24, 231—235, and Collegium, 1929, 98—101).—The official international method of filtration requires too long for myrobalans and crude quebracho extracts, with which it is practically impossible to comply with the regulations. Filtration under reduced pressure gives clearer filtrates. A 500-c.c. filter flask is fitted with a rubber stopper and cylindrical separating funnel (120 c.c. capacity) with stopcock and side tube. A glazed Buchner funnel (2.75 in. diam.), fitted with a stopcock on the stem, is connected to the separating funnel. The side tubes of the filter flask and separating funnel are connected through a T-tube to a vacuum pump. A Whatman No. 5 paper is placed in the Buchner funnel and drawn down by running 25 c.c. of tan liquor over it. The two stopcocks being left open, the vacuum is increased, a mixture of 1 g. of kaolin in 30 c.c. of tan liquor is filtered to form a kaolin pad, the vacuum is allowed to fall, and tan liquor is passed through continuously for 1 hr., by which time sufficient filtrate for evaporation is collected in the separating funnel. Good concordance is claimed for the method.

D. WOODROFFE.

Determination of insoluble matter in tannin extracts.

C. RIESS (J. Amer. Leather Chem. Assoc., 1929, 24, 229—231, and Collegium, 1929, 103—4).—The procedure agrees with that of McCandlish and Atkin (cf. preceding abstract) with the difference that suction is applied by a suction tube.

D. WOODROFFE.

Modified Wilson-Kern extractor [for hide powder].

H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1929, 24, 244—245; cf. A., 1921, 819 A).—The upper part of the Wilson-Kern extractor has been eliminated, and the middle portion, finished off at the top as a bottle neck, is fitted with a 1-hole rubber stopper and tube provided with a stopcock to regulate the flow of ash water.

D. WOODROFFE.

Determination of water content of glue and gelatin.

E. SAUER and H. DELLENUS (Z. angew. Chem., 1929, 42, 552—555).—An aqueous solution of 50—80 g. of material is made up to 1 litre at 40°, and 10 c.c. are evaporated to dryness, preferably in an aluminium dish, and dried to constant weight at 110°.

J. S. CARTER.

Determination of insoluble matter in tanning extracts.

A. TURNBULL (J. Soc. Leather Trades'

Chem., 1929, 13, 65—68, and Collegium, 1929, 101—103).—See B., 1929, 405.

See also A., June, 646, Setting of gelatin sols (LOTTERMOSER and MATTHAES). Swelling of gelatin (NORTHROP and KUNITZ). Hydrolysis of gelatin (YAITSCHIKOV).

Water supply for tanneries. AUERBACH.—See XXIII.

PATENTS.

Material for tanning and its manufacture. C. J. HERRLY and A. M. MATHESON, ASSTS. to NIACET CHEMICALS CORP. (U.S.P. 1,695,655, 18.12.28. Appl., 7.3.28).—Rapid tanning agents are made by condensing 2 mols. of a phenol with paraldehyde at 45—55° in presence of a little sulphuric acid and sulphonating the product. C. HOLLINS.

Tanning of hides. G. POVARNIN (RUSS. P. 3364, 15.9.24).—The hides, with or without the usual pretreatment, are treated with furfuraldehyde or diacetyl or with solutions containing them. L. A. COLES.

Treatment of hides and skins. H. DODGE (U.S.P. 1,690,969, 6.11.28. Appl., 28.11.27).—Dried hides and skins are immersed in an aqueous solution of nitre, sodium bicarbonate, and formaldehyde. R. BRIGHTMAN.

Manufacture of [waterproof] glue. A. HOCHÉ, ASST. to LITMO ADHESIVE & PRODUCTS CO. (U.S.P. 1,710,687, 30.4.29. Appl., 11.10.22).—Chrome-tanned leather (2—5% Cr₂O₃) is treated with successive baths of sulphuric acid of decreasing acidities, e.g., *d* 1.162, 1.075, and 1.037, for 30 min. in each, and the extracted leather is dissolved by heating with water. D. WOODROFFE.

Water-resistant animal glue. C. E. HRUBESKY and F. L. BROWNE (U.S.P. 1,712,077, 7.5.29. Appl., 14.3.28).—The glue consists of animal glue, water, paraformaldehyde, and a suitable amount of acid. H. ROYAL-DAWSON.

Adhesive material and its manufacture. L. BRADSHAW and H. V. DUNHAM (U.S.P. 1,703,134, 26.2.29. Appl., 17.12.26).—Starch, casein, and a seed meal rich in protein are incorporated with sufficient alkali for liquefaction in water. R. BRIGHTMAN.

Fertiliser (U.S.P. 1,695,081).—See XVI.

XVI.—AGRICULTURE.

Influence of substituted cations on the properties of soil colloids. M. S. ANDERSON (J. Agric. Res., 1929, 38, 565—584).—In an investigation of the change in properties that different soil colloids may undergo when the exchangeable bases are substituted by different cations, determinations have been made of the heat of wetting, adsorption of water vapour, swelling, moisture equivalent, cataphoresis, and hydrogen-ion concentration of widely different colloidal materials when saturated with calcium, magnesium, potassium, sodium, hydrogen, and the cation of methylene-blue. The order of cation effects on heat of wetting and moisture adsorption is Ca > Mg > Na > H > K > methylene-blue, and on the remaining properties the order is Na > K > Ca > Mg > H. The varying effects are ascribed to differences in solu-

bility and dissociation tendency of the colloid-cation combinations. The influence of a cation appears to be in direct proportion to its percentage of complete replacement. The amount of change suffered through replacement varies widely in different colloidal soil materials. The ranges of alteration of different soil colloids correlate fairly well with magnitudes of the properties of the untreated or calcium-saturated materials, with the total content of exchangeable bases, and with the SiO₂/(Al₂O₃ + Fe₂O₃) ratio of the colloid. This is explained on the ground that many properties of the soil colloids are directly influenced by the total content of exchangeable bases, which is to some extent dependent on the SiO₂/(Al₂O₃ + Fe₂O₃) ratio. The range of alteration of the colloidal soil material should be considered in judging what soils would show appreciable change in properties following heavy applications of a single fertilising salt and also in indicating those alkaline soils which will become impenetrable on leaching. In most soil colloids except those of saline soils the general properties will be indicated by the total base-exchange capacity, or one of the determinations associated with it. E. S. HEDGES.

Soil structure. I. Mutual interaction of the sols of ferric hydroxide, silicic acid, and permanganate. V. N. SYMAKOV and S. P. KRAYKOV (State Inst. Exp. Agron., Bur. Soils Bull. No. 3, 1928; Proc. Internat. Soc. Soil Sci., 1929, 4, 54—55).—The proportions in which the sols mutually coagulate each other are determined, and the conditions of complete mutual coagulation of mixtures of 3 sols are examined and discussed. A. G. POLLARD.

Soil structure. I. Dependence of stability of soil structure on adsorptive complex and silt. II. Aggregate analysis as a method for determining real soil structure. A. T. TIULIN (Agric. Exp. Stat. Perm, No. 2, 1928; Proc. Internat. Soc. Soil Sci., 1929, 4, 49—51, 51—54).—I. The stability of soil structure as determined by washing out the sample with water at a definite pressure depends on the saturation capacity of the soil for calcium when the absorbing complex is saturated with calcium and magnesium, and is inversely proportional to the silt content. It can be expressed in the relationship percentage of silt/adsorptive capacity for calcium. This relationship is designated a "colloid-mechanical factor of soil-structure stability."

II. Structural aggregates in soil are classified as (1) real aggregates, bound together by gels of bi- and ter-valent cations, and (2) false aggregates bound together by gels, or even suspensions, containing uni-valent cations. The real aggregates are stable in water, whereas false aggregates disperse. Soil structure depends on the first class. Real aggregates can be separated in water by Robinson's or the sieve method without pretreatment. False aggregates are separated in the air-dry condition with sieves, and those of smaller dimensions by Robinson's method using benzene in place of water. From the values obtained the amounts of real aggregates in the corresponding fractions are deducted. In comparative experiments more exact values for the real aggregates are obtained by deducting

from the experimental values the amounts of elementary particles in the various fractures as obtained by normal mechanical analysis. There is a general parallelism between the "colloid-mechanical factor" and the results of aggregate analysis, although the latter method gives a more exact valuation of the actual soil structure. Comparative aggregate analyses of a number of soils classified these in the same general order as their crop-producing powers. Structural differences between the surface and subsoil due to tillage are reflected in the differences in aggregate analyses. A. G. POLLARD.

Composition of absorbing soil complex. A. T. TULIN (Agr. Exp. Stat. Perm, No. 1, 1927, 1—28; Proc. Internat. Soc. Soil Sci., 1929, 4, 38—40).—Parallel determinations of the saturation capacity (calcium) and humic content of the adsorbing complex of soils of various types are recorded. Increased humic content is coincident with increased adsorptive capacity, although the relationship is not uniform in all soils. In chernozem soils the ratio of carbon in the adsorbing complex (Gedroiz) to saturation capacity for calcium is constant. The value of the constant differs for different soil types. If the separated adsorbing complex after treatment with sodium chloride is coagulated with tervalent aluminium salts, no further exchange reactions in the complex can occur. This condition of the complex is designated "passive." Pretreatment with barium or calcium hydroxide renders the complex active, and the barium and calcium ions can take part in exchange reactions. A. G. POLLARD.

Alterations in the degree of acidity of soils by drying the samples. B. AARNIO (Bull. Agrogeol. Inst. Finland, 1928, No. 26; Proc. Internat. Soc. Soil Sci., 1929, 4, 27—28).—Changes in the acidity of soils during drying are more rapid at higher temperatures, all soils examined becoming more acid. The reaction of moist soils at 25° increases rapidly at first but falls again later. Drying at 100° increases the acidity rapidly, the exchange acidity as well as the reaction of the water extract being affected. At higher temperatures (400°) a neutral clay became more acid and later more alkaline, approximating to the unheated soil. An acid clay, the reaction of which was unchanged at 400°, became less acid as the temperature was raised and was neutral at 1000°. Such reaction changes are most marked in heavy soils, are small in loams, and scarcely appreciable in sands. In soils rich in humus the changes are relatively smaller. The alteration of reaction probably results from the diminished total surface area brought about by drying, with the accompanying liberation of ions (acid salt-forming bases—Steenkamp), whilst at temperatures of 400° and upwards chemical reactions occur whereby the ions are again combined and the acidity decreases. A. G. POLLARD.

Physical properties of sugar cane soils and their value in practical agriculture. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, 195—254; Proc. Internat. Soc. Soil Sci., 1929, 4, 59—60).—Comparison of reports of cane growers with mechanical analyses of numerous soils by the methods of Mohr and of Goldschmidt showed no relationship between the size of particles and the permeability, ease of working, or

crop yields. Better correlation was possible when Atterberg's method was adopted for mechanical analysis. The calculation of hygroscopicity from mechanical analyses by Briggs' method proved valueless. Hygroscopicity is an important factor in soil examination. Using this value and the moisture content the total water supply available for the plant can be calculated. The water requirement for cane is 300 litres/kg. of plant (confirming Leather's values). A. G. POLLARD.

Relations between different properties of sugar cane soils. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1927, No. 48, 1197—1213; Proc. Internat. Soc. Soil Sci., 1929, 4, 89—90).—A relation exists between the reaction of a soil and its phosphate content, the latter being low in acid soils and high in alkaline ones, with a fairly constant value over the range p_H 6.5—7.5. This is ascribed to varying solubility, and therefore ease of removal by leaching, of the three simple phosphates of calcium. Nitrification in soils is not dependent on soil reaction, but varies directly with the phosphate content. The activity of the nitrifying organisms depends on the supply of phosphate. A. G. POLLARD.

Soil acidity and sugar cane. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1927, No. 41, 1027—1043; Proc. Internat. Soc. Soil Sci., 1929, 4, 83).—Examination of numerous field soils together with culture experiments in which the soil reaction was artificially adjusted showed the maximum growth of sugar cane occurs at p_H 7. A. G. POLLARD.

Reaction of sugar cane soils in Java. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1927, No. 6207—6228; Proc. Internat. Soc. Soil Sci., 1929, 4, 87).—Liming of acid soils increases the yield of cane. Sugar yields were higher in cane grown on neutral or alkaline soils than on acid soils. Soil reaction and phosphate deficiency were related, and the lower yields from acid soils may be indirectly the result of this. A. G. POLLARD.

Changes in some physical properties of soils brought about by lime. A. GEHRING (Z. Pflanz. Düng., 1929, 8B, 239—254).—The application of marl, quicklime, and slaked lime to soils increased their pore space without markedly affecting their absolute moisture contents. Such treatment of field soils led to increased soil temperature, variations in which showed a general relationship to the corresponding changes in pore space. The maximum and most definite effects of these calcareous materials were observed with quantities corresponding to lime required to produce a 70% saturation of the soils with bases. A. G. POLLARD.

"Kalkammon" [as fertiliser]. K. NEHRING (Z. Pflanz. Düng., 1929, 8B, 233—239).—No appreciable losses of nitrogen occurred during the storage under ordinary conditions of "Kalkammon" (a mixture of ammonium chloride with 30% of chalk). In pot experiments the material proved physiologically slightly acid, but in field trials the soil reaction was not affected. In normal soils the fertiliser value of "Kalkammon" was similar to that of ammonium sulphate or sodium nitrate, but in acid soils with sensitive plants (e.g., barley) it was not fully effective. A. G. POLLARD.

Iodine question in agriculture. W. GAUS and R. GRIESSBACH (Z. Pflanz. Düng., 1929, 13A, 321—425).—An extensive and critical review of existing knowledge is given, and numerous research reports are recorded. In field trials no definite increases in crop yields from iodine manuring were observed. The higher iodine contents of plants following iodine manuring were irregular and generally confined to the stems and leaves. In some cases actual decreases in the iodine contents of roots resulted from the iodine treatment. Sodium iodate was more effective than potassium iodide in increasing the iodine content of sugar beet. Among the plants examined spinach and lettuce had the highest iodine contents. Of the iodine manure applied only a very small proportion was absorbed by the plant (seldom more than 0.1%). Continuous heavy dosing of tomato soils with iodine during the growing period resulted in large increases in the leaf iodine content and smaller ones in the fruit. Under these conditions the greater part of the iodine enters the plants by the leaves. The absorption of iodine by leaves from iodine vapour is approximately 100 times as great as from potassium iodide solution. The accumulation of iodine by plants is in no way connected with any vital processes occurring in the plant tissues. The iodine in soil is mainly concentrated in the finer fractions. Considerable variations were observed in the iodine contents of sea coast soils. On light, permeable soils no permanent increase in iodine content resulted from manuring with seaweed. In recently dyked soils the iodine content was greatest in the upper (A-) horizons, but in older soils the tendency was for the greatest iodine accumulation to appear in the B-horizons. The iodine liberated during the burning of coal in furnaces etc. does not become evenly distributed throughout the atmosphere, but remains concentrated locally. It is precipitated only with rain, there being no appreciable absorption of iodine by alkali solutions exposed to the air. A. G. POLLARD.

Influences of the development of higher plants on the micro-organisms of the soil. II. Influence of the stage of plant growth on the abundance of organisms. R. L. STARKEY (Soil Sci., 1929, 27, 355—378; cf. B., 1929, 447).—Examination of numerous soils under field and greenhouse conditions show that growing plants tend to increase the number of soil organisms. This effect increases with the age of the plants, and differs with the nature of the plant and the species of soil organism. The greatest proportional increases were observed in the *B. radiobacter* group. Nitrogen-fixing bacteria, actinomyces, and filamentous fungi were but little affected. The influence on the soil organisms decreases after the death of the plants, but is not associated with the size of the plant or the extent of its root system. A. G. POLLARD.

Determination of the surface area of soils by adsorption of liquids. A. NEUGEBOHN (Diss., Breslau, 1927; Proc. Internat. Soc. Soil Sci., 1929, 4, 55—56).—The Mitscherlich method for determining the surface area exposed by a soil is not only imperfect in technique but theoretically unsound. The process for measuring total surface area in soils by hygroscopicity is adversely criticised. The quantity of water

absorbed by soil is not proportional to the total surface exposed since capillarity, swelling phenomena, and hydration are also concerned, and there is no uniformity in the thickness of the aqueous film on the soil surface.

A. G. POLLARD.

Determination of the degree of humification of forest litter. L. KRÓLIKOWSKI (Z. Inst. Gleboznawczego Univ. Poznawskiego, Wplyneto 21, XI, 1927; Proc. Internat. Soc. Soil Sci., 1929, 4, 37—38).—Robinson and Jones' method for examining the humification of soil constituents etc. was successfully applied to various forest litters. The transition from the upper horizon A_0 to the sedimentary humus layer A_2 is gradual, and the difficulty of accurate sampling of individual horizons prevents close correlation of the degree of humification with the age and genus of the forest trees or the natural conditions. A. G. POLLARD.

Errors in the determination of humus in soil. M. COUVRE (Giorn. Chim. Ind. Appl., 1929, 11, 149—150).—When soil contains dolomite or magnesite the combustion method of determining humus should be discarded, since it becomes difficult to expel all the carbonic acid by treatment with phosphoric acid, and excess of the latter renders troublesome the subsequent evaporation to dryness. Moreover, commercial phosphoric acid often contains sufficient proportions of organic matter to vitiate the results. In determining humus by the loss of weight on heating, the loss of water of crystallisation and of constitution undergone by normal mineral components of the soil may constitute a source of error; if the soil is slightly moistened with ammonium nitrate solution, oxidation of the humous material may be effected at a comparatively low temperature (in an oven at 250—300°), and subsequent treatment of the residue with ammonium carbonate solution not only reconverts the oxides formed into carbonates, but also replaces the water lost. Tests on kaolin confirm these views. Oxidation by means of permanganate and sulphuric acid is the most suitable method for determining humus if a number of determinations are made, but chlorides and other extraneous substances reacting with permanganate must be present in only minimum quantity. Calculation of the organic matter from the proportion of nitrogen in soil is not to be recommended, as the percentage of nitrogen in humus is variable and humus readily absorbs appreciable amounts of nitrogenous compounds. Any method is subject to the sampling error, since at most 2 g. are taken for analysis. The presence in the soil of vegetable and animal residues may introduce a considerable error, which may be minimised by the following procedure: 10 g. or more of the soil are added gradually to a 50- or 100-c.c. cylinder containing saturated sodium sulphate solution and standing in a flat dish. When most of the soil has settled, more of the solution is added to make the liquid overflow and carry the lighter matter with it. The liquid is stirred occasionally with a rod and the process is repeated. The matter thus removed is collected on either an ordinary or a Gooch filter, washed, and dried at 100°. The carbon of the residue, calculated as humus, may amount to 5—10% of the total present in the soil. T. H. POPE.

Permanganate method for the determination of the oxidisability of organic matter in water and aqueous soil extracts containing chlorides. J. N. ANTIPOV-KARATAËV (State Inst. Exp. Agr., Leningrad, Bull. Bur. Soils No. 3, 1928; Proc. Internat. Soc. Soil Sci., 1929, 4, 40–42).—To the sample (50 c.c.) are added 3 c.c. of sulphuric acid (1 : 3) and a weighed excess of silver sulphate to precipitate the chlorides (determined in a separate sample). After at least 1 hr. (the liquid being occasionally stirred) 10 c.c. of 0.01N-potassium permanganate are added, while stirring, and the liquid is heated gently for 10 min. If more permanganate is required further heating is necessary. 10 c.c. of 0.01N-oxalic acid are then added, and when the liquid has cleared completely the excess is titrated back with permanganate. Blank tests are necessary.

A. G. POLLARD.

Application of the strychnomolybdc process to the determination of phosphoric acid in soil. C. ANTONIANI and S. BONETTI (Giorn. Chim. Ind. Appl., 1929, 11, 154–155; cf. A., 1928, 979, 1265).—20 g. of the soil are boiled for 1 hr. with 30 c.c. of hydrochloric acid (*d* 1.18), 20 c.c. of nitric acid (*d* 1.40), and 50 c.c. of water. The cooled solution is made up to 500 c.c. with water, mixed, and filtered through a pleated paper, 50 c.c. of the filtrate being neutralised to phenolphthalein with 10% sodium hydroxide solution and the precipitated basic salts redissolved by addition of a few drops of 10% nitric acid. The clear liquid is mixed with 45 c.c. of the strychnomolybdc reagent and the precipitate, after 1 hr., is collected, washed with 100 c.c. of nitric acid (10 c.c. of acid [*d* 1.40] made up to 100 c.c.), and dried in a steam-bath to constant weight. Multiplication of the weight of precipitate by 0.0257 gives P_2O_5 , the volume occupied by the soil being allowed for by multiplying by 0.983 (experimental figure). To determine P_2O_5 soluble in 1% citric acid solution, 100 g. of the soil are treated in a Stohmann flask with the citric acid solution until all carbonate-carbon dioxide is expelled, a further quantity of 750 c.c. of the solution being then added. The flask is shaken for 5 hrs. in a revolving shaking apparatus and then kept for 12 hrs., after which 250 c.c. of the liquid (filtered if necessary) are evaporated to dryness on a water-bath. The residue is gently calcined with a few crystals of ammonium nitrate until all organic matter is oxidised, the ash being then heated with a little water and a few drops of nitric acid for some minutes on a water-bath, and the liquid is filtered. The neutralised filtrate is treated as in the determination of the total phosphoric acid. Test analyses yielded satisfactory results. T. II. POPE.

Gravimetric determination of carbonates in soils. N. A. CLARK and E. R. COLLINS (Soil Sci., 1929, 27, 407–414).—The customary method involving treatment of the soil with acid and the removal of the carbon dioxide by aspiration is modified. Ascarite is used as an absorbent and the air current is previously dried with phosphorus pentoxide. Dilute hydrochloric acid (1 : 10) is used and the soil suspension stirred instead of shaken during the aspiration. The operation is carried out at room temperature. At 50° there is a slight increase in the amount of carbon dioxide obtained.

A. G. POLLARD.

Acidimetric titration of silica and aluminium with an extension of the theory of acidimetric titrations. S. KÜHN (Sonderh. Ung. Kön. Geol. Anstalt Budapest, 1928, 34; Proc. Internat. Soc. Soil Sci., 1929, 4, 34).—A rapid method (10–15 min.) is described for determining silica in general analysis and aluminium in soil analysis in the presence of other soil constituents.

A. G. POLLARD.

Detection and significance of manganese dioxide in soil. W. O. ROBINSON (Soil Sci., 1929, 27, 335–350).—Manganese dioxide in soils decomposes hydrogen peroxide so much more rapidly than do other normal soil constituents that its presence in soil may be established by this means. Determinations are not satisfactory, since the rapidity of evolution of oxygen is dependent on the fineness of division of the manganese dioxide. The greater part of the manganese dioxide in soil appears in the silt fraction, a smaller amount in the sand, and none in the clay or colloidal fraction. In the soils examined manganese dioxide appears to be formed in concretionary deposits as a result of the precipitating effect of chalk.

A. G. POLLARD.

The seedling method [for determining soil nutrient values]. H. NEUBAUER (Z. Pflanz. Düng., 1929, 8B, 219–232).—The technique and practical application of the Neubauer and Schneider's original method (*ibid.*, 1923, 2A, 329) are discussed.

A. G. POLLARD.

Apparatus for mechanical analysis of soils. J. PADEREWSKI (Rocz. Nauk Koln. i Lesnych, 1928, 19; Proc. Internat. Soc. Soil Sci., 1929, 4, 48).—A modified elutriator apparatus is described in which the three vessels are arranged directly above one another, thus affording simplicity of manipulation and ease of repetition of determinations with the same soil suspension.

A. G. POLLARD.

Efficient soil tube jack. C. A. TAYLOR and H. F. BLANEY (Soil Sci., 1929, 27, 351–353).—The essential point of the jack is a lever operating a hinged conical grip closing on the soil tube at each lift and releasing and sliding down the tube with the return motion of the lever.

A. G. POLLARD.

Differential apparatus for measuring the volume of soils. J. PADEREWSKI (Stat. agric. d'exp. Kutno, 1925; Proc. Internat. Soc. Soil Sci., 1929, 4, 56–57).—Apparatus is described for the measurement of soil volumes by determining differences in air pressure resulting from the introduction of the soil sample into a closed space.

A. G. POLLARD.

See also A., June, 667, **Determination of phosphoric acids** (SOMEYA; STOLLENWERK and BAURLE; DWORZAK and REICH-ROHRWIG). 719, **Aluminium toxicity** (MCLEAN and GILBERT). 728, **Absorption of carbon dioxide by roots** (BERGAMASCHI). **Growth of the tomato** (NIGHTINGALE and others). **Behaviour of the olive under uranium radiations and ionised air** (PETRI). **Influence of nitrate ion on growth of summer wheat** (GOEDEWAAGEN). **Nutrition of higher plants with ammonia** (PRIANISCHNIKOV). **Nitrogen metabolism of forest soil** (FEHÉR).

PATENTS.

Dry disinfectant for seeds. L. and F. MEYER (CHEM. FABR. L. MEYER) (B.P. 286,220, 21.2.28. Denm., 28.2.27).—Iodine compounds such as mercuric iodide are mixed with known fungicidal materials. *E.g.*, a mixture of talc with mercuric chloride and iodide is used against bunt in wheat, stripe disease in barley, and smut in oats. Alternatively, mixtures of potassium iodide and mercuric chloride may be used. A preparation containing talc, mercuric chloride, potassium iodide, and tricalcium arsenate is used against crown gall in roots. A. G. POLLARD.

Disinfection of seeds and composition therefor. E. W. SCHMIDT, ASST. to J. D. RIEDEL—E. DE HAEN A.-G. (U.S.P. 1,711,545, 7.5.29. Appl., 2.7.26. Ger., 20.11.25).—A mixture of powdered calcium silicofluoride and copper carbonate is claimed. H. ROYAL-DAWSON.

Fertiliser. G. BARSKY and F. W. FREISE, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,695,081, 11.12.28. Appl., 25.6.23).—Leather scrap, or other nitrogenous organic waste material, is dissolved in sulphuric acid, and calcium cyanamide is stirred in. A porous solid fertiliser is obtained. C. HOLLINS.

Production of fertilisers. NORSK HYDRO-ELEKTRISK KVAELSTOF A./S. (Norw. P. 42,724, 9.9.24. Addn. to Norw. P. 41,452).—The mixture obtained by treating with nitric acid mineral silicates containing potassium, *e.g.*, leucite, is evaporated to dryness and then fused to expel nitrous gases; the gases are worked up to nitric acid and the residue is used as a fertiliser. L. A. COLES.

[Production of phosphatic] fertilisers. COMP. DE L'AZOTE ET DES FERTILISANTS SOC. ANON. (Swiss P. 122,821, 23.7.26).—Calcium phosphate etc. is treated at 20–100° with an acid, *e.g.*, sulphuric acid, in the presence of the ammonium salt of an acid which forms an insoluble calcium salt, *e.g.*, ammonium sulphate, and, if necessary, potassium salts, *e.g.*, potassium sulphate. L. A. COLES.

Emulsifiable oil (U.S.P. 1,695,197).—See II. **Spent sulphite liquors** (U.S.P. 1,710,272).—See V. **Treatment of phosphorites** (Russ. P. 3464).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Sugar factory filter cloths. I—III. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1929, 53, 321–329, 457–463, 483–492).—I. On a basis of 1000 dzn. of roots, the total filtering area used in beet-sugar factories is 27.2–68.6 m.²; the frequency of re-dressing is 4–40 days of the campaign, and the life of the cloths is 10–80 days. Jute cloths last on the average 10–14 days, and cotton up to 80 days. About 54% of the material used is cotton, 31% a mixture of cotton and jute, and 15% a mixture of linen and jute. Most factories now prefer cloths having the strengthened edge.

II. Filter-cloths of cotton, linen, jute, and of mixtures of these were submitted to various physical tests, as tearing strain, extension, rate of flow of water and molasses, and were also examined for hygroscopicity, density, regularity, etc., but these observations failed to lead to any definite standards by which their filtering and wearing qualities could be judged.

III. Cotton and linen cloths during use lost 63% of their original strength, as determined by the tearing strain, and jute about 46%, though these values are subject to wide variations, even in the same factory. Extension tests showed use to diminish this value, so that a cotton cloth which originally had a value of about 4% showed after use one about 1% lower. Figures for the rate of flow showed an immense retardation for a mixture of cotton and linen, but for pure cotton and jute fabrics the differences were considerably less, showing, in fact, sometimes a greater rate of flow. In general, these tests are complicated by other factors bearing on the problem of filtration. J. P. OGILVIE.

Combined milling and diffusion. W. R. McALLEP (Internat. Sugar J., 1929, 31, 279–280).—It seems improbable to the author that more economical means will ever be developed for extracting the first 80–90% of the sugar from cane than pressure in a couple of mills with ordinary maceration. Subsequent to this, however, the diffusion principle should be applied by removing the bagasse from the mill, and extracting the greater part of the remaining sugar by circulating the maceration water systematically. In this way extraction could be improved at moderate cost, whilst grinding at a greater rate than at present without the installation of costly additional mills is possible. J. P. OGILVIE.

Elimination of colloidal matter [from beet juices]. R. J. BROWN (Sugar Press, 1929, 13, 14–15).—As a positive colloid for the removal from beet juice of the negatively-charged colloidal matter, chromium oxide hydrosol proved to be the most effective, the iron oxide preparation being the least so. Alumina hydrosol also gave good results, and when equal volumes of it and beet juice were mixed a good coagulation resulted, the filtrate containing only a negligible quantity of colloidal matter. J. P. OGILVIE.

De Vecchis' process for extracting sugar from beet. I. GIORDANO (Giorn. Chim. Ind. Appl., 1929, 11, 155–163).—Descriptions are given of earlier attempts to dry beet, and of the plant and detailed working, including the economics, of the De Vecchis process. T. H. POPE.

Evaporation of [beet] juices in the presence of active carbons. V. STANĚK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1929, 53, 493–499).—A small amount (0.2%) of decolorising carbon present in the thin juice during its evaporation acted very favourably in diminishing its final colour. "Carboraffin" gave better results than "Standard Norit." When the active carbon was used in combination with sodium sulphite (0.01%), a juice resulted which was distinctly lighter in colour than the original. J. P. OGILVIE.

Effect of carbon monoxide and other impurities of saturation gas on carbonation [of sugar solutions]. R. SIGMUND (Z. Zuckerind. Czechoslov., 1929, 53, 473–479).—Using saturation gas containing 40% CO₂ throughout, it was observed that the most rapid saturation was achieved with the pure gas. It was diminished with increasing amounts of carbon monoxide, so that with 10% of this impurity the rate fell to less than half. Even 0.5% appreciably retarded

the operation. Hydrogen sulphite and tarry matters exerted no marked effect. J. P. OGILVIE.

Determination of sugar in bagasse by cold extraction : new figure for milling control. H. EGETER (Archief, 1928, 36, II, 691—729).—In the cold-extraction method of Khainovsky and Egeter (Med. Java-Proefstat., 1927, No. 4) for the determination of sucrose in bagasse, a lower polarisation is obtained than in the ordinary method, this having been proved to be due in the new procedure to the presence of intact cells incapable of extraction without heat. Based on this observation, the author elaborates a method of estimating the quantity of juice left in the unruptured cells after milling, and it is proposed that this figure should be used as a standard for the control of milling in order to indicate the efficiency of crushing.

J. P. OGILVIE.

Analysis of cane sugar. P. HONIG (Archief, 1928, 36, II, 639—647).—Reducing sugars are determined by Schoorl's method (cf. Internat. Sugar J., 1919, 21, 578, 621) after clarification with normal lead acetate, followed by sodium phosphate. The size of grain is found by a series of standard Tyler sieves giving fractions from above 1.65 mm. down to less than 0.3 mm. (dust). Colour determination is expressed by values obtained by means of the König-Martens spectrophotometer after carefully filtering the solution and adjusting its p_H to 7.0, measurements being made at six different wavelengths. J. P. OGILVIE.

See also A., June, 682, **Oxidation of dextrose by alkaline copper solutions** (LUNDIN). 683, **Synthesis of sucrose** (ZEMPLÉN and GERECES). **Condensation of dextrose and laevulose ; isosucrose** (IRVINE and others). 702, **Beet-saponin** (REHORST). 722, **Sugar-hydrolysing enzymes** (WEIDENHAGEN).

Sugar cane soils. ARRHEIUS.—See XVI.

PATENTS.

Dehydration of vegetable substances [e.g., sugar-beet slices] of organic character. B. J. OWEN, Assr. to SUGAR BEET & CROP DRIERS, LTD. (U.S.P. 1,713,619, 21.5.29. Appl., 5.11.27. U.K., 5.11.26).—See B.P. 285,115 ; B., 1928, 311.

Reduction of sugars to alcohols (U.S.P. 1,712,952).—See XI.

XVIII.—FERMENTATION INDUSTRIES.

Modern malting methods. G. CHABOT (Woch. Brau., 1929, 46, 208—211).—The alkaline steep advocated by Moufang is criticised, and experimental results are given which indicate that a steep-water adjusted to p_H 6—7 is best as regards uptake of water by the barley. With slightly more alkaline or acid steep-waters, the p_H tends to adjust itself to the above value, but with too acid waters (p_H about 4) this may be slight or the water may become still more acid. This effect is more marked when sulphurous acid is used for neutralising a calcium carbonate water than when sulphuric acid is used. The use of alkaline steep-water leads to darkening of the grain. F. E. DAY.

Storing and redrying malt. G. D. CLARKSON (J.

Inst. Brew., 1929, 35, 282—287).—The moisture content of malt which is to be crushed for brewing should not much exceed 3%. For the construction of bins for the storage of malt, wood alone or brick or concrete with wood lining is the most satisfactory material, for it does not damage the grain and is impervious to moisture. Probably the best method for redrying malt which has taken up moisture during storage is in a "drum." Heat must be applied slowly during the first 12 hrs., and the temperature raised to 38°. In 18 hrs. the temperature should reach 49° and in 24 hrs. 64°, at which stage a strong draught is necessary. For amber or thick-skinned malt 77—82° may be reached in 48 hrs., but for pale malt those temperatures should not be reached until 72 hrs. have elapsed. By redrying, the colour increases 1—3 points, the matters soluble in cold water rise slightly, and the diastatic power remains almost stationary. C. RANKEN.

Use of Irish moss as copper finings [in brewing]. C. RANKEN (J. Inst. Brew., 1929, 35, 287—291).—The flocculation of peptone in boiling wort by means of Irish moss remains practically uninfluenced by the concentration of the peptone and other wort constituents. It is sensitive to the reaction of the wort and to the concentration of the moss per barrel of wort. To obtain optimum flocculation, a greater amount of free acidity in the wort must be met with the addition of a greater amount of Irish moss per barrel. Should a greater amount of moss be added than that required for the p_H value of the wort, the resulting wort will become hazy on the production of acid during fermentation, with possible prejudicial effects on the yeast. The insoluble cellulosic skeleton of the moss is also an important factor in the clarification of the wort. C. RANKEN.

Biology of sarcina. J. FUCHS (Woch. Brau., 1929, 46, 203—208, 213—215).—The development of sarcina in beer is not directly due to incomplete saccharification in the mash tun, but may be so caused indirectly owing to weakening of the yeast. For the cultivation of the organisms slightly alkaline (p_H 8) media are most favourable especially in presence of ammonia, and a test for slight sarcina infection is based on this. In beers the presence of complex proteins is favourable to the growth of sarcina, hence badly modified or high-nitrogen malts and conditions which favour poor proteolysis in the mash tun should be avoided. Especially favourable are the products of the autolysis of yeast. Experiments are described illustrating these points and indicating a direct antagonism between yeast and sarcina, the former being apparently capable of excreting a lysin destructive to sarcina. There is no evidence of parasitism of sarcina on, or symbiosis with, yeast.

F. E. DAY.

Wine distillates and wine brandies. E. RUPPIN (Z. Unters. Lebensm., 1929, 57, 243—244).—It is shown on theoretical considerations based on the law of Guldberg and Waage that, contrary to the statement of Büttner and Miermeister (B., 1929, 373), unadulterated wine brandy may contain much less than 100 mg. of ester in 100 c.c. of pure alcohol. The necessity for tasting tests in evaluating these products is emphasised.

W. J. BOYD.

Examination and evaluation of wine distillates and wine brandies. H. WÜSTENFELD and C. LUCKOW (Z. Unters. Lebensm., 1929, 57, 242—243).—Contrary to the views of Büttner and Miermeister (B., 1929, 373), the necessity of tasting tests in evaluation of still wines and their distillation products is emphasised and the fixing of minimum values is considered undesirable. The value of analytical data is admitted in conjunction with tasting tests on the individual fractions obtained by rectification. W. J. BOYD.

Detection of fruit wine in wine by identification of dibenzylidenesorbitol. C. VON DER HEIDE and K. HENNIG (Z. Unters. Lebensm., 1929, 57, 240—241).—The observations of Werder (Mitt. Gebiete Lebensm. u. Hyg., 1928, 19, 294) that fruit wine always contains sorbitol whilst wine never does were confirmed. It is necessary, however, in using Werder's method to examine the precipitate by washing it with alcohol and ether to remove water and various organic substances and then extracting the dibenzylidenesorbitol with benzene, recrystallising it from benzene, and determining its m.p., which should be 162° (uncorr.). The presence of tribenzylidenemannitol, m.p. 213—217° (uncorr.), may otherwise lead to wrong conclusions. W. J. BOYD.

Detection of isopropyl alcohol in presence of acetone [in spirit]. W. MEYER (Chem.-Ztg., 1929, 53, 410—411).—50 g. of spirit are distilled and the first 5 c.c. of distillate are tested for methyl alcohol and acetone. The second fraction of 20 c.c. is divided, half being oxidised with dichromate and sulphuric acid. An increase in acetone content after oxidation indicates the presence of isopropyl alcohol in the original spirit.

J. S. CARTER.

See also A., June, 721, **Malt amylase** (SABALITSCHKA and WEIDLICH). **Takadiastase** (OHLSSON and SWAETICHIN). 722, **Enzymes and light** (OYA). **Sugar-hydrolysing enzymes** (WEIDENHAGEN). **Action of invertase** (COLIN and CHAUDUN). **Barley-malt catalase** (CHARMANDARJAN). 724, **Alcoholic fermentation with dried yeast** (KISCH and LEIBOWITZ). **Fermentative bacteria in maceration juice** (KOSTYTSHEV and SCHULGINA). **Conversion of acetic acid by *Mucor stolonifer*** (BUTKEVITSCH and FEDOROV).

Starch value of frozen potatoes. LAMPE and KILP.—See XIX.

PATENTS.

Yeast food [for bread-making]. O. R. BROWN (U.S.P. 1,712,025, 7.5.29. Appl., 22.8.27).—The food is composed of potassium nitrate, calcium phosphate, and magnesium sulphate. H. ROYAL-DAWSON.

Manufacture of sparkling wine. C. M. CHARTIER (F.P. 628,964, 8.2.27).—Wine with added sugar is fermented with yeast at 25—28° in an enamelled steel vessel in which the pressure of carbon dioxide is allowed to reach and is retained at 5 atm. The fermented liquid is filtered under air pressure into a collection vessel where it is chilled to 2—5° and saturated with carbon dioxide. After repeated filtration under pressure of carbon dioxide the wine is bottled. The process requires 8—10 days and yields a wine ready for immediate consumption. C. RANKEN.

Manufacture of fruit wine. P. K. ERBEN (Swiss P. 122,802, 2.8.26).—The wine, which retains its brilliancy and has a low alcohol content, is prepared by partly fermenting pressed juice and, after removal of the yeast, adding a sulphite to destroy bacteria. After clarification with a liquid containing tannin, the juice is kept cold to retard fermentation, and as soon as the yeast has separated is filtered through an ultra-filter so that a secondary fermentation may be avoided. C. RANKEN.

XIX.—FOODS.

Action of chlorine on wheaten flour in treatment with gologas. M. VUK and A. GÖMÖRY (Z. Unters. Lebensm., 1929, 57, 177—191; cf. Vuk and Spányár, B., 1929, 299).—Chlorine forms with the starch neither a chemical nor an adsorption compound which has any influence on the properties of the flour. It alters the balance between the gliadin and glutelin, so that it becomes difficult to obtain the gluten by washing the flour. The proportion of the various amino-acids in the gliadin and glutelin of untreated, chlorinated, and over-chlorinated flours, respectively, were determined as far as possible. The greatest decreases were in the quantities of amide-nitrogen and glutamic acid, and to a less extent of aspartic acid and arginine. The water-soluble nitrogen was found to increase on chlorination. It is concluded that the glutamic and aspartic acids, in the form of glutamine and asparagine, are so united to other amides, mono- or di-amino-substances, that they are set free on treatment of the flour with gologas, and become soluble as free amino-acids or their hydrochlorides. The chlorine forms addition products with the unsaturated glycerides of the flour fat, and the carotin is bleached by conversion into the water-soluble dicarotin. W. J. BOYD.

Electrical conductivity of milk. I. Detection of abnormal milk from diseased cows. J. KRENN (Z. Unters. Lebensm., 1929, 57, 148—177).—Determination of the electrical conductivity of milk is shown to be of great utility in the detection of milk from cows with diseased udders. Such milk has invariably a higher electrical conductivity than normal milk, owing to its higher sodium chloride content. W. J. BOYD.

Rapid analysis of butter. N. CHARLIERS (Chim. et Ind., 1929, 21, 931—936).—In the saponification *n*-propyl alcohol is used, and under the specified conditions this is salted out along with the higher fatty acids. The small quantity remaining in the aqueous phase has no disturbing effect. The butyric acid removed in the separated alcohol is made constant in quantity by adding potassium stearate, equivalent to 1 g. of stearic acid, to the solution after saponification. Butter (5 g.) is heated under reflux for 20 min. with 25 c.c. of 6% caustic potash solution in *n*-propyl alcohol, 0.2 c.c. of a 2% solution of phenolphthalein in *n*-propyl alcohol is added, and the excess of alkali is titrated with *N*-sulphuric acid. The saponification value is calculated in the usual way. If *n* c.c. of *N*-sulphuric acid have been used, there are added further: (a) (31.8 — *n*) c.c. of water; (b) 100 c.c. of a solution of 10 g. of stearic acid in 4 c.c. of 75% caustic potash solution, mixed with 10 c.c. of *n*-propyl alcohol and made up to 1 litre; (c) 10 c.c. of a solution of 100 g.

of coconut oil in 40 c.c. of 75% caustic potash solution, mixed with 100 c.c. of *n*-propyl alcohol and made up to 1 litre; (d) 5 c.c. of 33% sulphuric acid; (e) 15 g. of anhydrous sodium sulphate; and (f) a pinch of kieselguhr. The mixture is shaken, allowed to rest for 20 min., and 125 c.c. are filtered off. Then 50 c.c. of distilled water are added with some pieces of pumice, and 110 c.c. of distillate are collected and titrated with 0.1*N*-caustic soda. The butyric acid number is $(k-t) \times 1.75$, where *k* and *t* are the numbers of c.c. of 0.1*N*-caustic soda used in the determination and in a blank test, respectively. Cacao butter should be used for the blank experiment. The values obtained for the butter content by this method are quite as concordant with those calculated from the Leffmann-Beam value, as are those obtained by the methods of Kuhlmann and Grossfeld (B., 1926, 447) and Van Raalte (B., 1926, 563).

W. J. BOYD.

Chemical detection of vitamin-C. B. GLASSMANN and A. POSDEEV (Z. Unters. Lebensm., 1929, 57, 191—200).—The Bezssonov reaction (B., 1921, 747) supposed to be due to vitamin-C is given by tannins at ordinary temperatures, and by carbohydrates and other plant substances at the temperature of the boiling water-bath. Until the vitamins have been isolated and their reactions studied, their detection is only possible biologically.

W. J. BOYD.

Effect of fumigation by hydrogen cyanide on fresh fruit and vegetables. P. BUTTENBERG (Z. Unters. Lebensm., 1929, 57, 204—211).—No effect on plums, grapes, shallots, and onions has been established, and the effect on parsley and tomatoes is doubtful. Early potatoes do not withstand the treatment, but winter potatoes are resistant. In sprouted, wintered potatoes the germ and surface layer of the tuber may be destroyed. On aeration of potatoes after fumigation, an effect like that of frost may be produced. Freshly-harvested Swedish turnips are less resistant than those kept through the winter. Apples, bananas, pears, oranges, lemons, quinces, cucumbers, different kinds of cabbage, carrots, radishes, lettuces, rhubarb stalks, beets, turnips, celery, spinach, and comfrey are damaged. The damage is not always so great as to exclude an early consumption of the produce, and may not appear for some weeks.

W. J. BOYD.

Loss of iodine and other inorganic substances from spinach in the usual cooking process. A. MIERMEISTER (Z. Unters. Lebensm., 1929, 57, 235—239).—A loss of 44% of the total mineral matter was observed in cooking 2 kg. of spinach in 3 litres of boiling tap water to which 15.8 g. of salt had been added, the water being finally poured off and the vegetable washed with distilled water. Of this loss 59% was due to K_2O , amounting to 57% of the quantity of that base originally present, but the cooked and washed spinach still contained a notable excess of inorganic bases over acids. 100 g. of fresh spinach contained 0.044 mg. of iodine, of which 0.0035 mg. was lost in cooking. The iodine appears to be organically combined, and consequently insoluble. Methods of analysis are described.

W. J. BOYD.

Determination of starch value of sound and

frozen potatoes, with reference to their use for distilling and drying. B. LANPÉ and W. KILP (Z. Spiritusind., 1929, 52, 177—178).—Two samples, partly frozen in the clamp, were obtained, one apparently sound, the other softened and attacked by bacteria. The starch contents by (1) calculation from sp. gr., (2) acid hydrolysis and Fehling's solution, (3) hydrolysis, fermentation, and determination of the alcohol, and (4) subtracting 5.8% from the dry matter, were, respectively, 16.7, 16.97, 16.82, and 16.77% on the sound and 12.0, 23.45, 24.26, and 24.75% on the unsound sample. The applicability of the determination from the sp. gr. and the effect of the damage resulting from freezing on the value for distilling and drying are discussed. (Cf. B., 1929, 338.) F. E. DAY.

Rapid determination of the oil content of paprika. J. DÖMÖRÖR (Z. Unters. Lebensm., 1929, 57, 239—240).

—The material (2.5 g.) is shaken for 1 hr. with 40—60 c.c. of ether, or the quantities may be modified according to the quality of the product, the relation of material to solvent being kept the same. The solvent is decanted off through three filter papers previously wetted with ether. The filtrate is passed through the filters a second time, and filters and residue are well washed with ether. The clear filtrate is transferred to a tared flask, the ether evaporated off, the oil dried at 105°, and weighed. The whole procedure is completed in 4 hrs.

W. J. BOYD.

Rapid method of detecting lemonade containing saccharin. J. VONDRÁK (Z. Zuckerind. Czechoslov., 1929, 53, 501—503).—Lemonade sweetened with saccharin can be detected by its low refraction or low density. The former may be determined by the immersion or sugar refractometer, and the latter by the saccharometer or by means of floats made of approximately equal parts of carnauba wax and asphalt, adjusted so that they neither float nor sink in 6% sugar solution. This concentration may be taken as the minimum allowable in lemonade.

W. J. BOYD.

Goose-liver preparations. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1929, 57, 212—216).—Definitions of "pâté de foie gras" by various authorities are quoted, and analyses of various products of this nature from different sources are given. W. J. BOYD.

Examination and evaluation of cray-fish soups. E. BAIER and H. BARSCH (Z. Unters. Lebensm., 1929, 57, 224—234).—Methods for the detection of artificial colouring matter in cray-fish soups are described involving extraction of the pigment with alcohol and testing of its affinity for wool, its reaction with concentrated sulphuric acid, and its behaviour in ultra-violet light, in which solutions of the natural colouring matter show fluorescence and those of synthetic dyes do not. Microscopical examination of the foodstuff itself and observation of its taste and odour when cooked are useful in the examination of these products.

W. J. BOYD.

Iodine values. GIRAL. **Comestible flours.** ANDRÉ.—See XII. **Germicidal washing solutions.** MYERS.—See XXIII.

PATENTS.

Maturation of wheat, maize, pulse, and other carbohydrate-containing cereals and seeds, and

the flour or other similar products derived therefrom. R. W. DUNHAM (B.P. 311,121, 30.5. and 5.6.28).—Apparatus is described for artificially continuing the ripening process of the endosperm of grain, comprising several compartments which the product is made to traverse, and in which it may be repeatedly subjected in one to heat and light, and in the next to cold and darkness. W. J. BOYD.

Making of cheese. H. P. KERNEN (U.S.P. 1,708,099 and 1,708,100, 9.4.29. Appl., [A] 18.2.27, [B] 21.11.27).—Sterile cheese is made directly in one continuous process by coagulating the milk, forming the curds, and then rapidly ripening by (A) developing the requisite acidity in the curds by artificial means in a relatively short space of time, viz., by heating for 12 hrs. at 30–40°, (B) adding $\frac{3}{8}\%$ by wt. of each of calcium phosphate, calcium lactate, and ammonium citrate. The whole is then sterilised by heat in the presence of an emulsifying agent while stirring, and allowed to cool.

F. R. ENNOS.

Manufacture of cheese. W. D. RICHARDSON, Assr. to SWIFT & Co. (U.S.P. 1,711,032, 30.4.29. Appl., 26.7.27).—Dried milk powder is mixed with whole milk to form a mixture having approximately the moisture content desired for the finished cheese, and the product is coagulated.

W. J. BOYD.

Preservation of eggs. A. V. STEWART, and FARMA CREAM PRODUCT CO., LTD. (B.P. 311,877, 24.3.28).—Eggs are coated with an 8% solution of celluloid in glycol monoethyl ether. The solution may be coloured if desired. Various methods of application are described. It is claimed that eggs so preserved remain fresh for 12 months.

E. B. HUGHES.

Food product and its production. CALIFORNIA FRUIT GROWERS' EXCHANGE (B.P. 283,580, 3.1.28. U.S., 15.1.27).—Prior to dehydration of fruit juice, 4% or less of a gum-like hydrophilic colloid such as pectin, agar, or gelatin is dissolved in it, so as to suppress hygroscopicity in the fruit juice solids. W. J. BOYD.

Percentage composition of diluted substances (B.P. 285,383).—See I. Yeast food (U.S.P. 1,712,025).—See XVIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Buffer capacities of acacia and tragacanth. J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1929, 18, 469–473).—With emulsions prepared with acacia the buffering action of acacia on acid or alkali added to the emulsion influences the stability of the emulsion. The buffer capacity is more effective in the neutralisation of acids, the instability of the emulsion beginning on the alkaline side at a lower concentration of alkali than it does on the acid side with a corresponding concentration of acid. Dilution with water has little influence on the p_H of acacia solutions. With tragacanth the buffering effect does not appear to have as great an influence on the stability of the emulsion as does acacia. The Van Slyke " β " (cf. A., 1922, i, 893) for acacia in 0.1M-solution at p_H 3 is 0.034. E. H. SHARPLES.

Emulsifying properties of certain salts of arabic acid. J. C. KRANTZ, JUN., and N. E. GORDON (J. Amer.

Pharm. Assoc., 1929, 18, 463–468).—Metallic salts of arabic (from acacia), oleic, valeric, gluconic, *i*-galactonic, dihydroxystearic, salicylic, and gallic acids have been prepared and their emulsifying properties compared. An explanation for the production of oil-in-water emulsions by acacia and tragacanth is proposed.

E. H. SHARPLES.

Comparative study of methods of determination of citral in lemon oil; constants of the Spanish oil. O. FERNÁNDEZ and A. MOSCARDO (Anal. Fis. Quim. [Tecn.], 1929, 2, 265–278).—The methods of Walther (B., 1900, 78; 1901, 289), and of Bennett for the determination of citral, using hydroxylamine hydrochloride, yield high results, whilst that of Bennett and Salamon (B., 1928, 68) gives concordant, but slightly high results. Tiemann's method, based on the use of sodium sulphite and titration of the liberated alkali, yields high results. The method of the U.S. Pharmacopœia, using phenylhydrazine, is satisfactory, but that of Ardagh and Williams (A., 1926, 189) yields low results. The following data are recorded for Valencian lemon oil: d_{4}^{25} 0.8587–0.8673, n_D^{20} 1.473, $[\alpha]_D^{20}$ –68.5° to –69.17°, citral content 2.4–2.65%. Oil from unripe fruit had d_{4}^{25} 0.8577, n_D^{20} 1.474, $[\alpha]_D^{20}$ –67.3°, and a noticeably higher citral content (3.2%). R. K. CALLOW.

See also A., June, 666, Ultra-violet colorimetry and fluorescent substances (EISENBRAND). 668, Determination of arsenic (VILECZ; GNESSIN). 676, Decoic acids as bactericides (STANLEY and others). 707, Caffeino-salicylic acid (SCHOORL). Alkaloids of Angostura bark (TRÖGER). Microchemical reactions of physostigmine (WAGENAAR). Acids from brucine (WIELAND and MÜNSTER). 708, Determination of brucine and analysis of nux vomica (KLJAT-SCHKINA and STRUGADSKI). New strychnos alkaloid (WIELAND and OERTEL). 709, Apomorphine alkaloids (CALLOW and others). Sulphur derivatives of aromatic arsenicals (EVERETT). 710, 10-Chloro-5:10-dihydrophenarsazine and its derivatives (GIBSON and JOHNSON). 711, Organic compounds of mercury (NESMEJANOV). 725, Mercuric oxycyanide compounds and *Gonococcus* (TAKAORA). 729, Methyl alcohol from tobacco smoke (NEUBERG and KOBEL). Determination of nicotine in fresh green tobacco (BODNÁR and NAGY). 730, Phloroglucinol drugs (PEYER and LIEBISCH).

PATENTS.

Manufacture of lipoids, particularly phosphatides. "PHARMAGANS" PHARM. INST. L. W. GANS A.-G., W. KOLLATH, and H. MAGISTRIS (B.P. 311,436, 10.1.28. Addn. to B.P. 285,417; B., 1929, 110).—The dialysis solutions described in the prior patent are subjected to a rapid preliminary concentration, e.g., in compression evaporators, and are then evaporated rapidly to dryness at a low temperature, e.g., by the Krause or Hatmaker process, exposure to the air being reduced to a minimum during the processes. The dry products, the solutions, or the original material may be exposed to the action of ultra-violet rays.

L. A. COLES.

Extraction of natural animal and vegetable material. H. SCHMIDT (G.P. 451,000, 17.11.25).—The

material, *e.g.*, gum benzoin, tolu balsam, storax, cloves, sandal wood, etc., is extracted with a light, readily volatile solvent which, in the same operation, is displaced by a high-boiling liquid, *e.g.*, benzyl alcohol or its esters, ethyl phthalate, glyceryl triacetate, etc., which absorbs completely the constituents extracted by the volatile solvent.
L. A. COLES.

Manufacture of pinacol. H. BERLIN, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,703,220, 26.2.29. Appl., 20.12.26).—Acetone is agitated and boiled with aluminium (sheet or shavings) and carbon tetrachloride. Mercuric chloride is then added and, after 10–15 min., further carbon tetrachloride is gradually added as the reaction slows down. The product is diluted at 40–50° with water, the filtrate and washings are evaporated in presence of sodium carbonate, and the residue is crystallised from water. Anhydrous pinacol is obtained by treating the molten hydrate with 50% sodium hydroxide, or, alternatively, 50% sodium hydroxide may be added direct to the reaction mixture when addition of carbon tetrachloride is complete.
R. BRIGHTMAN.

Manufacture of therapeutically active basic nitro-derivatives of 9[10]-aminoacridine. I. G. FARBENIND. A.-G. (B.P. 283,510, 11.1.28. Ger., 11.1.27).—Powerfully active bactericides are obtained by condensing a 10(m)s-chloro-3-nitroacridine with a diamine containing a primary and a tertiary amino-group, or by condensing with ammonia or a primary amine a 10-chloro-3-nitroacridine containing already a strongly basic tertiary amino-group in a suitable substituent. An ether group may replace the 10-chloro-atom in the starting-material. The following are described: 3-nitro-10- β -diethylaminoethylamino-8-ethoxyacridine [dihydrochloride, m.p. 245–246° (decomp.)] from 10-chloro-3-nitro-8-ethoxyacridine by way of the 10-phenoxy-compound and β -diethylaminoethylamine; 3-nitro-10- γ -diethylamino- β -hydroxypropylamino-8-ethoxyacridine, m.p. 108° [dihydrochloride, m.p. 226–227° (+ 5H₂O)]; 3-nitro-10-p- β -diethylaminoethylaminoanilino-8-ethoxyacridine, m.p. 120° (decomp.); the corresponding 10-p- γ -diethylamino- β -hydroxypropylaminoanilino-compound, m.p. 131–132°, from γ -diethylaminoethyl- β -hydroxypropyl-p-phenylenediamine, b.p. 185°/2.5 mm., which is obtained from p-aminoacetanilide and epichlorohydrin with subsequent treatment with diethylamine and hydrolysis; the corresponding 10-p- γ -(β' -diethylaminoethylamino)- β -hydroxypropylaminoanilino-compound, m.p. 86°, from γ -(β' -diethylaminoethylamino)- β -hydroxypropyl-p-phenylenediamine, b.p. 230°/3 mm.; 3:7-dinitro-10-p- β -diethylaminoethoxyanilino-8-ethoxyacridine, m.p. 155°, by condensing 2-nitro-p-phenetidine with 2-chloro-4-nitrobenzoic acid to a diphenylamine, m.p. 254–255°, which is cyclised to the acridone and converted into 10-chloro-3:7-dinitro-8-ethoxyacridine, m.p. 194–196°, and finally condensed with p-aminophenyl β -diethylaminoethyl ether, b.p. 175°/13 mm.; 3-nitro-10-amino-8- β -diethylaminoethoxyacridine, m.p. 237–238° [dihydrochloride, m.p. 265° (+ 3H₂O)], by the action of ammonia on the 10-chloro-compound, m.p. 159–160°, obtained by condensing 2-chloro-4-nitrobenzoic acid with p-aminophenyl β -diethylaminoethyl

ether to a diphenylamine, decomp. 226°, cyclising, and treating the acridone with phosphorus pentachloride.

C. HOLLINS.

Organic compound of mercury and its manufacture. E. C. WHITE (U.S.P. 1,692,237, 20.11.28. Appl., 27.1.27).—Dibromosulphonfluorescein, from o-sulphobenzoic anhydride and resorcinol, dibrominated, is boiled with mercuric acetate, the precipitate is dissolved in just sufficient alkali, and the solution is evaporated to yield a disinfectant, germicidal mercury derivative suitable for local application or injection.

C. HOLLINS.

Manufacture of stable medicinally active salts of p-aminophenylstibinic acid. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 311,448, 11.2.28).—The acid is converted into a salt of a secondary amine, *e.g.*, diethylamine or piperazine.
L. A. COLES.

Esters of cholesterol with unsaturated acids. W. MINNICH, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,690,647, 6.11.28. Appl., 10.11.24. Switz., 19.11.23).—See B.P. 243,510; B., 1926, 141.

[Manufacture of] 8-amino-6-alkoxyquinolines. W. SCHULEMANN, F. SCHÖNNHÜFER, and F. MIETZSCH, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,703,365, 26.2.29. Appl., 30.1.26. Ger., 29.4.25).—See B.P. 275,277; B., 1927, 797.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., June, 660, Phototropic mercury compounds (RAO and WATSON). Formation of latent images (TOY and HARRISON).

PATENTS.

Manufacture of photographic silver salt emulsions. I. G. FARBENIND. A.-G. (B.P. 283,223, 7.1.28. Ger., 7.1.27).—Sensitiveness of photographic silver salt emulsions is increased 30–50-fold by the addition of organic compounds, containing 1 or 2 sulphur atoms, which give silver derivatives gradually transformed into silver sulphide. Such are organic disulphides containing carboxyl groups (disulphidoacetic acid, $\alpha\alpha$ -disulphidopropionic acid), certain thiol derivatives [thiomalonic ("thiomalic") acid, potassium dithiocyanate, phenyl $\beta\beta$ -dithiolvinyl ketone], and some cyclic sulphur compounds (cyclic disulphide of β -thiolthiocinnamic acid). In the example 0.015 g. of potassium dithiocyanate is added to an emulsion made from 1 kg. of silver nitrate.
C. HOLLINS.

Colour photograph or film and its production. F. E. IVES, Assr. to C. W. FENNINGER (U.S.P. 1,695,284, 18.12.28. Appl., 20.10.26).—Undesirable effects of blue toning on the silver bromide in a colloid layer containing a coloured image produced in multicolour processes are corrected by treatment with a solution of 45 g. of sodium sulphate and 3 g. of potassium bromide in 2 litres of water.
R. BRIGHTMAN.

[Recording and projection apparatus for] photography and cinematography in colours. Soc. CIVILE POUR L'ETUDE DE LA PHOTOGRAPHIE ET DE LA CINÉMATOGRAPHIE EN COULEURS (B.P. 293,047, 29.6.28. Fr., 30.6.27).

XXII.—EXPLOSIVES; MATCHES.

Comparative tests of the initiating powers of lead azide and mercury fulminate detonators. B. CSERNECZY (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 169—171).—The method employed was in principle that of Wöhler, but phlegmatised picric acid was substituted for phlegmatised T.N.T. as the colour of the smoke from the explosion could be used to indicate whether combustion was complete, being black when complete and yellow when incomplete. The arrangement of the apparatus was that of Hess. The results showed that a mixture of lead azide and lead styphnate is much more powerful than fulminate as a primary charge, and that tetryl is a better secondary charge than trotyl. A recess in the base of the detonator increases the initiating power of copper-tube detonators, but diminishes that of aluminium-tube detonators. Prolonged storage in a moist atmosphere diminishes neither the ease of ignition nor the initiating power of lead azide-lead styphnate detonators. S. BINNING.

See also A., June, 664, Fluorine and chlorine as explosive gas mixtures (FREDENHAGEN and KREFFT). 730, Purification of picric acid (BENEDICT).

XXIII.—SANITATION; WATER PURIFICATION.

Germicidal properties of alkaline washing solutions, with special reference to the influence of hydroxyl-ion concentration, buffer index, and osmotic pressure. R. P. MYERS (J. Agric. Res., 1929, 38, 521—563).—The investigation was undertaken to determine some of the factors influencing the germicidal efficiency of various alkalis and basic salts, particularly in regard to the cleaning of milk bottles. Tests were made with several commercial washing powders and attention was paid to the relation between hydroxyl-ion concentration and germicidal efficiency. In general, the powders giving high p_H values were more effective as germicides than those giving low p_H values, but a powder containing chlorine was an exception to this. The effectiveness of the poorer powders was increased by the addition of sodium hydroxide. On dilution the efficiency of the powders decreased, but at various rates, and the decrease in efficiency seemed to be closely correlated with the decrease in hydroxyl-ion concentration. A combination of high hydroxyl-ion concentration and high temperature is effective in destroying spores of *Bacillus cereus* within a few minutes, whilst either factor alone is ineffective. A study was made of the influence of the hydroxyl-ion concentration, buffer index, and osmotic pressure of alkaline solutions on their germicidal action, using spores of a spore-forming aerobe to measure germicidal action. The death rate of spores in the alkaline solutions did not follow the logarithmic rate, but increased as disinfection proceeded. The death rate was increased by (1) an increase in the hydroxyl-ion concentration when the buffer index and osmotic pressure were kept constant, (2) an increase in the buffer index, keeping the hydroxyl-ion concentration and the osmotic pressure constant, (3) an increase in the osmotic pressure when the hydroxyl-ion concentration and buffer index were kept constant. Of these three factors, the osmotic pressure has the least

influence. Neutral sodium hypochlorite is a more effective germicide than alkaline sodium hypochlorite having the same amount of available chlorine. The alkaline hypochlorite solution with available chlorine present in 0.01% concentration was similar in germicidal action to 0.25*N*-sodium hydroxide. Measurements were conducted on the rate of hydrolysis of gelatin in strongly alkaline solution. The effect of osmotic pressure was not appreciable, but the higher the buffer index and the hydroxyl-ion concentration, the greater was the rate of hydrolysis. A titration method, which consists of determining the amount of alkali that is capable of maintaining a p_H above 12.0, has been proposed as a means of measuring quickly the germicidal power of an alkaline washing solution. E. S. HEDGES.

Sterilisation of water and other liquids by the application of electrical potentials. G. LAKHOVSKY (Compt. rend., 1929, 188, 1069—1071).—Data are given for the bactericidal action produced by the application of electric potentials to silver electrodes immersed in liquid media in the case of *B. coli* and *B. typhosus*.

E. A. LUNT.

Chlorination of the water supply, particularly for tanneries. M. AUERBACH (Collegium, 1929, 104—109).—Two methods are employed: (a) direct, in which chlorine gas is passed into the water supply, and (b) indirect, in which some water highly charged with chlorine is mixed with the water supply. The amount of chlorine necessary depends on the individual water, but is usually 0.1—0.3 g./m.³ for a drinking water. Less than this suffices for a tannery water. There is no deleterious action on the pelt. Chlorinated water hinders putrefaction even if it contains no free chlorine. Chlorine is useful in deodorising and disinfecting tannery effluents. A large amount of chlorine is required to destroy anthrax spores.

D. WOODROFFE.

See also A., June, 672, Continuous still for conductivity water (DE WITT and BROWN). 725, α -Bromo-soaps as germicides (EGGERTH).

Phenol recovery from coal-tar effluents. HATCH.—See II. Cast-iron pipes. BRADSHAW. Mercurial poisoning. STELLING.—See X. Determination of organic matter in water. ANTIPOV-KARATAEV.—See XVI.

PATENTS.

Removal of scale or incrustations from boilers or preventing the formation of same. E. D. FELDMAN (B.P. 311,550, 7.5.28).—A composition is claimed consisting of an alkali soap of a fat or fatty oil which, after sulphonation, has been heated for 1 hr. at 150° and 3 atm. pressure, with or without the addition, while hot, of resin, resin soap, or ordinary soap containing fatty acids, so as to form an emulsion. The addition of 0.16 p.p.m. thereof to the contents of a boiler will remove pre-existent scale, and a little added to the feed water from time to time will then keep the boiler clean. C. JEPSON.

Brine evaporation (B.P. 309,104—6). Air for ventilation (B.P. 311,304).—See I. *cyclo*Hexyl compounds (U.S.P. 1,703,186).—See III.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 2, 1929.

I.—GENERAL; PLANT; MACHINERY.

Rapid graphical method for calculation of steam-distillation problems. E. M. BAKER and E. E. PETTIBONE (Ind. Eng. Chem., 1929, 21, 562—564).—The method described enables the temperatures of steam distillation of organic compounds at various pressures to be read graphically. If the temperatures required for steam distillations are plotted against the temperatures at which water exerts vapour pressures equal to the sum of the partial pressures of steam and the substance being distilled, a straight line is obtained. Data are given for the construction of steam-distillation lines for carbon disulphide, acetone, chloroform, carbon tetrachloride, benzene, isopropyl iodide, chlorobenzene, aniline, and for several mixtures of higher paraffins.

H. INGLESON.

A.F.N. system of lixiviation. A. FONTAINE (Caliche, 1929, 11, 1—4).—A method of lixiviation is described whereby the solvent ascends in counter-current to the material to be extracted. Advantages claimed for the method are the intimate contact achieved between the two phases, resulting in rapid lixiviation and high yield, and the economy effected in heat and in manual labour.

H. F. GILLIE.

Jellies and gels. F. KIRCHHOF (Kautschuk, 1929, 5, 100—103, 140—142).—A review of characteristics of gels, with some reference to industrial products of this type.

D. F. TWISS.

Electrical heating. CARLETON.—See XI.

PATENTS.

Heat-exchange coil. J. STRINDLUND, Assr. to G. D. JENSEN Co. (U.S.P. 1,713,456, 14.5.29. Appl., 5.5.27).—The pipe coil for the inner fluid is in the form of a vertical cylindrical vessel, the helical pipe being formed in the thickness of the wall, and between the turns of the pipe holes are formed through the walls to permit free circulation of the outer fluid which is contained in an outer casing.

B. M. VENABLES.

Apparatus for heat-treatment of fluids. W. WINSHIP (U.S.P. 1,712,372, 7.5.29. Appl., 30.3.26).—A number of vertical tubes are completely immersed in the fluid, which is in contact with both the inside and outside of the tubes. The tubes are of high-resistance material, are connected electrically in series (all or in groups), and are heated electrically.

B. M. VENABLES.

Apparatus for effecting the exchange of heat between fluids. F. BAILEY and F. H. JACKSON (B.P. 311,889, 2.4.28).—A heat exchanger of the type wherein heat-absorbing material is exposed alternately to the heating and cooling fluid is constructed of metallic ribbon

alternately flat and corrugated (or accordion-pleated), the pair being wound round a bobbin spirally, like a clock spring. There are several stories of spirals, the gases passing through edgeways, and the corrugations are preferably oblique to the ribbon, the obliquity being opposite in adjacent spirals.

B. M. VENABLES.

Cold interchanger for gas separation plant. M. FRÄNKEL (B.P. 294,944, 1.8.28. Ger., 1.8.27. Addn. to B.P. 246,172).—In a cold interchanger of the intermittent-regenerator type, in which the cold-absorbing mass comprises strips of metal, the filling is arranged in a number of zones, and at the cold end there is a greater mass of metal and smaller space for the gas than at the warmer end, progressively according to the temperature and volume of the gas. The strips are preferably corrugated, with the corrugations inclined to the general direction of flow. One method of assembling the strips comprises taking a pair of strips with corrugations transverse to the strip, but oppositely inclined, and winding the pair in a flat spiral up to the diameter of the casing, the whole forming one zone.

B. M. VENABLES.

Refrigerant lubricant and method of lubricating refrigerating machinery. R. F. MASSA, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,696,642, 25.12.28. Appl., 9.10.24).—Glycols, preferably trimethylene glycol, containing less than 2% of water are used as lubricants.

R. BRIGHTMAN.

Artificial ice. M. EULE, Assr. to A. HARTMANN (U.S.P. 1,713,596, 21.5.29. Appl., 1.9.27. Ger., 28.9.26).—Clear ice is manufactured by removing the less soluble constituents from ordinary undistilled water, adding 5—6.5 g. of anhydrous sodium carbonate per m.³ of water, and freezing the water thus treated.

W. G. CAREY.

Dryer. W. T. MORIN, Assr. to PACIFIC ABRASIVE SUPPLY Co. (U.S.P. 1,713,237, 14.5.29. Appl., 11.1.28).—A chamber is provided with fixed flues in its walls, and the material to be dried is injected into the chamber downwardly, in such a manner as to avoid contact with the walls, but to be heated therefrom by radiation only. The heating gases, after passing through the flues, have a controllable choice of two routes: (a) directly upwards through the material, or (b) through a heat exchanger in which air is heated and passed through the material.

B. M. VENABLES.

Dryer. L. J. ROBB, Assr. to HEYL & PATTERSON, Inc. (U.S.P. 1,706,708, 26.3.29. Appl., 3.7.26).—A drying chamber is provided with an inclined chute, having perforated top and bottom plates through which the drying gases pass, and with an outlet above the top plate for discharging the material.

L. A. COLES.

Dryer. D. S. BAKER (U.S.P. 1,706,993, 26.3.29. Appl., 29.6.26).—The passage for the drying medium through the apparatus is provided with reheaters situated between each pair of a series of supports for the material being dried. The extent of the reheating is controlled by regulating the proportion of the stream of drying medium brought into contact with each reheater, and without varying the quantity of heat supplied to the reheaters. L. A. COLES.

Crushing machine. L. P. WALKER (U.S.P. 1,712,369, 7.5.29. Appl., 23.7.27).—In a gyratory crusher the usual pulley and bevel gear drive a central pinion which drives a number of surrounding pinions each provided with an eccentric driving member or wide cam; the cams act in unison to gyrate the end of the crusher shaft. B. M. VENABLES.

Machine for disintegrating, grinding, or threshing. B. BIALY (B.P. 289,889, 3.5.28. Poland, 6.5.27).—The machine comprises a closed casing (which may have a smooth interior) and a high-speed rotor comprising one or more discs with projections; all or some of the discs are formed with central apertures, the drive being transmitted from the shaft across the apertures by spider arms that act as fan blades. B. M. VENABLES.

Laboratory mill. S. W. WILEY (U.S.P. 1,706,643, 26.3.29. Appl., 8.11.26).—An outer casing fitted internally with adjustable stationary knives contains an inner rotating member also fitted with knives. A removable screen slides into lateral grooves in the lower part of the casing, and is held in place by a hinged door. L. A. COLES.

Mixing machine. J. T. SIMPSON (U.S.P. 1,706,417, 26.3.29. Appl., 30.8.26).—The machine is provided with means for conveying material through it, mixing and mulling the material, and adjusting the conveyor so as to vary the length of time during which the material is subjected to the mixing and mulling operations. L. A. COLES.

Apparatus for treating solutions. H. FRISCHER (U.S.P. 1,711,638, 7.5.29. Appl., 30.11.27. Ger., 6.12.26).—An apparatus suitable for dissolving, filtering, and washing comprises a sieve- or filter-drum provided with stirring blades on its outside, rotating within a vessel. B. M. VENABLES.

Concentrating apparatus [for solutions]. G. H. PASSELECQ (U.S.P. 1,711,614, 7.5.29. Appl., 12.1.25. Fr., 14.1.24).—A number of evaporating chambers have a common vapour outlet, but the solution passes through the chambers in series, being reheated in the pipes between chambers in such a way as to produce transfer of the fluid by thermosiphon action. B. M. VENABLES.

Device for cooling liquids. L. W. HASSENSALL (U.S.P. 1,712,701, 14.5.29. Appl., 20.4.28).—Frozen gas is placed in a receptacle which is submerged in the tank of liquor to be cooled, the outlet for gas being through a Π -shaped pipe from the open or perforated end of which the gas bubbles out. B. M. VENABLES.

Plant for the production of crystals. J. R. ELLISON (B.P. 311,935, 10.5.28).—A continuous crystal-

liser in the form of a long rocking trough from one end of which the crystals are intended to be discharged, suspended in the mother-liquor without the aid of a mechanical conveying device, is provided with travelling blade members, which travel to and fro adjacent to the inner surface of the trough and serve to loosen, but not to convey, crystals adhering to the surface of the trough. B. M. VENABLES.

Operation of centrifugal separators. AKTIEBO-LAGET SEPARATOR (B.P. 296,670, 27.8.28. Swed., 3.9.27).—Solid matter which is collected on the wall of a bowl of a separator (used, say, for cleaning oil) is discharged by means of a flush of water, or other liquid such as brine, intermittently applied without stopping the bowl. The flush liquid is preferably led through special channels to the zone where the solids have collected so that it will have acquired a good tangential speed. Just before admitting the flush it is preferable to slow up the bowl, which is accelerated immediately after, thus producing eddies. By using an intermittent instead of continuous flush the amount of oil wasted in the emulsion of solids, oil, and water is much reduced. B. M. VENABLES.

Centrifugal treatment of substances. L. D. JONES, Assr. to SHARPLES SPECIALTY CO. (U.S.P. 1,711,533, 7.5.29. Appl., 25.6.27).—A centrifugal machine from which the substance or a constituent is delivered in dispersed form has that substance maintained in contact with an atmosphere saturated with the vapour of the substance, to prevent undesirable changes in the composition of the dispersion. B. M. VENABLES.

Brake for centrifugals. R. A. STEPS (U.S.P. 1,713,502, 14.5.29. Appl., 23.6.26).—A brake drum is fastened to the belt pulley with the interposition of heat-insulating material. B. M. VENABLES.

Concentrator. A. H. STEBBINS (U.S.P. 1,712,589, 14.5.29. Appl., 28.4.27).—A vertical receptacle has side walls which converge downwardly; at the bottom are a number of concentric annular collecting zones for different products. The pulp is supplied in the form of a thin annular sheet in a downward direction at a part of the receptacle where the diameter is largest. B. M. VENABLES.

Effecting reaction between liquids tending to form tight emulsions. F. H. MCBERTY, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,698,270, 8.1.29. Appl., 16.8.24).—The liquids are circulated continuously and separated after partial reaction, the separated liquids being mixed and returned to the stream, and the operation repeated until reaction is complete. *E.g.*, used motor crank-case oil and trisodium phosphate solution are circulated to and from a tank, the circuit including a heater to raise the temperature of the oil to, *e.g.*, 80°. Part or all of the stream from the heater is run through a centrifuge, and the two components are returned to the tank. Agglomerated carbon accumulates in the centrifuge, and when the remainder is taken up by the sodium phosphate solution the latter and the clear oil are separately discharged from the circuit. R. BRIGHTMAN.

Fractionating apparatus. F. P. RISDON, Assrs. to TEXAS CO. (U.S.P. 1,711,656, 7.5.29. Appl., 21.8.24).—

Bubbling trays with loose bubble caps are assembled in nests, the top tray only of each nest having means to secure the caps. The nests are supported on flanges inside the shell of the tower. B. M. VENABLES.

Condensation of vapours. W. HILDEBRANDT, ASSR. to GASOLINE CORP. (U.S.P. 1,712,825, 14.5.29. Appl., 23.3.21 Renewed 6.10.28).—The vapour passes from a distilling header, through a continuous cooling coil, to a collecting header at a lower level. From the collecting header pipes rise to a gas header, and another pipe falls to a liquor collector and settler from which lead separate draw-off pipes for lighter and heavier liquids (oil and water), the latter leaving at the bottom of the settler but being afterwards brought up to a level only slightly below the oil outlet. B. M. VENABLES.

Recovery of gases or vapours taken up by adsorbents. N. V. NORITVEREENIGING VERKOOP CENTRALE, ASSEES. of ALGEM. NORIT MAATSCHAPPIJ (B.P. 308,313, 11.1.28. Ger., 11.1.27).—The saturated adsorbent is treated with a hot or boiling liquid, *e.g.*, water, having a higher b.p. than the adsorbed substance. An aqueous solution of an organic substance may be used as the displacement liquid, the solution being one which is soluble in the adsorbed substance and also easily adsorbed itself. Or the adsorbent may be impregnated with a lyophile colloid which permits adsorption of the vapour when dry and releases the adsorbed substance when treated with hot or boiling water or other aqueous liquid. A. B. MANNING.

Air cleaner. M. L. BLAIR, ASSR. to AC SPARK PLUG CO. (U.S.P. 1,712,947, 14.9.29. Appl., 4.12.26).—The air enters an outer cylindrical chamber at one end in a whirling manner, and passes through a filter to an inner chamber constituting the outlet pipe for clean air. Tangential outlets for heavy dust are provided in the outer cylindrical wall. B. M. VENABLES.

[Oscillation damper for] manometers and the like. H. WADE. FROM L'AIR LIQUIDE, SOC. ANON. POUR L'ETUDE & L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 311,967, 21.6.28).—A damper is inserted in the pipe leading to the manometer comprising a number of elastic discs clamped together round their periphery and provided with holes which are in staggered relation to each other. The discs are arranged to bend in only one axial direction for each group, but there are preferably two groups, one on either side of an apertured rigid wall, the discs of the one group being thus constrained to bend in the opposite direction to those of the other group. B. M. VENABLES.

Oxides of sulphur for fire prevention (B.P. 312,746).—See VII. **Drying of solid matter from liquids** (B.P. 312,433).—See XVIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Explosions in coal mines and permitted explosives: historical record. (SIR) F. L. NATHAN (Fuel, 1929, 8, 256—295).—A summary is given of the results of the various investigations into the causes and the prevention of explosions in coal mines, both in Great Britain and abroad, from 1828 to the present day. The factors which have been principally studied are

(a) the influence of the presence of coal dust on the ignitibility of gaseous mixtures, (b) the composition and testing of safety explosives, (c) the length and duration of flame from an explosive as affecting the ignition of firedamp-air or coal dust-air mixtures, and (d) the influence of the dimensions of the gallery, the density of loading, the size of the detonator, etc. on the charge limit. A. B. MANNING.

Recent research on production and utilisation of coke. W. T. K. BRAUNHOLTZ (Gas World, 1929, 90, Coking Sect., 65—68).—A number of different cokes have been examined by the "Micum" trommel test (the standard German test for the mechanical strength of coke) and the shatter test (standard English and American method). It is found that if 50 lb. instead of the specified 50 kg. of coke are used in the full-scale trommel test, more breakage occurs with the smaller quantity, owing presumably to the cushioning action with the larger amount. If the trommel length is halved, 50 lb. of coke used in such an apparatus give the same result as 100 lb. of coke in the full-size trommel test; this is an advantage when the quantities of coke available are small. There is an approximate concordance between the results given by the trommel and shatter tests, but it is concluded that the trommel index of a coke cannot be calculated with accuracy from its shatter index or *vice versa*; the two tests place the cokes in the same order of merit as regards their tendencies to "fines" production, but it is shown that the trommel test is far more drastic than the shatter test. Experiments on the determination and nature of the volatile matter in coke and a comparison of the weights of different cokes occupying 1 cub. ft. are described. The sieve analysis, distribution of the ash, and the proximate analysis of a number of coals, as charged to the ovens, are also given, and it is indicated that (a) the degree of fineness of the coal charged to coke ovens shows little variation from plant to plant, (b) there is a distinct tendency for the middle sizes to contain least ash, and (c) Cumberland coking coals have higher moisture and volatile matter contents than Durham coals.

C. B. MARSON.

Absorption characteristics of coals. B. PENTEGOV and R. NJANKOVSKAJA (Mem. Univ. d'Etat Extr. Orient, 1927, No. 6, 18 pp.; Chem. Zentr., 1928, ii, 2762).—Brown and bituminous coals are differentiated by their adsorptive power towards ferric chloride, oxalic acid, and methylene-blue in aqueous solution.

A. A. ELDRIDGE.

Measurement and value of the plasticity of coal. J. A. JACKSON (Gas World, 1929, 90, 715—717).—A coal briquette, 0.5 in. long and weighing 2 g., is heated in a silica tube, and rests on a thermocouple; the rate of heating is adjusted to give a rise of 10° per min. On the coal rests a glass or silica pointer the movement of which indicates expansion of the briquette, which occurs freely as the coal becomes plastic. Results are given for a number of coals, showing m.p., plastic range, degree of expansion, and proximate analysis, and the values obtained with coals suitable for use in continuous vertical retorts are indicated.

R. H. GRIFFITH.

Nitrogen compounds in coal. K. ISHIBASHI (J. Fuel Soc. Japan, 1929, 8, 64—65).—A sub-bituminous Fushun coal treated with phenol yielded 30% of extract. The original coal contained 2.02% N, of which 20.8% was contained in the extract and 77.7% in the residue. On carbonisation of these two fractions at 1000° the former evolved 14.4% and the latter 20% of its nitrogen in the form of ammonia. From an examination of the nitrogen compounds in the extract it is concluded that they are the source of the basic compounds in the tar, whilst those in the residue form the principal source of the ammonia. A. B. MANNING.

Liquefaction of coking coal. H. NOVÁK and J. HUBÁČEK (Paliva a Topeni, 1927, 9, 145—158; Chem. Zentr., 1928, i, 1120).—A coking coal from Schneidemühl was hydrogenated at 400—500° under 200 atm. pressure. Within 2 hrs. 50% of the dry coal had been converted into oil. Tin, antimony, bismuth, etc. had no catalytic effect on the reaction. The products of the hydrogenation, which in amount were about double those obtained by ordinary distillation, differed completely from the latter in chemical composition. They were quite mobile at the ordinary temperature, and contained more than 50% of constituents boiling below 250°. The low-temperature tar contained only 4% of benzene and solar oil. The benzene and petroleum of the low-temperature tar was equal in quality to that obtained from crude oils. The higher-boiling oils were formed of gas oils and paraffinic substances, thus resembling in composition the ordinary tar from this coal. A. B. MANNING.

Desulphuration of coal. E. GRÜNERT (J. pr. Chem., 1929, [ii], 122, 1—120).—A series of investigations has been carried out on the main reactions involved in the removal of sulphur from coal during distillation.

I. *Hydrolysis of sulphur by the reaction* $3S + 2H_2O = SO_2 + 2H_2S$.—Dry sulphur dioxide and hydrogen sulphide do not react at room temperature, but in the presence of condensed water reaction takes place rapidly (Noack, Diss., Dresden, 1925). At 100° no appreciable reaction occurs between the moist gases, but sulphur is deposited on cooling. The conclusions of Lewis and Randall (A., 1918, ii, 159) are therefore erroneous. The reaction at higher temperatures (450—800°) has been investigated by passing the gases in equivalent amounts into a reaction vessel and analysing the rapidly cooled emergent gases. The reaction takes place on the walls of the vessel. At 300° equilibrium is reached very slowly. At 450° it may be attained by the use of quartz powder as a surface catalyst. At 600° a catalyst is no longer necessary. Confirmatory experiments on the action of steam on sulphur have also been made. The values for the equilibrium constant, $K = [H_2S]^4 \times [SO_2]^2 / [H_2O]^4 \times [S_2]^3$, between 450° and 600° (1.18 to 0.0062) are in good agreement, when extrapolated, with the results of Randall and von Bichowsky (A., 1918, ii, 159), but do not agree with those of Lewis and Randall (*loc. cit.*) at 450°, the difference being attributed to the neglect of the latter to take into account the dependence of the equilibria in sulphur vapour on the total pressure. The application of Nernst's approximation formula shows that the heat

of the reaction $3S_2$ (gas) + $4H_2O$ (gas) = $4H_2S$ + $2SO_2$ is not 12.7 kg.-cal., as calculated from data for other reactions, but is of the order of 32.0 kg.-cal., whilst the value calculated from the reaction isochore is 28.0 kg.-cal.

II. *Hydrolysis of sulphur in presence of amorphous carbon.*—Preliminary investigations of this reaction have been made. At 150—450° the carbon does not itself react, but catalyses the gaseous reaction. The behaviour varies somewhat with the type of carbon employed, but at 600° and above the sulphur dioxide is removed by reduction, and the formation of carbon disulphide begins at 800°. At room temperature active charcoal catalyses the reaction between dry sulphur dioxide and hydrogen sulphide.

III. *Interaction of iron pyrites, amorphous carbon, and steam.*—When steam and nitrogen are passed over pyrites, the latter is decomposed slowly at 500° and rapidly, but not completely, at 600°, the sulphur being hydrolysed to sulphur dioxide and hydrogen sulphide. The hydrolysis of the ferrous sulphide is less complete at the higher temperature owing to the coherence of the protective layer of iron oxide formed. The results are in agreement with those of Foerster and Geissler (B., 1923, 401 A). The amorphous carbon used includes sugar- and wood-charcoals, active charcoal, and acid-treated semi-cokes. When such carbons are heated in a stream of nitrogen and in a stream of nitrogen and steam at 500—600°, a comparison of the resulting gas mixtures shows that the ordinary water-gas reaction occurs. When a mixture of steam and nitrogen is passed over a mixture of amorphous carbon and pyrites containing 5—10% S at 500—600°, about half the sulphur is found as hydrogen sulphide in the resulting gas. No sulphur dioxide is obtained. The sulphur not remaining as ferrous sulphide is adsorbed by the carbon. The gas contains more carbon dioxide and less carbon monoxide and hydrogen than it does when no pyrites is present, owing either to the reduction of the sulphur dioxide or to an alteration of the catalytic effect of the solid on the water-gas reaction. When a mixture of amorphous carbon and pyrites is heated in a stream of nitrogen, although sufficient hydrogen is present to combine with the sulphur, only a small proportion of the latter is removed as hydrogen sulphide. It is adsorbed by the carbon, and is only removed by steam. It is shown that the increased formation of hydrogen sulphide in presence of steam probably results from the hydrolysis of the sulphur, since the equilibrium in this reaction is attained more rapidly than the equilibrium in the reaction between hydrogen and sulphur, which also comes into consideration. R. K. CALLOW.

Determination of sulphur in coal by means of the nephelometer. B. TYKAČ and J. STREIT (Paliva a Topeni, 1927, 9, 133—134; Chem. Zentr., 1928, i, 1123).—Muer's nephelometer has been used to determine the sulphur content of coal. The sulphur is converted into a soluble sulphate and to the solution, acidified with hydrochloric acid, excess of barium chloride is added. The flame of a candle, or preferably the filament of a carbon-filament lamp, is viewed through a cylinder, which is then gradually filled with

the turbid solution until the flame or the filament just disappears. The height of the liquid column in cm. (c) is related to the number of mg. of sulphur present by the formula $S = 0.6 + 15.3c$, the constants of which, however, must be redetermined each time the apparatus is used against solutions of known sulphate content. The apparatus gives very accurate results.

A. B. MANNING.

Oxidation of ammonia from [coke-oven] crude by-product liquors. G. A. PERLEY and W. P. WHITE (Ind. Eng. Chem., 1929, 21, 564—567).—The catalytic oxidation of the ammonia evolved by passing air through the heated liquor was completely prevented by the deposition of sulphur derived from hydrogen sulphide on the surface of the platinum gauze used. Various methods in use commercially for the removal of hydrogen sulphide from gases were employed without success. When the crude ammonia-air mixture was passed through a solution of nickel sulphate in ammonium sulphate the subsequent oxidation could be carried out satisfactorily. The following series of reactions is suggested to explain the removal of the hydrogen sulphide: $\text{NiSO}_4 + 2\text{NH}_4\text{HS} + \text{O} \rightarrow \text{S} + \text{NiS} + (\text{NH}_4)_2\text{SO}_4 + \text{H}_2\text{O}$; $\text{NiS} + 2\text{O}_2 \rightarrow \text{NiSO}_4$. The loss of ammonia in this liquid is very small.

H. INGLESON.

Problems in the determination of unsaturated hydrocarbons in gases. I. Separation by fractional distillation. H. S. DAVIS (Ind. Eng. Chem. [Anal.], 1929, 1, 61—64).—For fractional distillation of gas mixtures boiling between -162° and $+13^\circ$, a special column has been designed which consists of a low-pitch glass spiral mounted inside an evacuated and silvered glass jacket. The liquefied mixture of gases is distilled from a Dewar vessel provided with an electric heating system, and the distillate is collected in a graduated vessel which is also immersed in a Dewar flask. The apparatus has been satisfactorily tested with mixtures of benzene and toluene, alcohol and water, and olefine mixtures; for storage of the pure gases which can be prepared in this way small steel bottles are recommended.

R. H. GRIFFITH.

Corrosion and metal protection in gasworks' practice. MAAS (Gas- u. Wasserfach, 1929, 72, 573—578).—A general review of the fundamental causes of corrosion and of the particular problems presented by gasworks' plant is given. Simple precautions are suggested to avoid accumulation of water in contact with metallic surfaces, and different methods for protection of service pipes, by means of calorising, coating with bituminous substances, etc., are described.

R. H. GRIFFITH.

Effect of distillation conditions on the consistency of road tars. C. O. CONDRUP and H. M. SPIERS (Gas J., 1929, 186, 856).—The consistency of a tar is considerably altered by heating at or near its b.p.; a vertical-retort tar, for example, changed from a value of 2.8 to 10.5 sec. when boiled for 3 hrs., and to a smaller extent at lower temperatures. The quality of a distillate may consequently be altered not only by the fractions which are collected, but by the rate at which these are allowed to cool. R. H. GRIFFITH.

Motor fuels and other products from the cracking of wood tars. J. C. MORRELL and G. EGLOFF (Ind. Eng. Chem., 1929, 21, 537—542).—A wood tar derived from the thermal decomposition of Douglas fir, cracked under pressures of 75, 100, and 120 lb./in.² and at temperatures ranging from 393° to 405° , yielded 26% of motor fuel free from tar acid based on the dry wood. By recycling the gas oil fraction a maximum yield of 33% of motor fuel was obtained. The anti-knock properties of the motor fuel were better than those of benzene, and analysis showed the content of aromatic and unsaturated compounds to be 97%. Pine tar oils from the destructive distillation of light-wood on cracking yielded about 22% and 41% of oil distilling within the motor-fuel range. Depending on the products desired, the pressure-distillate oil may be distilled to yield motor fuel and gas oil, or solvent oil, turpentine substitute, and pine oil equivalent. The gum-forming tendencies were in excess of those permissible in standard motor fuels, indicating the preferential use of such distillates in the manufacture of solvent oils, turpentine substitutes, flotation oils, paint thinners, etc. The light fraction (solvent oil) is a good solvent for crude rubber. Cracking should be carried out so that the residue is not run to coke, but remains suitable for the uses to which the original pine tar is applied. A mixture of tar derived from the thermal decomposition of hickory and oak cracked at 90 lb./in.² and 382° yielded approx. 24% of oil distilling in the motor-fuel range, but containing 52% of tar acids and 6% of tar bases. Refining of the cracked distillates follows that used for the cracked distillates of low-temperature tars and shale oils.

H. S. GARLICK.

Road tar. H. MALLISON (Petroleum, 1929, 25, 399—403).—Three German road tars, viz., (a) road tar I, (b) anthracene oil tar 50/50, (c) anthracene oil tar 60/40, each of which has been found suitable for road making, have d_{15}^{15} not above 1.225; not more than 1% of water; not more than 1% of light oil (b.p. up to 170°); 12.0—24.0, 1.0—15.0, and 1.0—10.0% of middle oil (b.p. 170 — 270°); 4.0—12.0% of heavy oil (b.p. 270 — 300°); not more than 5.0, 3.0, and 3.0% (by vol.) of phenols; not more than 5.0, 3.0, and 3.0% of naphthalene; 5.0—18.0% of free carbon; viscosity (Hutchinson) 3—15, 1—15, and 20—80 sec.; and 55—65, 45—55, and 55—65% of pitch (softening point 60 — 75°). Sample (a), owing to its high content of middle oil, has a steep viscosity-temperature curve and more readily becomes mobile on heating; sample (b) dries more rapidly on the road; and sample (c) is more viscous, resembling rather the English road tar (II), the corresponding values for which are: 1.240, 1%, 1%, 10—18%, 1%, 10—18%, 6—12%, 4% (by vol.), 5%, 24%, and 20—100 sec. Naphthalene tends to render a tar more readily fluid on warming, hence the ease of penetration into the road material is greater, but owing to its volatility the naphthalene content must be limited to cut down loss by evaporation. The "gum test" or "resinification test" of Herrmann, in which the behaviour of a thin film of tar on a glass plate at 20° is noted, does not really indicate ease of resinification, but rather the tendency to thicken by reason of the

evaporation of volatile constituents. "Free carbon," usually regarded as a filler, is the portion insoluble in benzene; this is in reality a mixture of carbon and valuable asphaltic substances. The quantity of material insoluble in tar oil or, preferably, aniline-pyridine is a better indication of the free carbon content. The quantity of bitumen that can be taken into actual solution in a road tar is not greater than 10–20%. By replacing part of the pitch content of the tar by an equal quantity of bitumen, the softening point remains at 41–43°, whilst the ductility (Dow) at 10° falls from more than 100 cm. for tar or for tar with 10% of bitumen, to 70 cm. and 16 cm. for tar with 15% and 20% of bitumen, respectively. W. S. NORRIS.

New Austrian specification for [testing] natural asphalts and petroleum asphalts. ANON. (Petroleum, 1929, 25, 404–410).—If it is necessary to melt the sample (methods for taking which are described) the container should be heated in an oil-bath at 75–100° above the softening point for the shortest possible time. The density of fluid or semi-fluid asphalt is determined by Lunge's method using a sp. gr. bottle; for solid asphalt a bath is prepared in which a sample, free from air bubbles, will just float, the density of the bath being then determined. Breaking point is determined by Church's method; softening point by the ring-and-ball method; ductility by the Dow-Smith ductilometer; flash point by means of an open-crucible, horizontal-flame, Marcusson apparatus, and the penetration test is carried out by Richardson's method. The bituminous content is ascertained by extracting 5–10 g. with 300 c.c. of chloroform, allowing it to settle, and filtering the insoluble portion, which is weighed after drying at 105°. For the determination of water the method D 95–23 T of the A.S.T.M. is employed, using xylene saturated with water. Ash is determined by ignition in a porcelain crucible, and sulphur by burning a sample in a current of air, using a transparent silica tube, and leading the products of combustion into neutralised hydrogen peroxide solution; the sulphuric acid in the latter is then titrated using 0.025*N*-sodium carbonate. Oleaginous constituents are determined by the method of Marcusson and Eickmann. The sample (20 g.) is dissolved in 30 c.c. of benzene and diluted with 440 c.c. of "normal benzene." After keeping, the filtered solution is washed three times with 30 c.c. of concentrated sulphuric acid, then with (50%) aqueous-alcoholic *N*-sodium hydroxide and with water, is evaporated, and the residue is weighed. A weighed portion of the residue is distilled to a coke residue and the paraffin content of the distillate determined by Holde's method. The acid value is determined by dissolving 5 g. in 25 c.c. of benzene, cooling, adding 100 c.c. of neutral 96% alcohol, and, after settling, titrating the filtered solution by means of alcoholic 0.1*N*-alkali hydroxide. Saponification value is determined by hydrolysing for 30 min. with 25 c.c. of benzene and 25 c.c. of alcoholic *N*-potassium hydroxide, diluting with 100 c.c. of neutral alcohol, and titrating. The stability of the sample is measured by determining the loss in weight when a portion is heated for 5 hrs. at 163° in an electrically-heated Heraeus air-bath.

W. S. NORRIS.

Petroleum asphalts and resins. N. I. TSCHERNOZHUKOV (Nef. Choz., 1928, 15, 670–673).—When heated with oxygen under pressure, paraffinum³liquidum yielded oxygenated compounds (e.g., C 71.64, H 8.81, O 19.55%) free from sulphur. Neither asphaltenes nor carbenes were formed. Crude Balakhani, Grozni, and Emba oils gave asphaltenic sediments, e.g., C₁₇₂H₁₇₆O₁₄S; C₁₃₆H₁₄₂O₁₂S. Baku and Emba white oils were heated with sulphur and then oxidised; asphalt-like compounds were obtained. With sulphuric acid the sulphur-treated oils liberate sulphur dioxide. Hence asphaltenes are formed from crude oil by unknown sulphur compounds in various stages of oxidation.

CHEMICAL ABSTRACTS.

Determination of softening points of pitches and asphalts by the Kraemer-Sarnow method. D. HOLDE (Petroleum, 1929, 25, 411–412).—In such determinations attempts to obviate the danger of mercury poisoning (cf. Mallison, B., 1928, 661) by replacing the mercury by a small brass rod lead to somewhat too high and irregular values. W. S. NORRIS.

Extraction of pyridine and phenol from crude benzene. H. A. J. PIETERS and M. J. MANNENS (Chem. Weekblad, 1929, 26, 286–290).—The procedure for removal of pyridine and phenol from the crude benzene obtained by the oil-washing of gas, by preliminary washing with dilute sulphuric acid and sodium hydroxide respectively, before the ordinary sulphuric acid and alkali purification, has been examined in detail for quantities of about 15 tons of crude benzene. The preliminary working gave recoveries of over 34 kg. of pyridine and 50 kg. of phenol in each case; a considerable economy is effected in the total sulphuric acid used, but this is offset by the greater quantity of alkali required. The methods of analysis are discussed; in the determination of pyridine, titration after distillation, using dimethyl-yellow and methylene-blue together, gives accurate results. S. I. LEVY.

Sulphur compounds in pressure-cracked naphtha and cracked naphtha sludge. D. S. MCKITTERICK (Ind. Eng. Chem., 1929, 21, 585–592).—212 litres of naphtha (*d* 0.786) from the cracking of the fuel-oil fraction (D 0.953) of California Midway crude, b.p. range 30–250°, were extracted with liquid sulphur dioxide at –20° to –30°. The extract oil was submitted to a series of fractional extractions, first with aniline and then with ethylene glycol diacetate at –19° to –20°. The fractions were combined into groups depending on their sulphur content and submitted to further extraction. The final yield was 13.6 litres of oil (*d* 0.900) containing 5.5% S, which was 40% of the total sulphur in the original naphtha. The sulphur compounds in the extract oil were further concentrated by systematic fractional distillation into 2.5° cuts or less. Thiophen isomerides partially separated in the fractional distillation were purified through their mercuric chloride derivatives, and their composition was confirmed by the m.p. of this and of a second derivative. The following compounds were identified:—thiophen, 2- and 3-methyl- and -ethyl-thiophens, and 2:3- and 3:4-dimethylthiophens. An acid sludge formed in the low-temperature refining of naphtha was diluted and the separated tar

steam-distilled. The distilled oil was treated with concentrated sulphuric acid and the oil from the sludge thus obtained steam-distilled and then fractionally distilled into 2.5° cuts. The sulphur compounds in the sludge oil readily reacted with aqueous mercuric chloride solution to give thick oily precipitates which congealed to brown solids. With methyl iodide, water-soluble crystalline sulphonium compounds which sublimed without melting were obtained. Oxidation with potassium permanganate yielded sulphones, and additive compounds were formed with bromine. The evidence points to the sulphur compounds being present as cyclic sulphides, although no definite substances were identified.

H. S. GARLICK.

Determination of free sulphuric acid in light-oil sludge. F. S. BACON (Ind. Eng. Chem. [Anal.], 1929, 1, 89—92).—Five different methods have been found which are satisfactory for determining the free acid content of light-oil sludges. (A) A water extract of the sludge is treated with acid 10% barium chloride solution. (B) The sludge is weighed on to a thick mat of asbestos in a Gooch crucible, washed with chloroform until the washings are colourless, and then treated with water to dissolve sulphuric acid which still adheres to the asbestos. (C) The sludge is mixed with chloroform and poured into ice-water, which is then titrated with alkali before and after boiling. (D) The rise in temperature which occurs on diluting the sludge with water is compared with that given by sulphuric acid alone; by allowing for the sp. heat of the tar, and assuming that no thermal effect is due to hydrolysis, the necessary data are available if the percentage of tar is known. (E) Aniline sulphate is prepared from sludge in chloroform solution, separated by filtration, and analysed by titration or gravimetrically. It is found that there is close agreement in the results of all five methods, but that those from (B) are generally somewhat lower.

R. H. GRIFFITH.

Separation of naphthenic soaps. V. SHIPEROVICH and V. GURVICH (Neft. Choz., 1928, No. 12, 26—33).—Plant for the evaporation of kerosene alkali sludge, containing 10—25% of naphthenic soaps, is described.

CHEMICAL ABSTRACTS.

Distillation of bituminous limestones for the production of Italian mineral oil. A. LA PORTA (Giorn. Chim. Ind. Appl., 1929, 11, 109—118).—In the Ragusa district, adjacent to populous centres and to the railways, there exists sufficient readily accessible bituminous limestone to give, with a mean yield of 4%, 200,000 tons of oil annually for a century, this representing about one fifth of the whole Italian consumption of fuel oils. The nature of the asphaltic rocks, the extraction of the oils, and the thermal balance, the cost of plant and working, and the characters of the oil obtained are discussed.

T. H. POPE.

Standardisation of conditions for measuring the detonation characteristics of motor fuels. R. STANSFIELD and F. B. THOLE (Ind. Eng. Chem. [Anal.], 1928, 1, 98—105).—In these tests three types of engine have been used and the effects of alterations in operation have been studied with each. Engine 1 was similar to the Ricardo E-35, but of smaller capacity, and its compression ratio could be varied from 3.9 to 7.6;

it was fitted with devices for heating inlet air and lubricant, and for controlling water-jacket temperature, ignition advance, air-fuel ratio, etc. Engine 2 was a single sleeve-valve type of 3.16 c.c. capacity, and compression ratio from 5.4 to 11.9; engine 3 was a modified single-cylinder Armstrong-Whitworth "B," with compression head of special design giving ratios between 4.0 and 8.0. With engine 1 only one satisfactory procedure was developed for comparing different fuels; the number of "pinks" occurring in a given time, above a standard intensity, was counted and was found to increase very rapidly with rise of compression ratio. Curves were plotted from these observations at three compression ratios with three different ignition advances, for a standard and for a test spirit; the mean difference in the ratios giving the same result was taken in evaluating the sample. With engine 2 a series of air-fuel ratios was taken to give a knock of standard audibility with fixed ignition; the minimum compression ratio was thus determined for every spirit. With engine 3 a number of methods were available as the audibility test was suitable, and adjustment of head to give a suitable intensity of pinking was also possible. More interesting results were obtained by the use of a Midgley bouncing pin in the compression head; this depends on matching blends, and the position of maximum pinking is shown by the amount of gas collected in the bouncing-pin voltmeter. For good results it was essential to run with much heavier pinking than was necessary for audibility tests, and matching had to be done with mixtures of known composition and not by additions to the sample under observation. The errors involved in these measurements were determined by extensive tests with known blends; engine 1 gave variations of 0.03 in compression ratio, equivalent to $\pm 1\%$ of benzene in *n*-heptane, whilst engines 2 and 3 gave 0.2 and 1.5%, respectively. The influence of other variables was also determined: (1) fuel-feed changes affected the results if a fixed-jet carburettor did not give maximum pinking conditions in all cases, but if the feed was adjusted to maximum audibility, no difficulties arose; (2) the choice of ignition plugs was important when pre-ignition was likely to occur; (3) engine speed and air and water temperatures affected the results to a small extent; and (4) errors were found due to unsuitable ignition setting, but more serious difficulties arose on attempting to adjust this, and the advance was kept constant throughout. The need for a new and plentiful standard pinking fuel is emphasised, as the cost of *n*-heptane is high and supplies are limited.

R. H. GRIFFITH.

Auto-ignition temperatures. III. (a) Mixtures of pure substances. (b) Gasolines. H. J. MASSON and W. F. HAMILTON (Ind. Eng. Chem., 21, 544—549; cf. B., 1928, 42, 699).—Curves showing the relationship between composition and values of the auto-ignition temperature (A.I.T.) for mixtures of carbon tetrachloride and ethyl ether with benzene show minimum A.I.T. values each below that of pure benzene. Similar curves for the addition of acetic acid and *p*-xylene to absolute alcohol show both a minimum and a maximum. A curve for mixtures of *n*-heptane and isooctane is perfectly regular, the relationship of the A.I.T. values being nearly additive as is the case of knock rating. The addition of:

pure lead tetraethyl and of lead tetraethyl in ethylene dibromide to a reference fuel gave curves with a distinct minimum at 2 c.c., the latter having a greater effect on the A.I.T. value than the same amount of pure lead tetraethyl. The standard reference fuel gave different A.I.T. values on platinum and quartz surfaces, whereas benzene gave identical results. The A.I.T. values of a number of straight-run gasolines compared with their knock ratings gave a smooth curve approaching a minimum at approx. 510°. A similar curve is shown for a number of cracked gasolines on a platinum surface of high catalytic activity. Experiments on the effect of composition of the igniting surface on the A.I.T. value showed that gasolines which detonate readily are very sensitive to the catalytic effect of the igniting surface and *vice versa*. The effect of the addition of lead tetraethyl is to lower the A.I.T. value of benzene and alcohol towards a minimum or optimum temperature, whilst in the case of *n*-heptane it is raised. With straight-run gasolines of varying knock rating, addition of lead tetraethyl either raises or lowers the A.I.T. value towards an optimum corresponding to decreased detonation characteristics. Carbon disulphide has a similar effect.

H. S. GARLICK.

Effect of cathode rays on hydrocarbon oils and on paper. Mechanism of cable deterioration. C. S. SCHOEFFLE and L. H. CONNELL (Ind. Eng. Chem., 1929, 21, 529—537).—Three samples of vaseline, viz., (a) taken directly from the jar, (b) carefully vacuum-dried in the tubes in which it was to be tested and only exposed to air after it had cooled, and (c) as (b) except that the tubes were separated and replaced, thus exposing the oil to air, were exposed as films between concentric glass tubes to a high electrical stress. Samples (a) and (c), containing air and therefore subjected to gaseous discharge, became masses of wax after 2½ days, whilst sample (b), air-free, was unchanged after more than 2 weeks. A similar experiment was made with a petroleum oil through which a continuous stream of oxygen was bubbled. Within 24 hrs. the oil had changed colour and become rancid, but no solid wax was formed. Further experiments showed that hydrocarbon oils react readily when bombarded by cathode rays in a manner similar to that obtained by means of a silent electric discharge. Condensation of the oil takes place with the evolution of hydrogen and saturated gaseous hydrocarbons and the formation of an insoluble product resembling the wax found in electrical power-transmission cables. Tests on a large number of mineral oils of different origin showed that (1) naphthenic oils give slightly less gas than Mid-continent oils, (2) with the same degree of refinement, low-viscosity oils give more gas than those of medium viscosity, (3) oils which give the least gas are those which contain the greatest fraction of unsaturated constituents. Similar tests made with cable paper and partially purified cellulose from cotton show these react to give water, hydrogen, and carbon dioxide with smaller amounts of carbon monoxide and methane. No marked difference in results was obtained if the experiments were carried out in an atmosphere of hydrogen or nitrogen. Paper impregnated with pure mineral oil alone or containing rosin when irradiated *in vacuo* gave practically the

same amount of moisture in each case. Paper with an initial copper number of 1.13 gave values after irradiation ranging from 12.4 to 43.1, and showed distinct acid properties. Paper subjected to corona discharge gave somewhat less gas than when irradiated; the gases produced had approximately the same composition, but that from the corona-discharge test had the higher moisture content. The paper showed no marked change of structure as in the previous experiments and the copper number increased only from 2.0 to 2.3.

H. S. GARLICK.

Sulphonated oxidation products of petroleum as insecticide activators. M. T. INMAN, JUN. (Ind. Eng. Chem., 1929, 21, 542—543).—A straight gas-oil distillate of Pennsylvania crude was subjected to catalytic vapour-phase oxidation and the products, consisting of alcohols, aldehydes, ketones, and saturated and unsaturated acids, with about 25% of unchanged hydrocarbons, were condensed. This mixture was sulphonated, washed, and neutralised with sodium hydroxide. The product, containing about 40% O and 1.5% S, was a stable, clear, maroon-coloured liquid, easily emulsifiable with water. A 1% solution was toxic to various aphids, leaf hoppers, and red mites. As an activator for other poisons, 1 pt. of the oxidised gas oil products added to 200 pts. of nicotine sulphate spraying solution reduced the amount of insecticide necessary for satisfactory kills to be obtained to ½ — ¼ of that commonly employed when soap is used as a spreader.

H. S. GARLICK.

Alundum crucible in the determination of carbon in crank-case oil. P. V. MCKINNEY (Chemist-Analyst, 1929, 18, 21).

Graphical methods and steam-distillation problems. BAKER and PETTIBONE.—See I. Flotation practice at the Sullivan mill. OUGHTRED.—See X.

PATENTS.

Method of combustion. J. T. COOK, Assr. to A. F. HAFENREFFER (U.S.P. 1,698,258, 8.1.29. Appl., 16.7.26).—Oil is delivered under about 30 lb./in.² in a rotating cone of rapidly dispersing particles to meet at an angle a similarly rotating cone of air under low pressure moving with low velocity. The mixture is ignited as it leaves the mixing chamber, and the spirally-moving ignited mixture is expanded and contracted laterally to complete combustion before being released for heating purposes.

R. BRIGHTMAN.

Distillation of carbonaceous materials. F. PUENING (U.S.P. 1,698,345—6, 8.1.29. App., 11.2.22).—(A) In the low-temperature distillation of bituminous coal, iron heat-transfer bodies are raised to a surface temperature of 760° in a gas furnace and discharged with the powdered coal on to a travelling belt conveyor within a closed chamber. From the conveyor the mixture drops to a second conveyor travelling in the reverse direction and is then discharged on an inclined grate through which the distilled coal passes while the iron bodies are advanced by a rotatory discharge and returned by an elevator to the furnace. The coal may be pre-heated, but preferably at least enough iron is employed to give double the amount of heat theoretically required to raise the coal to, e.g., 540°. (B) Coal is carbonised by

heat transfer in a distillation chamber divided by a series of narrow, vertical, flat, heat-storing plates, *e.g.*, of iron, into a series of narrow, vertical retort chambers which are first heated, *e.g.*, to 760° by gas contact. The gas supply is cut off and the retort chambers are filled with coal, the carbonised material being finally discharged into a hopper at the lower end of the chambers, a vertical multiple ram assisting the discharge, and the process is repeated.

R. BRIGHTMAN.

Distillation of solid carbonaceous material. H. KOPPERS (U.S.P. 1,712,082, 7.5.29. Appl., 5.6.22. Ger., 11.8.21.).—Solid heat-storing bodies are heated and then brought into contact with the material to be distilled in a suitable chamber. Distillation is brought about mainly by the sensible heat of the solid bodies, which are made to pass with the material through the chamber and are then separated from the distilled product. They may be used for preheating the material in a second chamber before being returned to the heating furnace.

A. B. MANNING.

Low-temperature distillation of bituminous coal. R. H. CARR and C. B. WATSON, ASSRS. to PURE OIL CO. (U.S.P. 1,698,907, 15.1.29. Appl., 15.4.26).—The coal is forced upwards by conveyors into a funnel-shaped retort, and superheated steam or oil vapour at, *e.g.*, 400–425°, introduced at the top of the retort, travels downwards in countercurrent with the column of coal. The coke is removed by gravitation through an outlet at the top of the retort, and the volatile distillation products travel downwards and escape from tuyères between the coal inlet and the hot zone.

R. BRIGHTMAN.

Low-temperature carbonisation of bituminous material. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,664, 5.5.28).—The material is passed down between internally-heated heating bodies and walls of louvre pattern, each of the former consisting of a cooled skeleton frame which supports the heating surfaces. The latter are preferably constructed of material containing silicon carbide, of pearlite or electro-cast iron, or of ceramic material of good heat conductivity. A carbonising unit comprises a number of heating bodies and louvre walls, the latter being combined in pairs, each pair enclosing a tar chamber through which the volatile products of distillation pass to the condensing system. The louvres are kept in continuous vibration by means of a striking device.

A. B. MANNING.

Production of metallurgical coke. HINSELMANN KOKSOFFENBAUGES. M.B.H. (G.P. 454,404, 4.9.23).—Coals of high volatile matter content are carbonised in two stages, at 400–500° and at 850–1000°, respectively. Direct internal heating is used, gases or vapours suitably preheated in two or more heating chambers being passed through the material.

A. B. MANNING.

Distillation retorts, gas generators, and the like. L. P. WINBY (B.P. 312,394, 25.2.28).—The lining of a tubular retort is provided with a number of ports throughout its length so that the heating gases may be by-passed when the formation of a plastic layer causes a blockage.

J. A. SUGDEN.

Water-gas generator. O. B. EVANS, ASSR. to I. C. COPLEY (U.S.P. 1,699,231, 15.1.29. Appl., 13.10.24).—The generator is divided by an internal bridge of refractory material into an upper and a lower fuel chamber separated by four unobstructed vertical passages to avoid a dead or cold mass of fuel at the axis of the fuel bed.

R. BRIGHTMAN.

Manufacture of water-gas. C. DAVIES, JUN. (U.S.P. 1,712,983, 14.5.29. Appl., 18.3.22).—The operations in the manufacture are carried out in the following order: (a) the fuel bed is supplied with air which has been preheated in a heat-transfer chamber; (b) steam is passed through the latter to the fuel bed, the ash in which is maintained at a sufficiently high temperature for slagging conditions, and the hot water-gas is discharged through the first heat-transfer chamber; (c) the fuel bed is again supplied with air preheated in the last-named chamber, and the hot products are used to preheat the fuel for the generator.

A. B. MANNING.

Apparatus for manufacture of fuel gas. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,686, 29.5.28. Addn. to B.P. 214,544; B., 1924, 549).—The gas producer described in the main patent or in B.P. 279,316 (B., 1928, 79) is modified by the addition of an enlarged secondary gasification chamber where a further supply of a gasifying agent may be added. By the provision of heating devices, *e.g.*, channels of refractory material traversed by combustion gases at about 1200°, in the fuel zone the apparatus may be used for the continuous production of water-gas.

A. B. MANNING.

Production of mixed gas from tar-free illuminating gas and water-gas. J. VON JASZOVSKY (Aust. P. 108,149, 10.4.25).—Two gasification chambers forming the upper part of a generator are charged in turn with coal. They are heated externally by the combustion of part of the water-gas which is produced continuously in the lower chamber of the generator, which is also heated externally. The illuminating gas produced in one upper chamber is passed through the hot coke in the other and is then mixed with the remainder of the water-gas from the lower chamber.

A. B. MANNING.

Production of hydrogen mixed with carbon monoxide and nitrogen from coke-oven gas. COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES ETABL. KUHLMANN (B.P. 286,291, 23.1.28. Fr., 3.3.27).—The preheated gas, with or without the addition of air or steam, is passed into a decomposition chamber packed with a filling, *e.g.*, coke, of high heat capacity and large surface. The temperature of the chamber is at least 1200° and the gas remains therein for about 1 sec. Regenerators are arranged on both sides of the reaction chamber, whilst between the latter and each regenerator, and in direct contact with these, are combustion chambers into which open heating gas and secondary air conduits. After passing the gas through the apparatus in one direction a current of air is introduced in order to burn any deposited carbon, after which the gas is passed through in the reverse direction. Before and after the passage of the air the apparatus is swept out with an inert gas.

A. B. MANNING.

Production of hydrogen from steam and carbon.

SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES, Assees. of DEUTS. BERGIN-A.-G. F. KOHLE- U. ERDÖLCHEMIE (F.P. 630,330, 5.3.27. Ger., 3.12.26).—Organic materials are carbonised at low temperatures and the coke residue is treated with steam at 500—800°. A. B. MANNING.

Oil-gas apparatus. A. J. BASSETT, Assr. to BRUCE MACBETH ENGINE Co. (U.S.P. 1,698,525, 8.1.29. Appl., 12.11.25).—The apparatus comprises a converter for converting oil into gas, an air pump, an oil supply, conduits for conveying air and oil to a mixer, and means for delivering the mixture into the converter. A branch air conduit connects the main air conduit to the oil supply. Means are provided for automatically regulating the pressure in the main air conduit in accordance with the gas pressure in the oil-gas delivery pipe. A. B. MANNING.

Manufacture of oil-gas. A. J. BASSETT, Assr. to BRUCE MACBETH ENGINE Co. (U.S.P. 1,698,526, 8.1.29. Appl., 12.11.25).—The supplies of air and oil to the converter are regulated by a valve in communication with the main gas-supply pipe, which operates to reduce the air-supply pressure, and hence the production of gas, as the demand for gas diminishes, or to increase it as the demand increases. The regulator operates by diverting air continuously in amounts inversely proportional to demands for gas, from the air-supply pipes to a pressure-developing pipe. R. BRIGHTMAN.

[Bituminous] waterproof composition. F. W. GOUGH (B.P. 312,467, 4.5.28).—A bituminous emulsion containing a small amount of an aromatic sulphonic or hydrogenated phenolic product is run into a hot argillaceous suspension containing a de-emulsifying agent (e.g., acetic or propionic acid, aluminium or ferric chloride, etc.). The mixture thickens to a non-sticky plastic mass which can be thinned with cold water and does not set until allowed to air-dry.

J. A. SUGDEN.

Manufacture of asphalt emulsions. A. L. HALVORSEN and P. M. TRAVIS, Assrs. to EMULSION PROCESS CORP. (U.S.P. 1,714,982, 28.5.29. Appl., 3.3.28).—Emulsions of asphalt with water are improved by adding to them 0.75% by weight, or less, of sodium phosphate.

H. ROYAL-DAWSON.

Production of oxides of nitrogen from ammoniacal liquor. D. L. JACOBSON, Assrs. to KOPPERS Co. (U.S.P. 1,713,045, 14.5.29. Appl., 22.8.27).—A gas containing oxygen, nitrogen, and ammonia is produced by blowing air through the liquor and, after the oxidation of any hydrogen sulphide present, is passed over a catalyst which effects the combination of oxygen and nitrogen. A. B. MANNING.

Conversion of hydrocarbons of high b.p. into those of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,789, 14.12.27).—The initial hydrocarbons are heated, preferably at 400—500°, under pressures of 20—50 atm., or if necessary under higher pressures, with catalysts of the type of the "ansolvo-acids" (cf. Meerweiu, A., 1927, 836). The components of the "ansolvo" acid, e.g., α -naphthoic acid and tin tetrachloride, α -naphthylcarboxylic acid

and aluminium chloride, etc., may be added separately to the material under treatment. The process may be carried out in an inert atmosphere, or in the presence of hydrogen, or of gases containing or producing hydrogen. A. B. MANNING.

Production of valuable resin-like hydrocarbons.

ZECHE DE WENDEL, Assees. of E. KUGEL and H. SCHWENKE (G.P. 454,307, 25.7.22).—The wash acid from the purification of benzol is neutralised with ammonia, and as the resinous substances separate they are diluted with benzol or solvent naphtha, the liquid at the same time being stirred and cooled. After separation from the ammonium sulphate, the solvent is removed from the resins by evaporation. A. B. MANNING.

Refining of petroleum, and of oils derived from coal. F. HOFMANN and W. STEGEMANN (B.P. 292,932—3, 25.6.28. Ger., 25.6.27).—(A) Petroleum fractions are treated with 3—4% of hydrogen fluoride; metal halides, e.g., boron fluoride, are added if desired. For thick oils the treatment is advantageously carried out at higher temperatures under suitable pressure, or the oil may be dissolved in a lighter oil. (B) Oils derived from the distillation of coal are similarly treated.

A. B. MANNING.

Refining of hydrocarbons. I. G. FARBENIND. A.-G., Assees. of W. PUNGS and E. GALLE (G.P. 453,883, 26.2.24).—Water-white, pleasant-smelling oils are produced by subjecting tars, low-temperature tars, or their fractions, containing phenols, sulphur, and unsaturated compounds, to the process described in B.P. 249,309 (B., 1926, 432). Although the oils still contain 0.1—0.2% S when they are passed over the catalysts, the latter retain their activity for long periods.

A. B. MANNING.

Refining of hydrocarbon oils. H. J. BRODERSON, Assr. to STANDARD OIL Co. (U.S.P. 1,698,428, 8.1.29. Appl., 7.6.24).—Hydrocarbon oil distillate heated to 26—38° is mixed with sodium plumbite solution etc. and delivered continuously through a perforated pipe to the conical bottom of the agitator below the surface of a further quantity of reagent. The oil rises through the solution and flows out continually at the top of the agitator, reagent being similarly withdrawn at the bottom and pumped to the mixer. R. BRIGHTMAN.

Treating hydrocarbon oil residues. F. M. ROGERS, Assr. to STANDARD OIL Co. (U.S.P. 1,698,452, 8.1.29. Appl., 28.8.22).—Asphalt distillation residues from crude oil containing asphalt are mixed with, e.g., 70% of "miners' oil" distillate, and treated with sulphuric acid of d 1.84. The sour oil is separated from sludge, washed, neutralised, and the diluent is distilled. E.g., asphalt residuum, m.p. 43°, penetration 200 at 250°, from Mid-continent crude, gave a residual oil, d 0.9345, flash point 268°, viscosity 320 sec. (Saybolt) at 99°.

R. BRIGHTMAN.

Refining of crude fuel oil. E. A. SPERRY, Assr. to SPERRY DEVELOPMENT Co. (U.S.P. 1,699,379, 15.1.29. Appl., 21.9.25).—The oil is centrifuged under pressure to prevent frothing, being heated either in the centrifuge or by previous passage through a superheater. In the latter case the centrifuge coil may be a cooling system.

R. BRIGHTMAN.

Refining of cracked benzenes. H. SUIDA and H. PÖLL (Austr. P. 108,165, 9.10.26).—The crude benzenes are treated with dry zinc dust and dry hydrochloric acid and are then distilled over zinc dust. A. B. MANNING.

Manufacture of motor fuels. H. D. ELKINGTON. From G. A. CRAMER (B.P. 312,245, 17.1.28).—One or more substances (*e.g.*, potassium oxalate or citrate) capable of preventing or diminishing knocking in the cylinders of an internal-combustion engine are dissolved in a liquid solvent insoluble in the motor fuel, *e.g.*, water, and the solution is dispersed in the motor fuel, with or without the use of an emulsifying agent and/or a stabiliser. H. S. GARLICK.

Reclaiming used lubricating oil. G. L. CHERRY, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,698,257, 8.1.29. Appl., 5.5.26).—Used oil, *e.g.*, car-axle oil, is heated to about 200° and treated with 1–3% of dry sodium hydroxide. After settling hot for 10–14 hrs., the oil is separated from the intermediate water layer and the sludge, washed with hot water, and centrifuged. R. BRIGHTMAN.

Separation of oils from mixtures of the same with solid substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,383, 22.2.28).—Residues obtained from the destructive hydrogenation of coal, tars, mineral oils, etc., or other mixtures of oils with solid materials, are treated at an elevated temperature with an organic solvent in which the oil is easily soluble at high temperatures, but difficultly soluble at low temperatures. After freeing the solution from undissolved residue, the solvent and oil are separated by mechanical means, *e.g.*, centrifuging, at a temperature preferably in the neighbourhood of the settling point of the oil. H. S. GARLICK.

Centrifugal separators (B.P. 296,670). **Reaction between emulsified liquids** (U.S.P. 1,698,270).—See I. **Liquid hydrocarbons** (B.P. 311,899).—See III. **Black colouring matter from peat** (B.P. 307,861).—See IV. **Ammonium sulphate** (B.P. 308,243 and 309,852). **Catalyst** (U.S.P. 1,698,009).—See VII. **Chemical reaction in hydrocarbon gases produced electrically** (B.P. 311,352).—See XI. **Soap** (B.P. 312,405).—See XII. **Printer's ink** (B.P. 312,745).—See XIII. **Leather oil** (U.S.P. 1,715,892).—See XV.

III.—ORGANIC INTERMEDIATES.

Graphical methods and steam-distillation problems. BAKER and PETTIBONE.—See I. **Cymene from cellulose.** BÖDTKER.—See V. **Preparation of alcoholic potash solution, and test for aldehyde in chloroform.** VALJASCHKO.—See XX.

PATENTS.

Regeneration of [mercury] catalysts. H. S. HIRST, S. W. ROWELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,888, 17.1.28).—The mercury (and iron) sludge from the manufacture of acetaldehyde from acetylene is dissolved in concentrated nitric acid, and sulphuric acid is added up to a concentration of 55% H_2SO_4 (*d* 1.45). All the mercury and iron are thus precipitated as sulphate, which is separated, redissolved

in 15–20% sulphuric acid, and returned to the process. C. HOLLINS.

Manufacture of liquid hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 311,899, 10.4.28. Addn. to B.P. 258,608; B., 1928, 255).—Gas mixtures containing methane and/or ethane, unsaturated hydrocarbons, especially olefines, or the higher homologues of the paraffin series, and hydrogen, in which the ratio of carbon combined with hydrogen to total hydrogen is greater than the C:H ratio in methane, are passed at 500–900° and under the ordinary pressure over the catalysts described in the main patent. High yields of benzene hydrocarbons are produced. The process may advantageously be applied to the gases resulting from the low-temperature carbonisation of carbonaceous materials. A. B. MANNING.

Concentration of volatile aliphatic acids [acetic acid]. HOLZVERKOHLUNGS-IND. A.-G. (B.P. 291,433—4, 26.4.28. Ger., 2.6.27, and Addn. B.P. 302,268—9, 27.4.28. Ger., 13.12.27).—In the manufacture of concentrated acetic acid by way of potassium hydrogen acetate, (A and C) the vapour of dilute acetic acid, or (B and D) a benzene or acetone oil extract of the dilute acid, is brought in contact with potassium acetate, preferably on the countercurrent system, using a concentrated solution of the salt. The acid salt formed (A and B) may be isolated, or (C and D) may be decomposed without isolation, the optimum temperature being about 170–220°, preferably at reduced pressure. C. HOLLINS.

Production of butadiene hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,945, 10.9.27).—In the pyrogenic production of butadienes from chain or cyclic paraffins or olefines of artificial or mineral origin, using silica, silicates, noble metals, or the catalysts of B.P. 297,398 (B., 1928, 884), improved yields result when the starting materials are diluted with at least half their volume of added steam. [Stat. ref.] C. HOLLINS.

Purification of β -phenylethyl alcohol. E. C. BRITTON, Assr. to DOW CHEM. Co. (U.S.P. 1,698,932, 15.1.29. Appl., 12.2.24).—Crude β -phenylethyl alcohol after partial elimination of alcoholic impurities by fractional distillation is esterified, *e.g.*, in toluene, with 1.1 mols. of phthalic anhydride or other dibasic organic acid; the β -phenylethyl hydrogen phthalate is distilled in steam and hydrolysed. R. BRIGHTMAN.

Preparation of tetrazoles. A. BOEHRINGER (B.P. 285,080, 3.2.28. Ger., 11.2.27. Addn. to B.P. 280,529; B., 1929, 350).—Azoimide or sodium azide reacts with esters (especially sulphonic esters) of amidoximes, $R \cdot C(NH_2)NOH$, or their Beckmann transformation products, or in presence of thionyl chloride, phosphorus pentachloride, phosphoryl chloride, etc., with amidoximes themselves, to give *C*-aminotetrazoles. Phenylacetamidoxime *O*-benzenesulphonate yields with sodium azide in boiling alcohol 5-amino-1-benzyl-1:2:3:4-tetrazole, m.p. 191°. The 5-amino-1-phenyl compound, m.p. 159°, is similarly obtained from benzamidoxime *O*-benzenesulphonate. C. HOLLINS.

Manufacture of higher alkylated guanidine derivatives. SCHERING-KAHLBAUM A.-G. (B.P. 285,873,

15.2.28. Ger., 24.2.27. Addn. to B.P. 279,884; B., 1929, 163).—In the process of the prior patent the amount of solvent used may be considerably less than that required for dissolution. C. HOLLINS.

Manufacture of materials from or comprising [wax-like] chlorinated hydrocarbons. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,421, 4.1.28).—Wax-like chlorinated naphthalenes, toluenes, etc. are mixed with 5–20% of a fat, wax, resin, pitch, or rubber, and/or alkaline substances such as sodium phosphate, lime, magnesia, pyridine, tetrahydroquinaldine, urea, etc. The irritant action on the skin is prevented. C. HOLLINS.

Manufacture of arylaminonaphthalene derivatives. I. G. FARBENIND. A.-G. (B.P. 282,111, 13.12.27. Ger., 13.12.26).—The Bucherer reaction is applied to the condensation of phenylenediamines, aminophenols, leucoindophenols, leucoindamines, etc. with naphthylamines, naphthols, aminonaphthols (other than 1:8), and polyamino- or polyhydroxy-naphthalenes, to give products useful as fur bases and for dyeing acetate silk. Examples are: *o*-aminophenyl- β -naphthylamine, m.p. 96–97°; *m*- and *p*-aminophenyl- β -naphthylamines; *p*-aminophenyl- α -naphthylamine; *p*-hydroxyphenyl- α -naphthylamine; 4-amino-4'- β -naphthylaminodiphenylamine-2(or -2')-sulphonic acid; *m*- and *p*-hydroxyphenyl- β -naphthylamines; 2:7-di-(*p*-hydroxyphenylamino)naphthalene, m.p. 249–251°; and the 1:5-isomeride, m.p. 278–280°. C. HOLLINS.

Manufacture of 2-aminonaphthalene-3-carboxylic [2-amino-3-naphthoic] acid and of intermediate compounds. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 309,516, 6.1.28. Addn. to B.P. 282,450; B., 1929, 467).—The intermediate iron compound of the prior patent is obtained by heating 2:3-hydroxy-naphthoic acid with iron turnings, ammonium chloride, and aqueous ammonia at 180–190° in a stone-lined autoclave; the pressure generated is 40–45 atm. C. HOLLINS.

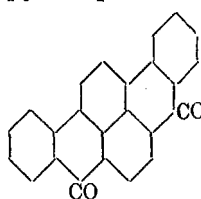
Production and use of cleaning, emulsifying, and wetting agents. ORANIENBURGER CHEM. FABR. A.-G., Assees. of CHEM. FABR. MILCH A.-G. (B.P. 283,864, 17.1.28. Ger., 17.1.27).—In the process of B.P. 275,267 (B., 1929, 292) low-molecular, in place of high-molecular, fatty acids may be used. Examples are: anthracene and propionic acid, or diisopropyl-naphthalenesulphonic acid and acetic acid, treated with chlorosulphonic acid. C. HOLLINS.

Production of wetting, cleansing, or emulsifying agents and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 309,842, 15.10.27).—Sulphonic acids possessing wetting-out properties are converted into their "triethanolamine" (tri- β -hydroxyethylamine) salts. C. HOLLINS.

Manufacture of anthraquinone derivatives. BRIT. DYESTUFFS CORP., LTD., and A. SHEPHERDSON (B.P. 308,049, 15.2.28. Addn. to B.P. 271,602; B., 1927, 550).—Potassium acetate and water may replace the sodium acetate crystals in the process of the prior patent. C. HOLLINS.

Manufacture of condensation products of the pyrenequinone series. I. G. FARBENIND. A.-G. (B.P. 287,050, 6.3.28. Ger., 12.3.27).

—4-Aroylbenzanthrone is cyclised by means of sodium aluminium chloride etc. to the corresponding dibenzpyrenequinones. 4-Benzoylbenzanthrone, m.p. 176°, obtained from benzanthrone-4-carboxylic chloride and benzene, gives 3:4:9:10-dibenzpyrene-5:8-quinone (annexed formula).



C. HOLLINS.

Manufacture of cyclic ketones, polycyclic ketones, and quinones. I. G. FARBENIND. A.-G. (B.P. 293,768, 11.7.28. Ger., 11.7.27).—Aromatic ketones and polyketones in general containing free *ortho*- and *peri*-positions suitably placed are cyclised by heating with sodium aluminium chloride at 110–120° in a current of air or oxygen. Examples are: 4-chlorobenzanthrone, m.p. 200°, from phenyl 4-chloro- α -naphthyl ketone, m.p. 80°; 4-cyanobenzanthrone, m.p. 234°; benzanthrone-4-carboxylic acid, m.p. 307°, from 4-benzoyl- α -naphthoic acid; 3:4:9:10-dibenzpyrene-5:8-quinone from 1:4-dibenzoylnaphthalene; 3:4:8:9-dibenzpyrene-5:10-quinone from 1:5-dibenzoylnaphthalene or 3-benzoylbenzanthrone; benzanthrone-3:4-dicarboxylic anhydride, m.p. 350°, from 4-benzoyl-1:8-naphthalic anhydride; product from 3- α -naphthoylbenzanthrone. C. HOLLINS.

Manufacture of arylaminoanthraquinones. K. ZAHN and P. OCHWAT, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,699,419, 15.1.29. Appl., 23.11.25. Ger., 12.12.24).—See B.P. 244,450; B., 1926, 866.

Vat dye intermediates (B.P. 307,950).—See IV. **Catalyst** (U.S.P. 1,698,009).—See VII. **Flotation agents** (B.P. 310,186).—See X.

IV.—DYESTUFFS.

Coupling of H-acid as a function of the acidity and in presence of strong electrolytes. G. R. LEVI and G. DELPONTE (Giorn. Chim. Ind. Appl., 1929, 11, 103–106).—The normal 85% yield of disazo derivative furnished by H-acid may be increased to 94–95% by replacing the neutral solution of H-acid by the freshly precipitated acid of distinctly acid reaction. The addition of the acid in solution, although it slightly accelerates the reaction, cannot be regarded as good practice as far as the yield or quality of the product is concerned. The yields of disazo compound are, in general, a function of the acid added, but phosphoric acid is an exception to this rule, as it behaves in the reaction as a weaker acid than its dissociation constant would indicate. The coupling process is retarded considerably by the presence of strong electrolytes, particularly alkali chlorides, perchlorates, and sulphates. T. H. POPE.

Coal-tar colours in foods. NICHOLLS.—See XIX.

PATENTS.

Manufacture of black colouring matter [from peat]. GOSUDARSTVENNAYA TORGOVAYA IMPORTNO-EKSPORTNAYA KONTORA GOSTORG, and S. KISLITZIN (B.P. 307,861, 15.12.27).—Raw peat mixed with solutions of

salt and alum is air-dried, distilled with tar, and the residuum acidified and boiled to precipitate a black colouring matter.

C. HOLLINS.

Manufacture of vat dyes of the 2-thionaphthen-3-indoleindigo series. I. G. FARBENIND. A.-G. (B.P. 285,389, 14.2.29. Ger., 14.2.27).—6-Aminothioindoxyl is condensed with a 5:7-dihalogenoisatin, and chlorinated in the 5:7-positions in the thionaphthen nucleus by treatment with sulphuryl chloride. Alternatively, a 5:7-dichloro-6-aminothioindoxyl is condensed with a 5:7-dihalogenoisatin. 5:7-Dichloroisatin gives a brown vat dye yielding rather redder shades than the dye from 5:7-dibromoisatin.

C. HOLLINS.

Manufacture of vat dyes of the anthracene series. I. G. FARBENIND. A.-G. (B.P. 296,758, 4.9.28. Ger., 7.9.27).—The vat dyes, probably carbazoles, obtainable by intramolecular condensation of 1:5- or 1:8-di-(α -anthraquinonylamino)anthraquinones ("trianthrimes") by the processes of B.P. 15,846 of 1909 (B., 1910, 939), B.P. 12,921 of 1910 (B., 1911, 531), G.P. 251,350 and 249,000, are treated with concentrated sulphuric acid below 5°, poured on ice, washed free from acid, and finally oxidised at 70–80° with hypochlorite, to give brighter vat dyes.

C. HOLLINS.

Manufacture of [vat] dyes and intermediates [of the anthraquinone series]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 307,950, 17.10.27).—3-Chloro-2-aminoanthraquinone is heated with antimony pentachloride in a nitro-solvent, or in a non-nitro-solvent with addition of nitrogen oxides, to give dichloro-flavanthrone, an orange vat dye.

C. HOLLINS.

Green dye of the anthraquinone series. J. OGILVIE, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,698,821, 15.1.29. Appl., 6.11.25).—1:4-Diarylaminoanthraquinones are sulphonated with 10–30% oleum in presence of boric acid below 50°. 1:4-Di-*p*-toluidinoanthraquinone with 10 pts. of 20–26% oleum at 25–50° in presence of 0.5 pt. of boric acid yields a bluish-green wool dye giving yellower shades than those obtained by sulphonation in absence of boric acid.

R. BRIGHTMAN.

Manufacture of products [acid dyes] of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 282,452, 19.12.27. Addn. to B.P. 276,408; B., 1927, 809).—Other alicyclic amines are used in place of the hydroaromatic amines of the prior patent. 4-Bromo-1-aminoanthraquinone-2-sulphonic acid is condensed with cyclopentylamine or bornylamine to give a clear blue acid dye.

C. HOLLINS.

Manufacture of vat dyes of the pyrenequinone series. I. G. FARBENIND. A.-G. (B.P. 287,845, 7.3.28. Ger., 26.3.27. Addn. to B.P. 287,050; B., 1929, 590).—The pyrenequinones of the prior patent are chlorinated or brominated.

C. HOLLINS.

Manufacture of new azo dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 309,879, 16.12.27).—2:3-Hydroxynaphthoic 3-alkoxy- β -naphthylamides have good affinity for cotton, and the padded fibre may therefore be rinsed so as to yield ice colours fast to rubbing. These arylamides may be kept in concentrated stock solutions for dyeing in a standing bath;

no addition of formaldehyde is necessary. The 3-methoxy- β -naphthylamide, m.p. 197–198°, is coupled on the fibre or in substance with diazotised 4-nitro-*o*-anisidine (bluish-red), 5-chloro-*o*-toluidine (bluish-red), *m*-4-xylydine (red), *p*-chloroaniline (scarlet), 5-chloro-2-aminophenyl β -naphthyl ether (red), 3-chloro-4-aminobenzanilide (yellow-red), 2:5-dichloroaniline (yellow-red), 2:5:5'-trichloro-2'-aminodiphenyl ether (red), 2:3-dichloroaniline (red-orange), 2:3:4-trichloroaniline (yellow-red), *m*-nitro-*p*-toluidine (claret), 2-chloro-5-nitroaniline (yellow-red), 5-chloro-*o*-nitroaniline (yellow-red), 4-nitro-5-amino-*o*-tolyl methyl ether (blue-garnet), *o*-aminoazotoluene (garnet), 4-chloro-*o*-anisidine \rightarrow 4-chloro-*o*-anisidine (dark violet), 1:5-diaminoanthraquinone (violet). The 3-ethoxy- β -naphthylamide gives similar shades.

C. HOLLINS.

Manufacture of [water-soluble] azo dyes [for lakes and wool dyeing]. I. G. FARBENIND. A.-G. (B.P. 283,897, 17.1.28. Ger., 19.1.27).—*o*-Nitroarylamines are coupled with a sulphonated acetoacetic arylamide, or the corresponding non-sulphonated dyes are sulphonated with 100% sulphuric acid or chloro-sulphonic acid. Sulphonation of *m*-nitro-*p*-toluidine \rightarrow acetoacetic *o*-chloroanilide for a greenish-yellow and of *m*-nitro-*p*-toluidine \rightarrow acetoacetic *o*-anisidine for a reddish-yellow is described.

C. HOLLINS.

Azo dyes and cellulose derivatives [acetate silk] dyed therewith. W. HENTRICH, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,695,654, 18.12.28. Appl., 4.2.27. Ger., 26.2.26).—Acetate silk is dyed with water-soluble monoazo dyes obtained by coupling a diazotised dinitro-*o*-aminophenol with aminonaphthols containing no sulphonic or carboxylic groups. Examples are: 4:6-dinitro-*o*-aminophenol with 1:8-, 1:6-, 2:8-, or 1:5-aminonaphthol (black to violet-brown, giving blacks and deep browns by diazotisation and development on the fibre); partially reduced 2:4:6-trinitro-*m*-cresol gives similar dyes. 4:6-Dinitro-*o*-aminophenol \rightarrow 1:8-aminonaphthol is especially claimed.

C. HOLLINS.

Vat dye derived from 3-halogenopyrazolanthrone. R. BERLINER, B. STEIN, and W. TRAUTNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,695,631, 18.12.28. Appl., 4.2.27. Ger., 3.3.26).—See B.P. 285,555; B., 1928, 362.

Azo dyes and their manufacture. H. WAGNER, H. EICHWEDE, and E. FISCHER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,694,568, 11.12.28. Appl., 22.12.25. Ger., 6.8.23).—See B.P. 256,808; B., 1926, 866.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemical examination of tissue of corn [maize] stalk. C. J. PETERSON and R. M. HIXON (Ind. Eng. Chem. [Anal.], 1929, 1, 65–67).—Maize stalks separated and cleaned by hand from bales have been examined by the general methods of Schwalbe (B., 1919, 407 A, 408 A). The experimental conditions for determination of lignin, cellulose, and pentosan are precisely described; results showed 34.3, 35.4, and 27.6%, respectively. Treatment of the stalks with 5% alkali, or with acid, and subsequent analysis of the products has been applied

to identification of the hemicellulose, and the results of both methods are shown to be of the same order, although not in exact agreement. The stalks were also separated into the outer shell, pith, and inner vascular bundles, but no large differences were found in the composition of these constituents, although variations in the physical properties of the cellulose were clearly detected.

R. H. GRIFFITH.

Determination of silk and cotton in asbestos yarns. H. SOMMER (Gummi-Ztg., 1929, 43, 2103—2104).—The asbestos content is determined by removal of silk and cotton with cuprammonium solution (cf. B., 1929, 277); the content of silk is found by difference after removal with a solution of copper sulphate (10 g.), glycerol (5 g.), and potassium hydroxide (in sufficient quantity to redissolve the precipitated hydroxide) in water (100 c.c.) or by boiling with *N*-sodium hydroxide. The residue from the latter determination is then used for determining the remaining cotton by cuprammonium solution.

D. F. TWISS.

Manufacture of sulphite-cellulose from pinewood. O. ROUTALA and J. SEVÓN (Cellulosechem., 1929, 10, 97—107).—Decomposition of pine wood with the usual calcium bisulphite solutions is only possible in exceptional cases when the composition of the wood is uniform, and its rosin content not too high. By complete or partial replacement of the calcium by magnesium or alkali metals better decomposition is obtained; cooking acids containing both magnesium and sodium as bases are specially satisfactory provided that the above limitations to the composition of the wood obtain. The chief cause of trouble is the difficulty of saturating the wood with liquor due to the rapid consumption of sulphurous acid, whereby difficultly soluble normal calcium sulphite is precipitated in the pores of the wood. This separation of calcium sulphite before complete saturation of the wood may be avoided by prolonging the time taken to reach the reaction temperature or by the addition of weak acids such as acetic or phosphoric acid. The addition of the alkali salts of the chosen weak acid considerably shortens the time of cooking, and their buffering action satisfactorily regulates the p_H of the liquid during cooking. This protective action results in better yield and quality of the cellulose, and a complete and uniform decomposition of non-uniform pine wood of high rosin content may be obtained in a somewhat longer time than is required for spruce wood. The addition of these salts causes better dissolution of the rosin, but it is not possible to reduce the content considerably; the remedy for this is to be found either in pretreatment of the wood or after-treatment of the cellulose. Up to 80% may be removed by extraction with small quantities of sodium hydroxide, and the addition of 0.5—1% of sodium hydroxide on the weight of cellulose in the separator or hollander should be the simplest way of removing the rosin. Decomposition of pine wood is possible by means of nitric acid, and good cellulose is obtained with no damage to the fibre, but the high consumption of chemicals and the production of hydrocyanic acid are drawbacks.

B. P. RIDGE.

Changes in moist sulphite-cellulose on storing.

E. HOCHBERGER (Papier-Fabr., 1929, 27, 282—284).—A method is described by which the isoelectric point of pulp may be determined. The isoelectric point of machine-dried, bleached pulp is changed slightly to the alkaline side by storage of the material for two months, but similar storage of moist pulp, bleached or unbleached, results in a marked change to the acid side. Moist pulp stored for this time absorbs considerably more 18% sodium hydroxide solution per unit weight under standard conditions of treatment than does fresh material in the air-dry state, and also develops greater acidity, as shown by determinations of its p_H by use of suitable indicators.

B. P. RIDGE.

Dependence of the bleaching number [of pulp] on the course of the cooking in the Ritter-Kellner process. H. NERAD (Papier-Fabr., 1929, 27, 277—282).—The chemical reactions involved in the cooking process and the effect of such factors as p_H value, temperature, time to reach the effective cooking temperature, etc. on the course of the process are discussed. A formula is deduced by means of which the bleaching capacity of a pulp may be derived mathematically from factors such as the percentage sulphur dioxide content of (1) the liquor at 100°, (2) the waste liquor, (3) the waste gases, etc., which depend only on the course of the cooking process.

B. P. RIDGE.

Cymene, a by-product in the manufacture of cellulose by the bisulphite process. E. BÖDTKER (J. Pharm. Chim., 1929, [viii], 9, 417—434).—The analysis, composition, and purification of crude cymene obtained in the bisulphite process for the manufacture of cellulose from wood is described. The chief impurities are terpenes, resins, fatty acids, and furfuraldehyde, and methods for their removal are discussed. Pure cymene is not oxidised on exposure to air; it has b.p. 177.1—177.2°/760 mm., d_4^{20} 0.8606, n_D^{20} 1.4920, and R_M 45.38 (calc. 44.92).

E. H. SHARPLES.

Effect of chemical agents, especially of oxidising influence, on the behaviour of cellulose. P. WAENTIG (Cellulosechem., 1929, 10, 81—85).—Purified cellulose is characterised by a fibrous structure, which is distinguished by a regular arrangement of anisotropic micelles or crystallites, and which possesses comparatively great resistance to swelling. Swellable regenerated cellulose is characterised by disorientation, possibly also by amorphorisation, of the micelle (which explains its increased swelling capacity), and alkali-soluble (chemically modified) cellulose corresponds with a reduction in size of the crystallite or micelle through intramolecular decomposition. In the formation of hydrocellulose and oxycellulose not only disorientation, or amorphorisation, of the crystallite occurs, but also intramolecular or intramolecular decomposition. The cellulose fibre is partly physically and partly chemically decomposed, but to a greater extent physically than chemically. The decreased viscosity of viscose solutions during ripening and the increased alkali-solubility of the regenerated viscose are explained in this way, since ripening is accompanied by absorption of oxygen from the air—hence by oxidation. The accelerated ripening of viscose in the presence of metals is due to the fact that the latter act as oxygen carriers. That sodium

hydroxide has no such effect in reducing the size of the cellulose micelles is shown by the effects of mercerisation in not reducing the strength or the α -cellulose content of the material. Cuprammonium acts in the same way as concentrated sodium hydroxide; the more rapid oxidation and fall of viscosity of the cellulose on keeping are promoted by the catalytic action of the copper. Differences between the viscosities of xanthate solutions prepared from cotton linters and cellulose wadding, respectively (which have been explained by the assumption that the former has a higher "micelle mol. wt." than the latter), depend on the previous history of the materials, since not only the oxy- or hydro-cellulose content of the material, but also the specific natural compound of the cellulose, must be considered. The effects on the viscosity of the viscose or cellulose nitrate solutions of cutting up the original material into small particles, and of valency changes in the cellulose complex due to oxidation, are discussed. B. P. RIDGE.

Reaction of cellulose with phenylhydrazine acetate. C. J. STAUD and H. LE B. GRAY (Ind. Eng. Chem. [Anal.], 1929, 1, 80—81).—The reaction described by Vignon (Compt. rend., 1899, 128, 579; cf. B., 1899, 579) has been modified by treating sulphite pulp (5 g.) with 15 c.c. of phenylhydrazine in dilute acetic acid, the whole being heated in a steam bath and then washed repeatedly with aqueous alcohol. After 16 hrs. no further increase in the nitrogen content of the residue was observed; this time was accordingly used in subsequent experiments with other materials. Attempts are made to correlate the extent of this reaction with the degree of cellulose degradation, but although a qualitative agreement appears to exist, no strict numerical comparisons can be made. R. H. GRIFFITH.

Strength testing of cellulose. G. HALL (Papier-Fabr., 1929, 27, 341—347).—American proposals for the standardisation of methods of testing the strength of cellulose are examined and criticised, and a report of such attempted standardisation in Sweden is made. The relative importance of physical tests used for paper, such as tearing strength, resistance to creasing, the bursting test, porosity, etc., in relation to degree of grinding, weight, and other properties of the material is examined, and recommended conditions under which these tests should be carried out are discussed.

B. P. RIDGE.

Effect of cathode rays on paper. SCHOEPFLE and CONNELL.—See II.

PATENTS.

Production of cellulosic material. EUROMERICAN CELLULOSE PRODUCTS CORP., Assees. of B. DORNER (B.P. 283,851, 31.12.27. U.S., 17.1.27).—After mechanical disintegration, straw etc. is leached by a slowly-moving countercurrent of water, cooked with just sufficient caustic alkali to react with the whole of the silica present, freed from silicate liquor, and further cooked in the ordinary way at a temperature below that at which the pentosans present are caramelised. The pentosans may be precipitated from the waste liquor by carbon dioxide, and the caustic alkali then regenerated from the solution. F. R. ENNOS.

Treatment of flax straw and other fibrous materials. Treatment of scutched vegetable fibres. VICKERS, LTD., and O. D. LUCAS (B.P. 309,210—1, 8.12.27).—(A) Unscutched vegetable fibres are digested first with water for 1 hr. at 182°, furfuraldehyde being distilled off, and then for 4—6 hrs. with 4—5 times their weight of a 5% caustic soda solution at 150—170°. (B) Scutched fibres are heated with a 1—5% caustic soda solution at 130—170° for 20 min. to 4 hrs. In each instance a plasticiser is added during the soda digestion, and the fibre, after treatment as above, is washed in water, immersed in acid, again washed, and dried for spinning. F. R. ENNOS.

Improving the spinning properties of cotton. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,688, 31.5.28).—Cotton is humidified by spraying it with aqueous solutions of organic sulphonic acids possessing high wetting properties; 10 litres of a 1% solution of, e.g., the sodium salt of butylnaphthalenesulphonic acid, is a suitable quantity per 100 kg. of cotton. Organic solvents and hygroscopic agents may be added if desired. D. J. NORMAN.

Obtaining artificial threads of natural silk from colloidal solutions of natural silk. T. MUTO, S. HIDA, and KANEGAFUCHI BOSEKI KABUSHIKI KWAISHA (B.P. 312,114, 18.2.28).—After partial coagulation, the thread is elongated by means of sliding friction applied by rollers around practically the whole circumference, and the coagulation is finally completed. F. R. ENNOS.

Manufacture of mothproofing media and protection of wool, skin, material, textiles, etc. against insects. I. G. FARBENIND. A.-G. (B.P. 285,825, 21.2.28. Ger., 21.2.27).—A mothproofing medium is applied as in B.P. 173,536 and 295,742 (B., 1922, 138 A; 1928, 810) in the presence of a wetting agent which is an aromatic sulphonic acid containing one or more aliphatic groups in the nucleus, e.g., *isopropyl*-naphthalenesulphonic acid. F. R. ENNOS.

Artificial silk-yarn mixtures. G. DIETRICH (B.P. 309,340, 15.6.28).—Artificial silk waste or staple fibre is spun with "African wild silk" (anaphe) to give a yarn of increased strength. F. R. ENNOS.

Manufacture of cellulose esters. I. G. FARBENIND. A.-G. (B.P. 284,298, 27.1.28. Ger., 27.1.27).—Insoluble or sparingly soluble cellulose or hydrocellulose esters of higher fatty acids are mixed in an anhydrous liquid, e.g., tetrachloroethane, toluene, etc., with an organic or inorganic acid, an acid anhydride, and the salt of a strong acid with a weak base, e.g., ferric chloride, or with a mixture of any of these, and are heated at a high temperature until the desired solubility of the ester is attained. F. R. ENNOS.

Manufacture of cellulose esters and articles made therefrom. H. DREYFUS (B.P. 308,322—3 and 309,201, [A, B] 13.9. and 15.12.27, [C] 28.9. and 15.12.27).—(A) Cellulose which has been pretreated with organic acids (cf. B.P. 263,938; B., 1927, 247) is esterified by means of a fatty acid anhydride in the presence of at least 2% each of a hydrogen halide and a zinc halide, preferably 5—10% of hydrochloric acid (calc. as HCl) and 5—10% of zinc chloride on the weight of cellulose.

in each case. (n) The process of (A) is applied to cellulose which has not been pretreated with organic acids. (c) Other halide salts, *e.g.*, iron, manganese, nickel, cobalt, or copper chloride, may replace the zinc halide in the above processes. D. J. NORMAN.

Manufacture of cellulose esters. II. DREYFUS (B.P. 311,790, 17.12.27).—Cellulose which has been pretreated with organic acids is esterified by a fatty acid anhydride in the presence of one or more halides of arsenic, antimony, or phosphorus in the presence or absence of a hydrohalide acid. Suitable quantities are 10–15% of, *e.g.*, antimony or phosphorus pentachloride, and 10–15% of hydrochloric acid on the weight of cellulose in each case. D. J. NORMAN.

Manufacture of esters of cellulose. I. G. FARBENIND. A.-G. (B.P. 285,858, 22.2.28. Ger., 23.2.27).—Cellulose esters of the higher homologues of acetic acid are prepared by warming cellulose for several hours with a halogenated acetic acid anhydride, the higher fatty acid, and a catalyst, *e.g.*, monochloroacetic acid or sulphuric acid. The ester is then precipitated by water in the usual way. F. R. ENNOS.

Preparing partially hydrolysed cellulose acetates and articles made therefrom. S. I. VLES (B.P. 310,434, 25.11.27).—In the process of B.P. 292,398 (B., 1928, 637) an acetate of much higher viscosity is obtained if the quantity of water present in the hydrolysing solution is increased to at least 50 pts. by wt. per 100 pts. of dry cellulose over and above that required for converting any excess of acetic anhydride into acetic acid. D. J. NORMAN.

Esterification of cellulosic materials. B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 310,556, 26.1.28).—Pyridine sulphuric anhydride is used as a catalyst in the esterification of cellulose. *E.g.*, 15 pts. of paper cellulose are added to a mixture containing 15 pts. of pyridine sulphuric anhydride, 20 pts. of glacial acetic acid, and 10 pts. of acetic anhydride previously heated to 100°. The reaction is allowed to proceed for about 15 min. at 100°, and, after cooling, the acetate is precipitated with water. The resulting product gives viscous solutions with acetone. By varying the conditions, products are obtained which contain different amounts of combined acetic acid and are soluble in different solvents. D. J. NORMAN.

Manufacture of cellulose aceto-esters containing higher acyl groups, and of cellulose esters containing halogen-substituted fatty acid groups. H. T. CLARKE and C. J. MALM, ASS'YS. to EASTMAN KODAK CO. (U.S.P. 1,698,048–9, 8.1.29. Appl., 18.1.28).—(A) Partly deacetylated cellulose acetate, containing, *e.g.*, 34.5–37.5% of acetyl, is esterified with anhydrides of higher fatty acids, in presence of chloroacetic anhydride, methoxyacetic anhydride, or other organic anhydride containing less than 10 carbon atoms which impels esterification. *E.g.*, cellulose acetate (5 pts.) added to a mixture of 20 pts. of stearic anhydride and 40 pts. of chloroacetic acid in presence of 0.05 pt. of magnesium perchlorate at 60–65° affords a product containing 30% of stearyl. A product containing 47.5% of stearyl is similarly obtained from 5 pts. of cellulose acetate

(36.6% acetyl), 15 pts. of stearic acid, 20 pts. of chloroacetic acid, and 20 pts. of chloroacetic anhydride. With cellulose acetate (37.5% acetyl) and 15 pts. of oleic acid the same mixture affords a product which on bromination in chloroform contains 15.7% Br. Cellulose acetate (34.5% of acetyl) affords a cellulose acetodibromostearate containing 19% Br. Similar products are obtained by esterification of a mixture of 5 pts. of cellulose acetate, 20 pts. of chloroacetic anhydride, and 40 pts. of chloroacetic acid, with 20 pts. of α -dibromostearic acid and 0.5% of magnesium perchlorate trihydrate. Cellulose aceto-di- and -tetrachlorostearates containing 7.3–9% and 20–23% Cl, respectively, are similarly obtained. (B) Cellulose material is treated with a halogen-substituted fatty acid containing more than five carbon atoms in presence of an organic acid anhydride which promotes esterification, but which does not itself esterify cellulose. Thus the reaction bath obtained from 8 pts. of cellulose, 40 pts. of chloroacetic anhydride, 20 pts. of chloroacetic acid, and 0.05 pt. of magnesium perchlorate trihydrate at 60–65°, with 15 pts. of α -bromostearic acid and 4 pts. of acetic acid affords a cellulose aceto- α -bromostearate containing 11.3% Br. The preparation of cellulose acetobromostearate (23% Br), acetochlorostearate (8.5% Cl), acetotetrachlorostearate (14% Cl), and aceto- α -bromohexanoate (5% Br) is described. R. BRIGHTMAN.

Production of viscose. I. G. FARBENIND. A.-G. (B.P. 287,492, 21.3.28. Ger., 21.3.27).—Crude cellulose xanthate is treated in one or two stages for a short time (5–10 min.) at not above 16° with aqueous alcohol—particularly methyl alcohol—which, taking into account the water content of the xanthate, contains 70–80% by vol. of anhydrous alcohol in the first stage. The mass is then pressed to remove the liquid, broken up without drying, and at once dissolved to form viscose. F. R. ENNOS.

Production of compounds resembling celluloid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 308,395, 12.1.28).—Cellulose esters or ethers are incorporated with a neutral phosphoric ester containing at least one radical of a primary aliphatic alcohol with more than four carbon atoms, the remainder belonging to the aliphatic or hydroaromatic series, *e.g.*, diisobutyl phosphate; other materials usually employed in the manufacture of artificial masses may be added if desired. F. R. ENNOS.

Solvents for nitrocellulose and for acetylcellulose. L. E. CLÉMENT, ASS'Y. to DU PONT-PATHÉ FILM MANUF. CORP. (U.S.P. 1,713,512 and 1,713,997, 21.5.29. Appl., 25.11.24. Fr., 30.10.24).—A mixture of anhydrous ethyl alcohol and acetone is used, the proportions, by vol., being: (A) 4:2, and (B) 1:1. H. ROYAL-DAWSON.

Manufacture of artificial [silk] products by dry-spinning processes. H. DREYFUS (B.P. 312,203, 18.1.28).—The spinning solution consists of a cellulose ester or ether with a volatile substance, *e.g.*, acetone, a non-solvent liquid of higher b.p. than the volatile substance, *e.g.*, xylene, and not more than 30% of the weight of the cellulose compound of a solvent of higher

b.p. than the non-solvent liquid, *e.g.*, diacetone alcohol.
F. R. ENNOS.

Manufacture of artificial filaments from viscose. W. HARRISON (B.P. 309,053, 1.11.27, 19.12.27, and 5.6.28).—Viscose threads of high strength are obtained, without the use of acids of high concentration as swelling agents, by spinning viscose solution, preferably prepared from unmatured or slightly matured alkali-cellulose, into an acid coagulating bath of such a type, *e.g.*, of p_H less than that of 7% sulphuric acid, that the viscose, though rapidly coagulated, is relatively slowly decomposed into cellulose, and stretching the filaments 25–100% or more during the transition stage from the coagulated viscose to cellulose. To provide more opportunity for adjusting the time of stretching, decomposition of the viscose may be delayed by passing the coagulated filaments into a bath of water containing substances, *e.g.*, neutral salts with or without the addition of glucose or the like, which, while permitting the swelling action of the water, will prevent dissolution of the filaments.
D. J. NORMAN.

Manufacture of artificial threads, bands, films, etc. from solutions of cellulose esters and ethers.

Manufacture of cellulose esters. RUTH-ALDO Co., Inc., Assees. of H. L. BARTHELEMY (B.P. 282,787 and 282,789, 28.12.27. Fr., 28.12.26).—(A) A solution of the cellulose ester or ether is coagulated in the desired form by means of alcohols or polyalcohols which are capable of taking up the volatile solvent of the ester or ether, *e.g.*, propyl alcohol, glycol, cyclohexanol, etc., to which a hydrocarbon such as xylol may be added. (B) After acetylation of cellulose in the presence of sulphuric acid as catalyst, the excess of acetic anhydride is destroyed by addition of an aqueous solution of formic or acetic acid, the whole being cooled to the initial temperature. A mixture of water, formic or acetic acid, and hydrochloric acid is then added to produce partial hydrolysis which is accompanied by destruction of the harmful cellulose acetosulphuric esters; the quantity of hydrochloric acid should be at least equivalent to that of the sulphuric acid used as catalyst, the total water in the mass being at the same time adjusted to 5–15% of the total acids present after the addition of the hydrolysing mixture.
F. R. ENNOS.

Manufacture of [waterproof] artificial material and articles from alkyl cellulose. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 308,284, 13.10.27).—A mixture of an aqueous solution of a cellulose alkyl ether with a colloidal substance, either as such or in the form of a solution or an emulsion, *e.g.*, a drying oil or an aldehyde condensation product, is made into the desired shape and treated by suitable means such as heating to convert the latter into a sparingly soluble or insoluble state. [Stat. ref.]
F. R. ENNOS.

Apparatus for spinning solutions of cellulose ethers or esters. RUTH-ALDO Co., Inc., Assees. of (Miss) M. KLEIN (B.P. 292,561, 22.6.28. Fr., 22.6.27).—Each cell is subdivided by one or more diaphragms into an upper zone containing the spinning nozzle and lower heating zones where aspiration is effected and which are traversed successively by the spun thread.

The diaphragms, which consist of a fixed portion and a central hinged portion, the latter containing a triangular notch which may be partially closed so as to leave only sufficient space for the passage of the thread, serve to prevent hot air passing into the upper zone.
F. R. ENNOS.

Treatment of fibrous materials for papermaking etc. E. V. HAYES-GRATZ. From C. LEYST (B.P. 311,945, 21.5.28).—Straw, esparto grass, etc., after pretreatment, if desired, by exposure in a moist alkaline condition at 30° for 3–6 days, is cooked with water and about 30% of lime for 6–8 hrs., washed free from lime, and ground to a flour-like consistency with or without an intermediate drying. Alternatively, the material may be first ground and then cooked with lime and water.
F. R. ENNOS.

Treatment of fibrous material [with synthetic resins]. A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,912, 18.12.28. Appl., 16.3.23).—Paper or other sheet fabric is impregnated with aqueous formaldehyde containing 10% of hexamethylenetetramine, dried, and passed through a mixture of cresol and 20% of tung oil previously treated with a little formaldehyde, or through a solution of soluble phenol-formaldehyde resole. The sheet is then heated at 100°, and may afterwards be hardened as usual.
C. HOLLINS.

[Discharge valve for paper] pulp strainers of the drum type. A. WHITE, W. M. WALLACE, J. and R. STEWART, Exors. of J. WHITE (B.P. 311,948, 26.5.28).

Sodium acetate (U.S.P. 1,695,742).—See VII. **Wool fat** (B.P. 286,252).—See XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effect of light during the bleaching of some coloured cellulose materials by hypochlorites. F. SCHOLEFIELD and C. K. PATEL (J. Soc. Dyers and Col., 1929, 45, 175–178).—When cotton or viscose, dyed with certain vat yellows and oranges, is subjected in the dark to the action of hypochlorite solutions no appreciable tendering takes place. On the other hand, if dyed material, saturated with hypochlorite, is exposed to the action of daylight or the light from the carbon arc of the Fadeometer, the cellulose is tendered when dyed with certain vat yellows, oranges, and reds. Indanthrene Yellow G produces no tendering under these conditions. The tendering action in the case of both sodium hypochlorite and hydrogen peroxide is to be explained by the absorption of rays of light by the yellow and orange dyes, which rays are not absorbed in the case of blues, violets, etc.
L. G. LAWRIE.

Dyeing without colouring matters. BRUÈRE (Ann. Falsif., 1929, 22, 132–139).—Soluble and metallic salts under certain conditions of acidity in the presence of sodium nitrite will develop colours on animal fibres or, after "pseudo-animalisation," on artificial silks, with or without the addition of phenolic compounds or amines; a wide range of permanent colours may thus be produced. In order to determine in specific cases whether the colouring material has been produced in this way, the ordinary procedure according to the tables of

Green, Rota, etc. is followed, when it will be found that the yellow colour with nitric acid given by the indigo group will usually be present in this class of dye (chromium colours often showing a violet colour); sulphuric acid gives only slight colours, and hydrochloric acid nothing distinctive; the ash will furnish useful information, and a comparison of the shade with the colour possibilities of the various cations will confirm the findings. Theoretically, dissolution of the animal fibres in hot sodium hydroxide followed by saturation with sulphuric acid should give a solution of sodium sulphate and the colour metal as sulphate, but in practice only iron can thus be readily detected, as the other metals form organo-metallic complexes.

D. G. HEWER.

Action of alkalis and other swelling agents on viscose silk. A. J. HALL (J. Soc. Dyers and Col., 1929, 45, 171—175).—Viscose suffers shrinkage in length, loss of lustre, strength, softness of handle, and elasticity by treatment with caustic soda (*d* 1.06—1.35) and subsequent washing. The harmful effects take place during the washing, and the viscose will retain its original properties if washed in solutions of certain inorganic and organic substances. The effect of caustic potash decreases as the temperature is raised, and at temperatures above 0° it has practically no deleterious action. By the use of caustic potash instead of caustic soda it is possible to mercerise cotton goods containing viscose without the artificial silk being adversely affected. The swelling action of caustic soda, concentrated sulphuric acid, and phosphoric acid on viscose increases similarly the affinity of the viscose for direct dyes.

L. G. LAWRIE.

PATENTS.

Colouring of cellulose esters and of solutions or compositions containing such esters. W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,840, 11.10.27).—A pigment is incorporated with dry, ungelatinised cellulose nitrate together with a gelatiniser and/or suitable oil such as linseed oil or castor oil.

C. HOLLINS.

Use of sulphonylates for stripping dyes. C. S. HOLLANDER, Assr. to ROHM & HAAS Co. (U.S.P. 1,696,164, 18.12.28. Appl., 5.2.27).—A soluble zinc salt (sulphate, chloride, acetate) is added to sodium formaldehyde-sulphonylate to stabilise the solution.

C. HOLLINS.

Treatment [mordanting and weighting] of cellulose derivatives [esters and ethers] and products made therewith. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. [A] 309,899 and [B] 309,876, 15.11.27. Addn. [B] to B.P. 258,874; B., 1928, 228).—(A) Mordanting and (B) weighting of cellulose ester and ether fabrics, yarn, etc. may be effected at lower temperatures in the presence of zinc salts (chloride).

C. HOLLINS.

Manufacture of [weighted] artificial silk. Soc. CHIM. DES USINES DU RHÔNE (B.P. 294,623, 13.1.28. Fr., 29.7.27).—The process of B.P. 275,553 (B., 1928, 639) is applied for preparing cellulose esters or ethers containing 2—25% of weighting agents, *e.g.*, barium sulphate. The resulting product when spun gives silk of subdued lustre.

D. J. NORMAN.

Sizing of artificial silk. M. GUINET (F.P. 630,929, 16.3.27).—The fibres are coated with a mixture of a sulphonated substance (sodium sulphoricinate) and a colloid, the latter being produced by the action of a boiling alkaline solution on animal skins or on gelatin.

A. B. MANNING.

Treatment ["animalisation"] of cotton and artificial silk fibres. CHEM. FABR. VORM. SANDOZ (B.P. 284,358, 28.1.28. Ger., 28.1.27. Cf. B.P. 195,619 and 249,842; B., 1926, 128; 1927, 71).—Cotton, mercerised cotton, viscose silk, or other regenerated cellulose silk is treated at 110° with benzene- or toluene-sulphonyl chloride and a tertiary base (pyridine) in presence or absence of a solvent (nitrobenzene). The treated textile can be dyed with any acid dye.

C. HOLLINS.

Production of dyeings fast to light. P. RABE, H. STÖTTER, B. WENK, and W. SCHEPSS, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,694,562, 11.12.28. Appl., 15.12.26. Ger., 22.12.25).—See B.P. 283,281; B., 1928, 189.

Dyeing of cellulose derivatives (U.S.P. 1,695,654).—See IV. **Printing preparations** (B.P. 311,795).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

"Poupin" system [for the lixiviation and crystallisation of caliche etc.]. H. GERLACH, WARNECKE, and F. BLANCKENHORN (Caliche, 1929, 10, 534—556; 11, 5—29).—Full technical details are given of the working of the system.

H. F. GILLBE.

The Trona enterprise [at Searles Lake, California]. G. R. ROBERTSON (Ind. Eng. Chem., 1929, 21, 520—524).—The lake consists of a solid crystalline layer beneath which is a brine containing approx. 34.6% of a complex mixture of salts with the following ions: chloride, sulphate, carbonate, borate, phosphate, bromide, sulphide, sodium, potassium, and lithium, together with traces of arsenic, calcium, iron, aluminium, and antimony compounds. Potassium chloride is present to the extent of 4.75%. The principles on which the extraction of this salt is based are illustrated by phase-rule diagrams, and a brief description of the main features of the plant employed is included. The daily output of the plant is 240 tons of potassium chloride, 130 tons of borax, and 10 tons of boric acid.

H. INGLESON.

Rapid determination of nitrogen by Kjeldahl's method. H. LUNDIN and J. ELLBURG (Woch. Brau., 1929, 46, 133—137, 147—149).—Details are given of a technique employing a reaction mixture of hydrogen peroxide with sulphuric and phosphoric acids and potassium sulphate, with copper and mercury as catalysts. Distillation is performed from the digestion flasks through air-cooled condensers, and the whole process may be completed in 1 hr.

F. E. DAY.

Chlorine Institute standard valves for chlorine containers under 15 tons capacity. R. T. BALDWIN (J. Amer. Water Works' Assoc., 1929, 21, 815—819).—A standard pattern valve adopted by American chlorine manufacturers is of the internal-thread type, repackable

under pressure, and has an outlet suitable for use with present standard pipe connexions. C. JEPSON.

Lixiviation. FONTAINE.—See I. **Oxidation of ammonia from coke-oven liquors.** PERLEY and WHITE.—See II. **Rhenania phosphate and ammonia-superphosphate as fertilisers.** DENSCH and others. **Solubility of phosphorites.** ROSANOV.—See XVI.

PATENTS.

Manufacture of beryllium oxide. H. A. SLOMAN and A. C. VIVIAN (B.P. 312,007, 27.8.28).—A concentrated solution containing beryllium sulphate is saturated with beryllium hydroxide, and basic beryllium sulphate, $\text{BeSO}_4 \cdot 4-5\text{Be}(\text{OH})_2$, is precipitated by diluting the solution with water in the presence of ammonium sulphate as coagulant. Beryllium oxide free from sulphate is obtained by heating a mixture of the product with carbon at a temperature below that at which the oxide is rendered insoluble in acids, e.g., at about 700° ; excess carbon subsequently being burnt away at a low temperature. L. A. COLES.

Production of sodium acetate. F. G. RAWLING (U.S.P. 1,695,742, 18.12.28. Appl., 11.5.27).—Sodium bicarbonate is added to the sulphite in the pulping of wood; the extract, which contains only one third of the lignin and no cellulose, is concentrated, sodium hydroxide and lime are added, and the mixture is heated at $200-280^\circ$. Sodium acetate is recovered from the filtrate. C. HOLLINS.

Production of ammonium sulphate by the treatment with gypsum of an ammoniacal solution used for the scrubbing of gases. UNION CHIM. BELGE, SOC. ANON. (B.P. 308,243 and 309,852, [A] 10.4., [B] 7.6.28. Belg., [A] 20.3., [B] 16.4.28. Addns. to B.P. 307,037; B., 1929, 472).—In modifications of the prior patent, (A) ammonium sulphate is precipitated by saturating the liquor with ammonia and removed, and excess of ammonia is expelled for re-use, before the liquor enters the scrubber, which is fitted with a cooler to facilitate absorption, and (B) sufficient ammonia is added to the liquor to effect almost complete precipitation of the ammonium sulphate; added water is removed as liquor instead of by evaporation, ammonia in the withdrawn liquor being recovered by heating it with milk of lime, and the liquor, after filtration to remove calcium carbonate is heated to expel residual carbon dioxide, which is returned to the scrubber. L. A. COLES.

Production of copper sulphate. M. SPEICHERT, Assr. to HÜTTENWERKE TEMPELHOF A. MEYER (U.S.P. 1,715,871, 4.6.29. Appl., 10.1.27. Ger., 13.1.26).—A copper-tin-lead-antimony alloy is melted with sulphur, the mixed sulphides so produced are roasted, the product is leached with sulphuric acid, and the liquor is freed from insoluble residue. F. G. CROSSE.

Manufacture of metallic [aluminium] chloride. R. DE M. TAVEAU and C. B. TYGERT, Assrs. to TEXAS CO. (U.S.P. 1,698,324, 8.1.29. Appl., 17.1.22).—Bauxite, or other aluminium ore, is dehydrated in a vertical dehydrating chamber, spaced with inclined baffles to agitate and distribute the falling bauxite and heated internally by gas burners near to the base. The de-

hydrated bauxite falls through a hopper into a chlorinating chamber, heated at $370-425^\circ$ externally and provided with movable horizontal baffles, in countercurrent with an ascending stream of phosgene introduced at the base of the chamber. The aluminium chloride passes from the upper end of the reaction chamber to a series of condensers, and the excess of phosgene is destroyed by leading it to the base of the dehydrator to assist the dehydration. R. BRIGHTMAN.

Manufacture of chromium compounds. I. G. FARBENIND. A.-G., and K. and K. S. CARPMAEL (B.P. 312,097, 20.12.27. Addn. to B.P. 259,447; B., 1926, 1013).—The coarse ore used as a filler in the process described in the prior patent is, after cleaning with sulphuric acid, repeatedly returned to the process until it contains nearly 70% of impurities, when it is ground, mixed with dolomite and sodium carbonate, and roasted to recover its chromium content. A. R. POWELL.

Alkali titanate and its manufacture. L. W. RYAN, Assr. to TITANIUM PIGMENT CO., INC. (U.S.P. 1,697,929, 8.1.29. Appl., 2.6.25).—Hydrated titanium oxygen compounds, e.g., metatitanic acid or basic titanic sulphate, are heated at $100-200^\circ$ with about 1.6–2 pts. of potassium hydroxide or carbonate, giving an alkali titanate, $d\ 2.3-2.9$, soluble in organic and dilute mineral acids. As example, potassium metatitanate thus obtained dissolved in oxalic acid at 80° affords titanium potassium oxalate.

R. BRIGHTMAN.

Recovery of titanium compounds. TITANIUM PIGMENT CO., INC., Asses. of W. F. WASHBURN (B.P. 288,569, 15.3.28. U.S., 12.4.27).—Finely-ground ilmenite is mixed with 72–92% sulphuric acid and steam is injected into the lower part of the reaction vessel until the temperature reaches 120° and the mass thickens; a small quantity of water is then forced into the lower part of this thick pulp, followed by vigorous streams of air to keep the mass in agitation and to render it porous, and finally the cooled solid mass is treated with just sufficient water to dissolve the iron and titanium sulphates. The clear solution is agitated with scrap iron or zinc to reduce ferric to ferrous sulphate, and the titanium is recovered by hydrolysis in the usual way. A. R. POWELL.

Catalyst. F. W. WEBER (U.S.P. 1,698,009, 8.1.29. Appl., 1.12.24).—Asbestos, amphibolite, or other siliceous material is digested with hydrochloric acid, and, after removal of the acid liquid, impregnated with excess of 10–20% sodium silicate solution to alkalinity. Excess of solution is removed, the mass dried, heated to incandescence, and, after washing out sodium chloride, impregnated with thorium nitrate or other rare-earth salt. On raising again to incandescence to convert, e.g., the thorium nitrate into the dioxide, a porous catalyst is obtained, resistant to heat and acids except sulphuric acid. The silica-thoria-asbestos catalyst is valuable in synthetic organic oxidations, e.g., vanillin, as a heat-reflecting illuminating material, a refining agent for crude or cracked oils, and for filtration of slimy gelatinous substances. R. BRIGHTMAN.

Separation of gases [hydrogen and carbon dioxide]. J. REILLY and E. W. BLAIR, Assrs. to

COMMERCIAL SOLVENTS CORP. (U.S.P. 1,706,707, 26.3.29. Appl., 8.4.25).—Fermentation gas etc. is washed under pressure with a solvent to remove the greater part of the carbon dioxide, and the process is repeated to remove the remaining carbon dioxide. The gases evolved on releasing the pressure from the second wash-liquor are returned to the process. L. A. COLES.

Sulphur-burning apparatus. O. BUSE, Assr. to GRASELLI CHEM. CO. (U.S.P. 1,714,657, 28.5.29. Appl., 2.11.25).—The apparatus consists of a gas conduit having within it an inclined corrugated plate, above which are means for delivery thereon of molten sulphur.

H. ROYAL-DAWSON.

[Furnace for] generating gaseous oxide of sulphur [for preventing or extinguishing fire or for disinfecting]. CLAYTON INSTALLATIONS, LTD., and W. A. MUIRHEAD (B.P. 312,746, 30.3.28).—Rapid generation of the gases is attained by distributing an inflammable liquid over the sulphur to ignite it and to heat up the furnace; by supplying a forced or an induced draught of air over the sulphur, the change over from one to the other draught being regulated automatically according to the temperature of combustion; by mixing absorbent non-inflammable material, e.g., asbestos in the form of mats, with the sulphur to act as wicks; and by external heating of the furnace, when required. L. A. COLES.

Oxides of nitrogen from ammoniacal liquor (U.S.P. 1,713,045).—See II. Hypochlorites (B.P. 311,218 and 311,253).—See XI. White lead (B.P. 311,986).—See XIII. Vulcanisation of rubber (B.P. 290,602).—See XIV.

VIII.—GLASS; CERAMICS.

Vitreous state. M. O. SAMSOEN (Bull. Soc. d'Encour., 1929, 128, 185—204).—Qualitative dilatometric observations with a variety of vitreous bodies show that anomalous expansion is not due to the presence of silica, but is a general property of vitreous substances. For all such substances there exists, independently of the viscosity, a temperature, or a short temperature interval, at which a physical transition occurs, and is manifested by discontinuity in the temperature-expansion curves. It is suggested that there exists for a liquid a certain viscosity above which the molecular mobility vanishes. This conception is supported especially by experiments with the binary system $\text{SiO}_2\text{--Na}_2\text{O}$. H. F. GILLBE.

Active silica. Soluble silica in clays. A. FIOLETOVA (Trans. State Exp. Inst. Silicate, Moscow, 1927, No. 21, 111—113).—Two samples of clay, dried at 100—110°, ground, and treated for 1 hr. with constant stirring with *N*-hydrochloric acid by Dementev's method, lost 0.05 and 0.09% of silica, respectively. After cooling for 2½ hrs., heating at 530°, and again cooling for 2½ hrs. the samples similarly lost 0.25 and 0.17% of silica. The result for extraction immediately after heating was 0.29 and 0.17%. Extraction after heating at 1300° gave 0.21 and 0% loss, respectively, of silica.

CHEMICAL ABSTRACTS.

Purification of clay by electrophoresis. A. S. TAL (Trans. State Exp. Inst. Silicate, Moscow, 1927, No. 21,

115—131).—An anode of lead containing 14% Sb and an ammoniacal electrolyte, with a peat extract depolariser, were used. When heated, the material gave a lighter product; the contraction for high temperatures was greater than that for 100°.

CHEMICAL ABSTRACTS.

Durability of arc furnaces. KOTHNY.—See XI.

PATENTS.

Lehrs. C. E. FRAZIER and J. SYLVESTER (B.P. 289,017, 16.2.28. U.S., 20.4.27).—The lehr consists of a double-walled sectional tunnel, with the hollow space between the walls at the front filled with insulating material. In the middle portion, sides and bottom only are so packed, and the outer shell has a refractory roof with hinged dampers. The side-wall chambers are without packing in the rear section and are adapted for air-cooling. A horizontal metal plate supports an endless, wire-woven belt throughout the length of the lehr. A metallic combustion chamber below this plate at the front of the lehr is connected by several horizontal metal flues to a chimney header, and this method of heating may be supplemented or substituted by electrical heating units. Damper grids are provided above the heating flues for temperature control. An entry door and a series of curtains prevent draughts within the lehr.

A. COUSEN.

Manufacture of plate glass and sheet glass for window panes from pure fused transparent silica. QUARTZ ET SILICE (B.P. 302,895, 23.11.28. Fr., 23.12.27).—Ingots of opaque fused silica are heated to 2000° and squeezed out into transparent sheets under high pressure, maintained until solidification occurs. A. COUSEN.

Heat treatment [of ceramic ware]. Tunnel kiln construction. T. G. McDUGAL, Assr. to A.C. SPARK PLUG CO. (U.S.P. 1,713,851—2, 21.5.29. Appl., [A] 12.12.25, [B] 30.7.27).—In both instances a tunnel kiln is provided with a longitudinal slot through which projects the travelling support, the mass of which is small compared with that of the heating walls. Heating is by radiation from the walls of a muffle.

J. A. SUGDEN.

Manufacture of rubber-bonded abrasive articles. CARBORUNDUM CO., LTD. From CARBORUNDUM CO. (B.P. 311,104, 15.5.28).—Abrasive particles are mixed with a rubber dispersion, compounded or otherwise, the latter being preferably in a somewhat viscous but stirrable condition, produced, for example, by the addition of glue or by concentration; the mixture is then coagulated, moulded, dried, and vulcanised. Alternatively, the thickening of the latex may be effected by coagulation into a curd-like condition.

D. F. TWISS.

IX.—BUILDING MATERIALS.

Hygrometer for use in timber-seasoning kilns. E. GRIFFITHS (Proc. Physical Soc., 1929, 41, 426—427).—The hygrometer is of the wet-and-dry-bulb type using mercury-in-steel thermometers. The bulbs and capillaries are enclosed in a steel tube which can be inserted through a 3-in. hole and explorations up to 12 ft. can

be made. One dial serves both thermometers, the two pointers being arranged so that one moves over the top face of the dial and the other is brought round from beneath on to the outside edge. An electrically-operated fan draws air past the two bulbs. The moistening of the covering over the wet bulb is effected by a reservoir and by connexion to an external water supply.

N. M. BLIGH.

Influence of the electric current on wood. B. F. SCHWARZ (Bull. Inst. Pin, 1928, 215—220; Chem. Zentr., 1928, ii, 2762).—An apparatus for the determination of the electrical resistance of wood is described, and the effect of the current is discussed.

A. A. ELDRIDGE.

Road tar. MALLISON.—See II.

PATENTS.

Ageing of calcined gypsum. S. G. MCANALLY (U.S.P. 1,713,879, 21.5.29. Appl., 4.10.26).—The pulverised product is agitated, while to each ton of it are added 70 lb. of water.

F. G. CROSSE.

Slowly-setting hydraulic materials with high initial resistance. E. GOFFART (B.P. 294,111, 13.7.28. Belg., 16.7.27).—The setting of a highly aluminous cement (so quick-setting as to be unusable) is delayed by the addition of a small amount of a colloid such as casein, albumin, gum, gelatin, etc.

J. A. SUGDEN.

Treatment of concrete surfaces. G. B. ELLIS. From CHEM. FABR. GRÜNAU, LANDSHOFF & MEYER A.-G. (B.P. 312,216, 22.2.28).—The surface of the mould is treated with a solution of the products of the alkaline hydrolysis of albumins or proteins (*e.g.*, alkali salts of lysalbic and protalbic acids). This treatment delays the local setting of the binding agent, so that the surface layer of the material may be washed off when the mould is removed.

J. A. SUGDEN.

Manufacture of cellular building materials. L. DESMARQUEST (B.P. 312,764, 18.4.28).—A mixture of clay etc. previously baked at 200—1100°, cement, unbaked clay, and, *e.g.*, powdered aluminium, made to a paste with water, is dried in moulds rendered waterproof, *e.g.*, by greasing, and the moulded blocks are calcined at about 1300°.

L. A. COLES.

Treatment of wood. L. P. CURTIN, Assr. to WESTERN UNION TELEGRAPH Co. (U.S.P. 1,698,622, 8.1.29. Appl., 16.4.27).—Wood or other cellulose material is rendered resistant to fire or fungus or insect attack by impregnation with a solution of ammonia and arsenious acid, containing upwards of 5% As_2O_3 , which deposits arsenious oxide on exposure to air, or with an emulsion of such a solution and a liquid hydrocarbon.

R. BRIGHTMAN.

Waterproof compositions (B.P. 312,467).—See II.
Laminated products (B.P. 299,441).—See XV.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Single crystals of iron. H. GRIES and H. ESSER (Arch. Eisenhüttenw., 1928—9, 2, 749—761; Stahl u. Eisen, 1929, 49, 879—880).—To obtain large single crystals of iron, the metal must be as pure as possible, and in any case the carbon content should not exceed

0.02%. The necessary deformation required to produce rapid crystal growth increases rapidly with an increase in the size of the original crystal grains, but rapid growth is obtained by prolonged annealing at 880° of metal containing 100—140 grains/mm.² after rolling or extending in a tensile machine to produce a deformation of 3—3.25%. At 880° annealing for 48 hrs. usually suffices, but at slightly lower temperatures a much longer time is required to produce a single-crystal rod of the dimensions used in the usual tensile test. When the original deformation is produced in a tensile machine there is a tendency for coarse secondary crystals to form on the surface of the single crystal; these may be removed by etching with a mixture of 1 vol. of concentrated hydrochloric acid and 9 vols. of concentrated nitric acid. The presence of less than 0.05% O in the metal has little effect on the production of single crystals, whereas with more than 0.055% O no single crystals can be obtained; these results indicate that the solid solubility of oxygen in iron is of the order of 0.05%. In recrystallisation tests on rolled single crystals, new crystals began to form on the faces of the rhombic dodecahedron after 16%, on octahedron faces after 21%, and on cube faces after 24.3% deformation. The new crystals formed on the cube faces were of much greater size than those on the dodecahedron.

Influence of alloying elements on the polymorphism of iron. F. WEVER (Arch. Eisenhüttenw., 1928—9, 2, 739—748; Stahl u. Eisen, 1929, 49, 839—840).—The elements which form solid solutions with iron may be divided into four groups, viz.: (I) those which lower the A3 point and broaden the γ -field, *e.g.*, the elements of the eighth group of the periodic system and manganese, which immediately precedes iron; (II) those which give an enclosed γ -field in the equilibrium diagram, *e.g.*, beryllium, aluminium, silicon, phosphorus, titanium, vanadium, chromium, arsenic, niobium, molybdenum, tin, antimony, tantalum, and tungsten; (III) those that give an equilibrium diagram in which the transformation lines lead out from one another into regions of heterogeneous equilibrium, *e.g.*, carbon, nitrogen, copper, zinc, and gold; and (IV) those that narrow the γ -field, *e.g.*, boron, sulphur, zirconium, and cerium. It thus appears that elements isomorphous with γ -iron increase the stability of the γ -phase, and those isomorphous with α -iron tend to repress the $\alpha \rightarrow \gamma$ transformation. All the elements of groups I and III, *i.e.*, those that increase the γ -range, occur at the lowest points of the atomic radius-atomic number curve, whereas the elements of groups II and IV occur chiefly on the descending portions of the curve, and the elements that are insoluble in iron generally on the ascending portions and at the peaks.

Specific volume of white pig iron. L. ZIMMERMANN and H. ESSER (Arch. Eisenhüttenw., 1928—9, 2, 867—870; Stahl u. Eisen, 1929, 49, 912—913).—The sp. vol. of white pig iron containing 3.5—3.9% C, 0.13—0.67% Si, and 0.06—0.1% Mn has been determined at temperatures up to 1300° by heating the metal, *in vacuo*, in a porcelain crucible. The mean value of the volume change during fusion is about 1.33—1.41% of the maximum value of the sp. vol. in the solid state.

A. R. POWELL

Adherence of thin sheets [of iron during hot-rolling]. W. TITZE (Stahl u. Eisen, 1929, 49, 897—903).—For the manufacture of thin iron sheets for galvanising or tinning the carbon content should preferably be between 0.08 and 0.16%, and the metal must be thoroughly deoxidised to prevent the sheets sticking together when doubled over in the last stages of hot-rolling. The most satisfactory tests for these conditions are the bending test on the quenched material and the flattening test on the white hot metal; in the latter test, the usual dip sample from the furnace is flattened under the hammer at a welding heat and then examined for unsoundness. For the production of sheets thinner than 0.55 mm., the addition of a small amount of copper or, better, ferroc carbon-titanium to the metal in the ladle is recommended. Deoxidation with titanium yields a better quality sheet for deep drawing, and to a great extent prevents segregation of sulphur and phosphorus; nevertheless, the upper portion of the ingot should always be cut off and used where a lower grade of metal is required. A. R. POWELL.

Third report on heterogeneity of steel ingots.

I. Introduction. II. Liquidus and solidus ranges of some commercial steels. III. Solubility of iron and manganese sulphides in steel. J. H. ANDREW and D. BINNIE. IV. Interim report on the density of molten steel. C. H. DESCH and B. S. SMITH. V. Effect of latent heat on solidification of steel ingots. N. M. H. LIGHTFOOT (Iron and Steel Inst., May, 1929. Advance copy. 71 pp.; cf. B., 1928, 409).—II. The phase changes in straight carbon, nickel, nickel-chromium, and nickel-chromium-molybdenum steels were compared with those given by Armco iron and four crucible steels by a thermal method. The liquidus and solidus curves for the commercial steels conform with respect to carbon content and temperature with the iron-carbon diagram, provided the manganese content is below 0.45%. The liquidus for nickel and nickel-chromium steels lies below, whilst the solidus practically coincides with, that of plain carbon steels. A method is given for calculating the freezing and melting ranges of low-nickel and low-chromium steels. The addition of molybdenum to nickel-chromium steels depresses the solidus by a proportional amount; the liquidus is unaffected. It is suggested that the decreased tendency to segregate shown by nickel and nickel-chromium steels is due to the narrower range between the liquidus and solidus and to the obliteration of the peritectic transformation. The addition of molybdenum to nickel-chromium steels widens the liquidus-solidus range on account of the segregation of molybdenum carbide. It is inferred that metals which form a solid solution with the iron phase depress the liquidus only, whilst those which form a carbide depress the solidus also. The peritectic reaction in carbon steels is discussed and its importance emphasised, and micrographs are shown of various sections from a specimen differentially heated and melted at one end. The liquidus and solidus curves derived from carbon steels run almost parallel with those of Carpenter and Keeling.

III. To determine whether the sulphides were soluble at all in solid steel the effect of these compounds on the magnitude and temperature of the Ar₃ change in iron

was determined. The results show definitely that sulphur may be retained in solution in an iron-sulphur alloy by rapidly chilling the melt, and that deposition of iron sulphide occurs on reheating the alloy to a temperature above 900°. The results with manganese sulphide were indefinite, but it appears probable that this compound is only very slightly, if at all, soluble in solid iron.

IV. The Archimedean principle was adopted. Preliminary results are given which are lower than those given by Berlin (B., 1927, 487), but in good agreement with those published by Benedicks, Berlin, and Phragmén (Iron Steel Inst., Carnegie Schol. Mem., 1924, 13, 129).

V. Mathematical.

M. E. NOTTAGE.

Influence of nitrogen on special steels and experiments on case-hardening with nitrogen. S. SATOH (Rev. Mét., 1929, 26, 248—258).—The increase in surface hardness due to absorption of nitrogen on heating in ammonia at 560—580° steels containing aluminium, titanium, manganese, zirconium, molybdenum, tungsten, or uranium, with and without chromium, has been determined. In all cases the added element produced a considerable increase in hardness, the maximum effects being obtained with aluminium and titanium. Boron, magnesium, copper, and cerium have only a relatively small action in nitrogen case-hardening. The use of nickel coatings on arc-welding electrodes has practically no effect in preventing the absorption of atmospheric nitrogen by the weld, although it tends to retard the separation of the characteristic nitride needles owing to their greater solubility in the nickel-iron solid solution.

A. R. POWELL.

Influence of the method of manufacture and the annealing atmosphere on the surface structure of tool steels. E. ZINGG, P. OBERHOFFER, and E. PIVOVARSKY (Stahl u. Eisen, 1929, 49, 721—725; 762—768).—Cementation of steel at 980—1000° in wood charcoal converts the surface austenite into free cementite which with plain carbon steels decomposes on prolonged annealing and slow cooling into temper carbon and γ -solid solution, the crystals of which are totally enclosed in a film of carbon so that further formation of austenite from gaseous carburising agents is prevented. The presence of 0.5% Cr, W, or V in the steel prevents decomposition of the cementite and carburising can continue up to 3% C. Prolonged annealing of high-carbon steels and high-speed tool steels without decarburisation or further cementation at temperatures up to 1300° may be effected in an atmosphere of 2 vols. of carbon monoxide and 1 vol. of hydrogen such as is produced by the incomplete combustion of acetylene with oxygen. This gas mixture is also suitable for salt-bath annealing as it prevents oxidation of the steel and also retards considerably the decarburising action of the fused salts; hence, hardened tool steels may be case-hardened at 580—600° in salt baths under an atmosphere of this gas mixture without surface etching or internal softening taking place. Diffusion, carburisation, and decarburisation experiments on a wide variety of steels indicate that the solubility of carbon in α -iron decreases from 0.035% at 720° to 0.006% at the ordinary temperature, and that at just below the A₁ point the rate of diffusion of carbon through α -iron is relatively great.

This rate is not affected by 0.5% Cr, 0.85% W, or 0.5% V alone, but when two or more of these elements are present together diffusion is much slower. The presence of impurities along the grain boundaries also retards diffusion, so that, by observing the behaviour of a steel during annealing above the A3 point and during subsequent decarburisation in moist hydrogen in the temperature zone between the Acl and Ar1 points, important conclusions as to its freedom from impurities which deleteriously affect its mechanical properties may be drawn.

A. R. POWELL.

Prolonged bending tests with steels. E. Houdremont and R. Mailänder (Stahl u. Eisen, 1929, 49, 833—839).—The fatigue strength of steel, S , under alternate bending stress may be expressed by the equation $S = 0.25(\sigma_s + \sigma_B) + 5$, where σ_s is the yield point and σ_B the ultimate strength. The influence of the composition, mechanical and heat treatment, and grain size of numerous steels on the ratio S/σ_B and $S/(\sigma_B + \sigma_s)$ have been investigated, and the results are recorded in a series of tables and graphs.

A. R. POWELL.

Tensile tests [of steels] at elevated temperatures. R. Guillet, J. Galibourg, and M. Samsoen (Compt. rend., 1929, 188, 1205—1208).—A modified Martens elasticimeter is described by which the extension of steel at 450° under the conditions of the usual tensile test may be measured. The results obtained for four samples of mild steel are tabulated; for plain carbon steels, after forging, the first elastic limit increases with the carbon content and is also increased by the presence of excess of silicon over the normal.

A. R. POWELL.

Properties of manganese in the basic open-hearth process. I. Kotaira and M. Maeda (J. Study Ferrous Met., 1928, 103, 110—133).—The effect of ferrous and manganous oxides on the m.p. and fluidity of the slag has been studied. Increase in the concentration of ferrous oxide causes decrease in the concentration of manganese in the steel. With increase of lime, the liberation of manganous oxide is favoured, and hence the amount of manganese in the steel is increased. Desulphurisation, and the effect of manganese on dephosphorisation, are discussed.

CHEMICAL ABSTRACTS.

Determination of molybdenum in steel. W. F. Murray (Chemist-Analyst, 1929, 18, 10).—The steel (5 g.) is dissolved in *N*-sulphuric acid (200 c.c.), ammonium persulphate (3—4 g.) is added, and the excess is removed by boiling. The solution, after dilution to 400 c.c. and saturation with hydrogen sulphide at 80° for 45 min., is filtered, the residue being washed with a dilute solution of hydrogen sulphide and ignited, the resulting oxide being dissolved in 20% sodium hydroxide (10 c.c.). The solution is boiled, filtered, diluted to 400 c.c., and slightly acidified with hydrochloric acid. After boiling (5 min.), 5% lead acetate solution (15 c.c.), 25% ammonium acetate solution (25 c.c.), and acetic acid (5 c.c.) are added with stirring. After being kept near the b.p. for 20 min., the solution is filtered and the precipitate is washed with hot water, ignited, and weighed as PbMoO₄. Tungsten, if present, is first determined in the usual way, the filtrate being evaporated

ated to fuming, diluted, boiled, and treated with persulphate.

CHEMICAL ABSTRACTS.

Determination of vanadium in steel. J. Kassler (Z. anal. Chem., 1929, 77, 290—298).—The steel turnings (3.75 g.) are dissolved by boiling with 40 c.c. of 1:5 sulphuric acid whereby the greater part of the vanadium remains in the carbide residue. The dissolved vanadium is recovered by diluting the solution with 100 c.c. of cold water and adding a suspension of zinc oxide in water until a little oxide remains undissolved. The solution is filtered, the residue washed twice with cold water and dissolved in hydrochloric and nitric acids, and the tungstic acid collected and washed. The filtrate is neutralised with sodium hydroxide, boiled with hydrogen peroxide, and poured in a thin stream (after addition of 1 g. of ferric chloride, if chromium is present) into 130 c.c. of hot 30% sodium hydroxide solution. The solution is boiled, cooled, diluted to 500 c.c., and 400 c.c. are filtered through a dry paper, acidified with hydrochloric acid, and treated with 20 g. of ammonium chloride and 25 c.c. of ammonia; the vanadium is separated from molybdenum by precipitation with a solution of 10 g. of manganese chloride and 5 g. of ammonium chloride in 50 c.c. of water. The precipitate is collected, washed, dissolved in sulphuric acid, and, after reduction with sulphur dioxide, the vanadium is titrated with permanganate. The small quantity of vanadium retained by the tungstic acid is determined colorimetrically with hydrogen peroxide after dissolving the precipitate in sodium hydroxide and acidifying with sulphuric acid.

A. R. POWELL.

Separation of aluminium as hydroxide from steels. W. J. Shaneman and J. J. Gallagher (Chemist-Analyst, 1929, 18, 6).—The solution of steel in hydrochloric acid, oxidised with nitric acid, is evaporated to a syrup and added dropwise to an excess of boiling 20% sodium hydroxide solution. After boiling, the cooled solution is diluted to a definite volume, of which half is treated with ammonium carbonate to precipitate the aluminium.

CHEMICAL ABSTRACTS.

Determination of sulphur in plain and alloy steels, pig iron, ferro-alloys, etc. K. Swoboda (Z. anal. Chem., 1929, 77, 269—277).—The metal (1 g.) in the form of turnings or powder is heated at 1150—1200° in a stream of oxygen for 3—6 min. and the gases are passed through silver nitrate solution whereby nitric acid is liberated and silver sulphite precipitated. Passage of the oxygen is continued for 7 min. after combustion is complete to remove carbon dioxide from the absorption flask, and the solution is titrated with 0.005*N*-sodium hydroxide using methyl-red as indicator. For the analysis of ferrochrome, high-speed steels, and high-chromium steels, 0.5 g. is used and the combustion effected at 1300°; to prevent sintering, some crushed electrolytic iron is mixed with the sample.

A. R. POWELL.

Crystalline changes in copper due to annealing. F. C. Howard and E. T. Dunn (Ind. Eng. Chem., 1929, 21, 550—553).—Photomicrographs of sections of cold-drawn copper rod have been taken, using a strong etching agent and relatively high magnification after the metal had been annealed at 700° for periods varying

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from 3 to 40 hrs. The constantly recurring regularity and uniformity of the lamination in the etched crystals may be due to some physical property of the substance.

H. INGLESON.

Precipitation of copper from mine waters at Britannia Mines, B.C. F. EBBUTT and W. E. SELNES (Trans. Canad. Inst. Min. Met., 1929, 31, 290—309).—Water from the Britannia Mines contains from 0.6 to 1.8 g./litre of copper as sulphate derived from the oxidation of pyrites and bornite in the crushed upper zones of the mine. This copper is recovered by running the water through an adit into settling tanks to remove silt, thence through a number of launders provided with perforated false bottoms on which is placed scrap tin-plate to precipitate the copper, which falls as a sludge through the perforations. To ensure efficient contact and to prevent accumulation of copper on the scrap, air is blown through the water from below the false bottom. The launders are used in series of two, in the first of which 66—75% of the copper is recovered and in the second 25—17%, giving a total recovery of just over 90%. The sludge from the first launder averages 65% Cu and that from the second 45% Cu; both sludges contain about 30% of moisture. The consumption of iron is about twice the theoretical owing to the presence of free acid and ferric sulphate in the water. The cost of recovering the copper is about 7.2 cents/lb.

A. R. POWELL.

Effect of mixing small quantities of cobalt with brass. D. IITSUKA (Mem. Coll. Sci. Kyoto, 1929, 12A, 179—198).—The ternary equilibrium diagram for the system copper-zinc-cobalt has been established in the region of copper 50—100%, zinc 0—50%, and cobalt 0—10%. Mechanical and corrosion tests were carried out on a number of alloys. β -Brass is improved in all its mechanical properties by addition of cobalt; α -brass is improved in strength but not in elongation.

C. W. GIBBY.

Influence of grain size on the corrodibility of brass and bronze. W. KÖHLER (Korrosion u. Metallschutz, 1928, 4, 227—230; Chem. Zentr., 1928, ii, 2748).—The influence of heating is examined.

A. A. ELDRIDGE.

Analysis of speculum metal. L. SCHWEITZER (Chem.-Ztg., 1929, 53, 457—459).—For the determination of copper and lead 0.5 g. of the powdered alloy is heated on the water-bath with 15 c.c. of a mixture of 1 vol. of bromine and 9 vols. of hydrobromic acid, d 1.38. The solution is evaporated to dryness and the residue again evaporated with 10 c.c. of the same mixture, whereby arsenic, antimony, and tin are volatilised. The residual bromides are boiled with 40 c.c. of 1:1 nitric acid to expel bromine, and the solution is diluted and electrolysed for copper and lead dioxide. Tin may be determined in a second 0.5 g. sample, if less than 12% Cu and 1% As are present, by direct dissolution in bromine and hydrochloric acid, reduction with iron, filtration to remove copper etc., and titration with iodine. An alternative and generally applicable procedure comprises dissolution of the alloy in nitric acid, collection of the metastannic acid, fusion with sodium peroxide, dissolution of the mass in hydrochloric acid, reduction of the solution with iron, and titration of the tin with iodine.

Antimony is determined in alloys containing less than 0.8% (As + Fe) and 15% Cu by titration with permanganate following dissolution of the alloy in sulphuric acid; in other cases the antimony must be isolated as the trisulphide in the usual way, and this is dissolved in sulphuric acid for permanganate titration. Arsenic is determined by distillation as bromide, sulphur by treatment of the alloy with concentrated hydrochloric acid and collection of the hydrogen sulphide in cadmium acetate solution, and nickel, iron, and zinc by the usual methods after separation of the hydrogen sulphide group.

A. R. POWELL.

Flotation practice at the Sullivan mill. C. T. OUGHTRED (Trans. Canad. Inst. Min. Met., 1929, 31, 310—322).—The ore consists of 13.6% of galena, 14.4% of zinc blende, 1% of iron pyrites, 61% of pyrrhotite, and 9.8% of rock matter, all in a very finely-disseminated form, so that it is necessary to crush the ore to 88% through 200-mesh before treatment in the flotation plant. The ore pulp from the grinding circuit containing 25% of solids is fed to lead rougher cells where the galena is floated using 0.85 lb./ton of a mixture of 60% of water-gas tar, 20% of coal-tar creosote, and 20% of cresylic acid as flotation agent; flotation of other minerals is prevented by addition of 3 lb. of sodium carbonate and 0.09 lb. of sodium cyanide per ton. The tailings from the lead cells pass to the zinc rougher cells where the blende is floated at 30° after adding 0.65 lb. of copper sulphate and 0.09 lb./ton of sodium dichromate together with 0.4 lb./ton of the above oil mixture. Cleaning of both lead and zinc concentrates is effected in separate series of clean-up cells. Complete flow-sheets and analyses of the various products obtained are included.

A. R. POWELL.

Development of the chlorine process of extraction of platinum metals from ores. R. A. COOPER and F. W. WATSON (J. Chem. Met. Min. Soc. S. Afr., 1929, 29, 220—228).—Froth flotation of South African norite ores effects an 87% recovery of the platinum metals in a sulphide concentrate weighing about 5% of the ore and assaying 7.5—10 oz./ton. After roasting to remove sulphur the concentrates are mixed with 15—20% of sodium chloride and heated at 500—600° in an atmosphere of chlorine for 4 hrs. The best results are obtained with a layer of ore 4—5 in. thick and without rabbling; the chlorine consumption is about 120—150 lb./ton. The product is only slightly caked, and is readily leached to give a solution containing over 90% of the platinum and 80% of the copper and nickel. The gold in the residue together with some of the remaining platinum and palladium may be recovered by cyanide leaching. The chloride solution is agitated with powdered limestone (20 lb./ton of solution) to precipitate copper carbonate containing about 2 oz./ton of platinum metals which may be recovered by smelting the precipitate to copper and electrolysis. The filtrate is agitated with 1 lb./ton of zinc dust and a little hydrochloric acid whereby a high-grade precipitate of platinum metals is obtained. The filtrate is treated with bleaching powder to recover nickel.

A. R. POWELL.

Röntgenographic study of the improvement of aluminium alloys. G. SHINODA (J. Jap. Min. Met.,

1928, 44, 514—562).—X-Ray studies were made of aluminium-silicon alloys to which was added potassium hydrogen fluoride, sodium fluoride, sodium peroxide, sodium carbonate, potassium carbonate, borax, or a calcium compound. The effect of the addition of sodium or calcium to aluminium-nickel alloys was also examined.

CHEMICAL ABSTRACTS.

Determination of the tin deposit on tin plates. F. PETER (Chem.-Ztg., 1929, 53, 438—439).—A piece of metal 100×200 mm. is cleaned in alcohol, dried, weighed, rolled into a coil, and dropped into boiling hydrochloric acid, *d* 1.08. When the tin coating has disappeared and the iron begins to be rapidly attacked, the coil is removed, washed first with warm water, then with alcohol, dried, and weighed. The loss in weight represents all the tin and some iron; the amount of the latter is determined in an aliquot part of the solution by oxidising with potassium chlorate, reducing with stannous chloride, and titrating with permanganate by the Zimmermann-Reinhardt method. The iron so found is increased by 0.4% to compensate for the impurities in the metal and the tin found by subtracting this weight from the loss in weight caused by the acid treatment. The results are correct to $\pm 1\%$.

A. R. POWELL.

Determination of sulphur in metallic antimony. C. G. SNYDER (Chemist-Analyst, 1929, 18, 6).—The sample (2—3 g.) is fused with sodium peroxide in a nickel crucible; the mass is extracted with water, and the solution heated until clear with concentrated hydrochloric acid (100 c.c.) and tartaric acid (10 g.). After dilution with hot water (400 c.c.), the solution is treated with barium chloride. CHEMICAL ABSTRACTS.

Rapid detection of tungsten in ores. A. PETROVSKY (Z. anal. Chem., 1929, 77, 268—269).—The finely-ground ore (0.1—0.2 g.) is boiled with 1—2 c.c. of concentrated hydrochloric acid and a small piece of lead foil for 1—2 min. A blue solution which on dilution with water gives a blue, flocculent precipitate indicates the presence of tungsten in excess of 0.5%. Niobium gives a similar blue solution, but the colour disappears on dilution.

A. R. POWELL.

Tensile strength and fracture of rolled strips of molybdenum. T. FUJIWARA (Mem. Coll. Sci. Kyoto, 1929, 12A, 165—178).—The tensile strengths of rolled strips of molybdenum have been investigated with reference to the direction of rolling. The tensile strength measured in the direction parallel to that of rolling is about 20% greater than when tension is applied at an angle of 45° or 90° to it. X-Ray examination of the fractures shows that they take place at the (211) and (110) planes of the cubic crystals of molybdenum.

C. W. GIBBY.

Hardness and abrasion testing of metals. G. A. HANKINS (Inst. Mech. Eng., June 30, 1929. Advance copy. 57 pp.).—A synopsis of the present state of knowledge of the subject, with special reference to the work done during 1921—27. Suggestions for further work are included.

Corrosion and metal protection in gasworks' practice. MAAS.—See II. Developers and metal tanks. ROSS and CRABTREE.—See XXI.

PATENTS.

Cupola furnace. J. ROBINSON (B.P. 312,401, 27.2.28).—A cupola furnace of the ordinary cylindrical type is provided with an offset chimney in its upper part and a central charging opening at the top of the shaft, which is closed with a plug which acts as a counterbalance to a charging bucket or elevator, and is so arranged that it is automatically removed as the bucket moves over the charging opening and replaced as the empty bucket swings away.

A. R. POWELL.

Furnace for melting metals. A. W. MACHLET (U.S.P. 1,713,543, 21.5.29. Appl., 15.4.26).—The furnace comprises a melting chamber in which a crucible is supported so that it almost closes the upper aperture which leads into a preheating stack in which is a concentrically arranged charging column for the crucible terminating in a feed hopper.

A. R. POWELL.

Condenser for zinc vapours from electric furnaces. F. THARALDSEN (U.S.P. 1,715,960, 4.6.29. Appl., 26.6.23. Norw., 3.11.21).—A block of deep, relatively narrow, separate, parallel channels is used, which are spaced apart and inclined downwards from the furnace chamber to a common container for collecting the products of condensation.

F. G. CROSSE.

Briquetting or consolidation of sponge-iron granules. F. L. DUFFIELD (B.P. 312,834, 25.6.28).—To obtain iron from ores containing large amounts of sulphur and phosphorus compounds, the ore, mixed with carbonaceous material and with or without lime, is passed through a reducing chamber whereby sponge-iron granules are obtained containing sulphur and phosphorus as impurities; the granules are then mixed with lime or limestone, pressed into briquettes, and melted, whereby the impurities are removed.

M. E. NOTTAGE.

Production of wrought iron. A. J. BRIGGS, ASSR. to ONONDAGA STEEL CO., INC. (U.S.P. 1,713,887, 21.5.29. Appl., 8.12.26).—Discrete particles of iron are mixed with slag-forming materials in the proportion to produce the desired fibrous structure of the iron, and the mixture is heated to a welding temperature in a rotary electric furnace from which air is excluded.

F. G. CROSSE.

Solder for cast iron and other metals for surfacing them. J. BUTLER, ASSR. to I. P. WALTON (U.S.P. 1,697,714, 1.1.29. Appl., 30.12.27).—A melt of 3 pts. of ground steel, 13.5 pts. of lead, and 3.5 pts. of tin is used as a solder for cast iron in conjunction with a flux consisting of 1 pt. of steel, 1 pt. of emery, 1.5 pts. of Tobin bronze, 1 pt. of plaster of Paris, 1 pt. of borax, 0.5 pt. of Epsom salts, and 0.5 pt. of salt.

R. BRIGHTMAN.

Treatment of silicon steel. A. F. MURPHY and W. JONES, ASSRS. to AMER. ROLLING MILL CO. (U.S.P. 1,714,038, 21.5.29. Appl., 15.4.27).—In the preparation of silicon-steel sheets for electrical purposes, the sheets are pickled, washed to remove acid, annealed, cold-rolled, and finally re-annealed.

F. G. CROSSE.

Ferrous alloy. L. W. MALLASEE, ASSR. to W. H. MALLASEE (U.S.P. 1,714,177, 21.5.29. Appl., 2.9.27).—Tin and aluminium are added to the molten ferrous metal at a pouring temperature.

F. G. CROSSE.

Manufacture of nickel-iron alloys. GEN. ELECTRIC Co., LTD., and R. W. W. SANDERSON (B.P. 312,411, 29.3.28).—Nickel and iron in a friable condition are simultaneously deposited electrolytically from a solution containing compounds of the two metals, a high current density being employed. Thus the metals may be deposited from a solution containing, per litre, 120 g. of nickel sulphate, 31 g. of ferrous sulphate, 13 g. of ammonium chloride, 6 g. of boric acid, 5 g. of quinol, and 2 g. of ammonium citrate, using two anodes, respectively, of iron and nickel, and a current density of 40 amp. per sq. in. of cathode. The magnetic properties of the deposited material, consisting of a mixture of nickel, iron, and nickel-iron alloy, are developed by heat treatment of the material in an inert gas.

J. S. G. THOMAS.

Alloy. G. G. MARSHALL and H. S. BOOTH (U.S.P. 1,713,766, 21.5.29. Appl., 27.2.23).—The alloy contains 2–12% Be, 46–90% Fe, and 4–42% Cr.

H. ROYAL-DAWSON.

Vanadium alloy. B. D. SAKLATWALLA, ASST. to VANADIUM CORP. OF AMERICA (U.S.P. 1,715,867, 4.6.29. Appl., 2.2.28).—A ferrovanadium alloy containing 85–95% V is claimed.

H. ROYAL-DAWSON.

Alloys and their manufacture. P. C. CHESTERFIELD, ASST. to CHESTERFIELD METAL CO. (U.S.P. 1,698,934 and 1,698,936, 15.1.29. Appl., 1.12.24).—(A) Alloys for high-speed cutting tools, which consist essentially of carbide crystals in a matrix of nickel and cobalt, are obtained by melting 10–45% of cobalt, 7–30% of nickel, and 0.5–3.5% of carbon, preferably added as carbide, with 25–35% of chromium and 15–35% of tungsten, the total amount of nickel and cobalt being between 30 and 70%. (B) The tungsten is replaced by 4–30% of vanadium or other metal, m.p. 1000–2100°, e.g., titanium or niobium, which forms hard carbides; the alloy may contain 15–55% Co, 7–30% Ni, 20–45% Cr, 10–25% V, and 0.5–3.5% C.

R. BRIGHTMAN.

High-speed alloy. P. C. CHESTERFIELD, ASST. to CHESTERFIELD METAL CO. (U.S.P. 1,698,935, 15.1.29. Appl., 1.12.24).—The alloy comprises 15–50% Co, 7–30% Ni, 20–45% Cr, 7–35% Mo, and a small amount of carbon.

F. G. CROSSE.

Metallic alloy. H. L. COLES and J. G. DONALDSON, ASSRS. to GUARDIAN METALS CO. (U.S.P. 1,698,212, 8.1.29. Appl., 5.8.24).—Heat- and oxidation-resistant alloy containing upwards of 60% W or a metal of the tungsten group, 10–25% Ni, and 2–5% C, obtained, e.g., by fluxing with calcium sulphate and fluoride a mixture of nickel sulphide or molybdenite and tungsten concentrates, is encased in copper or other metal of higher heat conductivity.

R. BRIGHTMAN.

Manufacture of alloys. W. M. GROSVENOR and V. P. GERSHON (U.S.P. 1,716,050, 4.6.29. Appl., 3.2.25).—The alloy contains about 20% of zinc, 70% or more of nickel, and up to 9% of copper.

F. G. CROSSE.

Metal [copper alloy]. F. J. READ, ASST. to CALAVERAS IRON & STEEL CO., and S. H. DEMAREST (U.S.P. 1,714,729, 28.5.29. Appl., 28.9.27).—The alloy contains 0.1–1% Ce and 5–10% Al, the remainder being chiefly copper.

F. G. CROSSE.

[Anti-friction metal] alloy. D. C. LEE (U.S.P. 1,714,679, 28.5.29. Appl., 17.11.26).—An alloy (4:1) of brass and Babbitt metal is claimed.

H. ROYAL-DAWSON.

Soldering of aluminium. P. ODAM (B.P. 300,969, 21.11.28. Fr., 21.11.27).—The solder consists of an aluminium alloy containing 5–12% Si, and is mechanically treated in the hot condition by working it into wires, rods, or thin plates; subsequent separation of silicon when melting the alloy is thereby avoided.

M. E. NOTTAGE.

Treatment of tin-bearing materials. D., M., S. R., and S. GUGGENHEIM, J. K. MACGOWAN, and E. A. C. SMITH (GUGGENHEIM BROS.) (B.P. 306,108, 30.5.28. U.S., 16.2.28).—Impure tin concentrates are ground to pass 200-mesh and, with or without a preliminary roast, are agitated with sulphuric acid, *d* 1.45, at 200° to remove iron, copper, etc. The residue is washed and digested with 20% sodium chloride solution to remove lead and silver, then with an acid sodium chloride solution to dissolve bismuth and any remaining arsenic and antimony. Tungstic acid is removed by leaching with 5% sodium hydroxide solution, and the purified cassiterite is smelted in an electric furnace with coal, sodium carbonate, and lime to produce metallic tin and a slag having a composition corresponding with that of the eutectic of Na₂SiO₃ and CaSiO₃. Owing to its low iron content, this slag is relatively free from tin, and the metal contains very little hardhead. The latter is removed by centrifuging the tin at 300°, and, after roasting at 600–700° until completely oxidised, is returned to the leaching circuit with further quantities of concentrate.

A. R. POWELL.

Flotation agents. E. C. R. MARKS. From AMER. CYANAMID CO. (B.P. 310,186, 26.4.28).—The dithiophosphates obtainable by the action of phosphorus pentasulphide on alcohols or phenols, e.g., on *isopropyl* alcohol or crude cresol, are used as flotation agents especially for extraction of sulphide ores. C. HOLLINS.

Controlling the action of pickling acids on metals. J. H. GRAVELL and A. DOUTY (B.P. 287,912, 28.3.28. U.S., 29.3.27).—A substance, e.g., a thiocyanate of an alkali metal or ammonium, which will combine with the nascent hydrogen, is added to the bath so that hydrocyanic acid is liberated within the bath. A foam-producing material, e.g., cellulose pulp waste liquor, evaporated or otherwise, is also added to prevent the liberation of acid spray into the air.

M. E. NOTTAGE.

[Metallically] coating metal. F. SMITH, ASST. to AMER. MACHINE & FOUNDRY CO. (U.S.P. 1,710,747, 30.4.29. Appl., 17.12.25).—Vaporised metal is ionised and electrically deposited and condensed on the metal to be coated.

J. S. G. THOMAS.

Electrolytic production of metals and apparatus therefor. I. G. FARBERIND. A.-G. (B.P. 285,824, 21.2.28. Ger., 21.2.27).—Electrolyte is caused to flow through the bath from below upwards, and the outflow is distributed along opposite sides of the cell so that movement of the electrolyte is substantially uniform with respect to all the electrodes.

J. S. G. THOMAS.

Electrodeposition of metals. ELECTRO BLEACH & BY-PRODUCTS, LTD., J. HOLLINS, and D. JEPSON (B.P. 312,395 and 312,403, 25.2.28).—(A) Anodic material is supported at the base of the anode compartment by glass or similar balls which are unaffected by the electrolysis, and provide spaces through which the sludge produced percolates. The balls are supported on a grid around which air under pressure is injected. (B) Anodic plates or strips are enclosed within graphite-impregnated sleeves or covers protecting them from the abrasive action of anodic material.

J. S. G. THOMAS.

Purification of magnesium and its alloys. G. MICHEL, Assr. to H. O. BERG (U.S.P. 1,698,647, 8.1.29. Appl., 5.2.25. Fr., 4.10.24).—See B.P. 261,528; B., 1927, 80.

Melting furnace (U.S.P. 1,715,678). **Magnetic material** (U.S.P. 1,715,541 and 1,715,543).—See XI.

XI.—ELECTROTECHNICS.

Durability of the structure of direct-arc furnaces under different working conditions. E. KOTHNY (Feuerfest, 1929, 5, 1—3, 75—80).—Data were collected by means of a questionnaire on the efficiency and durability of one acid and twenty-three basic arc furnaces operating under varying industrial conditions. In all cases the furnace roofs were constructed of silica bricks, the normal thickness being 250 mm. for furnaces up to 7.5 tons capacity. The method of setting up (wet or dry) had no effect on the durability of the roof. In most cases the thickness of the insulating layer in the hearth walls ranged from 100 to 125 mm.; the thickness of the whole wall varied considerably. Basic, tamped walls were usually more durable than walls made of bricks. Pauses during the working day had an unfavourable effect on the hearth walls. Insulation of the furnace hearth varied from 50 to 240 mm. in thickness. In nearly all basic furnaces the hearth proper was made of magnesite bricks covered with a magnesite or dolomite tamping mixture. No direct relation was found between the durability of the hearth and its total thickness or the proportion of brick and tamped material. The data collected indicated that, in general, the durability of the roof, hearth wall, and hearth of basic arc furnaces depends mainly on the quality of the refractory material used and the care and attention applied in the construction and maintenance of the furnace.

F. SALT.

Electrical heating by the container-resistance method. R. A. CARLETON (Ind. Eng. Chem., 1929, 21, 525—529).—The method of heating in which an electric current of high amperage at low voltage is passed through the walls of the vessel to be heated can be applied to large numbers of processes, *e.g.*, heating of drums, rotary roasters, ovens, pipes, and tubing used in the transfer of viscous liquids. It may be applied in nearly all processes in which the container used is a good electrical conductor. A scheme is suggested for using the method in the varnish industry. The costs and efficiency of conversion of the heat energy derived from coke, oil, and gas are compared with those of the container-resistance method of heating.

The chief advantages of the method are great flexibility automatic temperature control, even heating, and its ready application to vessels of different shapes.

H. INGLESON.

Cathode rays and cable deterioration. SCHOEFFLE and CONNELL.—See II. **Light sources for weathering systems.** SCHMUTZ and GAMBLE.—See XIII.

PATENTS.

Electric furnace. F. A. J. FITZGERALD, Assr. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,714,081, 21.5.29. Appl., 13.5.27).—A resistor chamber containing an inert atmosphere, and a heat-treating chamber containing a gas having a pyrochemical and physical action on substances treated, are separated by a septum which is unaffected during operation of the furnace.

J. S. G. THOMAS.

[Electrical] melting furnace. S. SCHNEIDER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,715,678, 4.6.29. Appl., 13.9.26. Ger., 14.12.25).—Gas under pressure is supplied to an electrically-heated melting pot having a removable cover in which is mounted the upper end of a discharge conduit extending through the cover from near the bottom of the pot. A recess in a second cover member is adapted to receive the first cover, and a discharge passage in this member receives the upper end of the discharge conduit.

J. S. G. THOMAS.

Electric dry cell. H. SHIMIDZU (B.P. 312,828, 19.6.28).—The whole operative content of the cell is contained and maintained automatically in compression within a vulcanised india-rubber casing. Excess of electrolyte is removed by pressure. J. S. G. THOMAS.

Diaphragms and the like for electrolytic cells. DR. A. WACKER GES. F. ELECTROCHEM. IND. G.M.B.H., and H. MÜLLER (B.P. 312,713, 9.3.28).—Finely-divided, slightly soluble material, *e.g.*, barium sulphate, quartz, glass, purified slag powder, corundum, asbestos, mixed with a glutinous binder, *e.g.*, solutions, emulsions, or suspensions of rubber, gutta-percha, balata, cellulose, is spread in a thin layer on a carrier, *e.g.*, of metal or fabric gauze, and dried at atmospheric temperature.

J. S. G. THOMAS.

Manufacture of electrolytes. R. SCHUSTER (U.S.P. 1,695,667, 18.12.28. Appl., 13.11.23. Renewed 8.8.28. Ger., 22.7.25).—A solution of sodium dichromate and sulphuric acid to which a soluble silicate has been added is evaporated in a revolving drum, and the dry product is sealed in air-tight containers for storage.

C. HOLLINS.

Electrolytic production of sterilising agents, germicides, etc. [*e.g.*, hypochlorites, chloroamines, etc.], and application of such agents. UNITED WATER SOFTENERS, LTD., and E. B. HIGGINS (B.P. 311,218 and 311,253, [A, B] 6.2.28, [B] 30.11.28).—(A) Solution to be electrolysed, *e.g.*, an aqueous solution of sodium chloride or ammonium chloride, flows from one or both electrodes arranged so that the distance between them is small relatively to the cross-section of either electrode. If desired, electrolyte flowing through the anode may flush away products of electrolysis from the cathode. (B) An aqueous solution of an alkali chloride, *e.g.*,

sodium chloride, is electrolysed, as described, in the presence of ammonia or a soluble ammonium salt.

J. S. G. THOMAS.

[Stabilising the output of a tantalum-lead-sulphuric acid] electrolytic rectifier. C. W. BALKE, ASSR. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,710,806, 30.4.29. Appl., 18.7.27).—The current density is controlled at about 1 amp. per 0.5–1.5 in.² of active tantalum surface exposed.

J. S. G. THOMAS.

Manufacture of storage battery plate. L. J. PEARSON, ASSR. to PHILADELPHIA STORAGE BATTERY CO. (U.S.P. 1,716,320, 4.6.29. Appl., 22.5.20).—A predetermined volume of air is circulated over storage battery negative plates containing metallic lead and sulphuric acid, and is then dried and heated prior to acting again on the plates.

J. S. G. THOMAS.

Manufacture of secondary battery plates. F. GARACA, ASSR. to LUTHY RES. LAB. (U.S.P. 1,713,825, 21.5.29. Appl., 18.11.27).—A paste consisting of sulphated active material and a dilute solution of sodium acetate is applied to supporting grids, and sulphates and acetates are removed from the paste before the forming process.

J. S. G. THOMAS.

[Filling for plates of] electric accumulators. B. HEAP, and CHLORIDE ELECTRICAL STORAGE CO., LTD. (B.P. 312,851, 26.7.28).—Finely-divided barium sulphate is produced throughout the active material of storage battery negative plates by the action of sulphuric acid on a lead-barium alloy or on barium hydroxide solution.

J. S. G. THOMAS.

Manufacture of Röntgen ray fluorescent screens. C. HAIDER (B.P. 297,037, 11.9.28. Ger., 12.9.27).—A substance which fluoresces under the action of Röntgen rays, *e.g.*, zinc sulphide, calc spar, magnesite, is spread between two flat plates, *e.g.*, of celluloid; the pressure of air between the plates is then reduced and the edges of the plates are cemented together.

J. S. G. THOMAS.

Production of electric radiating elements, particularly electron-emitting elements for electric discharge devices. E. HARSANYI (B.P. 287,098, 14.3.28. Ger., 14.3.27).—A metal, metallic oxide, or other compound is deposited upon a metallic body, *e.g.*, a roughened platinum wire, by electrophoresis from a colloidal suspension, electrolytic decomposition of the bath being prevented during the process by employing a sufficiently low voltage. On glowing, the deposited material yields a metallic oxide.

J. S. G. THOMAS.

Light-sensitive discharge device. [Photoelectric cells.] ELECTRICAL RES. PRODUCTS, INC., ASSEES. OF G. R. STILWELL (B.P. 288,539, 14.1.28. U.S., 6.4.27).—Light-sensitive material, *e.g.*, potassium, deposited upon a specially or critically positioned auxiliary member, *e.g.*, an axial, re-entrant, tubular, closed stem, within a tube, is revaporised and redeposited upon the inner surface of the outer walls of the cell. The method is applicable to the construction of photoelectric cells.

J. S. G. THOMAS.

Effecting chemical reactions in [hydrocarbon] gases by means of electrical discharges. P. H. HULL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P.

311,352, 10.1.28).—Hydrocarbon gas, *e.g.*, methane, is supplied to the arc in a number of adjustable streams arranged so that the arc is distended solely by one stream. If desired, the supplementary gas streams supplying gas to the arc may have a higher velocity than the primary stream, and may impinge on one another to produce a broad, flat disc of gas, or may impart a whirling motion to the gas.

J. S. G. THOMAS.

Manufacture of electric lamps, discharge tubes, and the like. A. LIERENFELD (B.P. 312,659, 27.2.28).—The lamp filament, during pretreatment, is subjected to the action of an intermittent electric current, traversing the filament in the opposite direction to the direction of movement of the filament through the pretreatment zone.

J. S. G. THOMAS.

Electric incandescence lamp. F. ECKHARDT and M. HOHNEKAMP (U.S.P. 1,713,752, 21.5.29. Appl., 5.5.27. Ger., 27.9.26).—A shunt composed of a sulphide ore, which is non-conducting when the normal operating voltage of the lamp is applied to it, but which conducts when a higher voltage is applied, is permanently connected as a shunt to the incandescence filament of the lamp.

J. S. G. THOMAS.

Manufacture of arc lamp electrodes. COMP. LORRAINE DE CHARBONS POUR L'ELECTRICITE (B.P. 289,042, 20.3.28. Fr., 21.4.27).—In order to stabilise the arc flame, an amount of non-oxidised or very slightly oxidised silicon, boron, or zirconium, up to about 2%, is incorporated in an arc lamp core containing carbon, rare earths, and alkali metals, or alkaline earths. The percentage of silicon, boron, or zirconium in the shell may be slightly increased to compensate for loss by lateral combustion.

J. S. G. THOMAS.

Electrical condenser. S. RUBEN (U.S.P. 1,715,789, 4.6.29. Appl., 10.10.24).—The space between aluminium electrodes coated with aluminium sulphide contains cupric sulphide making contact with the aluminium sulphide and supplying free sulphur ions under electric pressure.

J. S. G. THOMAS.

Magnetic material. Magnetic core. G. W. ELMEN, ASSR. to BELL TELEPHONE LABS., INC. (U.S.P. 1,715,541 and 1,715,543, 4.6.29. Appl., [A] 19.9.27, [B] 20.6.28).—(A) A magnetic material of very constant permeability over a range of magnetising forces including that employed in continuous loading of electrical signalling conductors contains iron, nickel, and cobalt, and at least one of the elements molybdenum, chromium, tungsten, manganese, vanadium, tantalum, zirconium, copper, and silicon to increase its resistivity. (B) A loading coil comprises a core composed principally of material containing 8–80% Ni, 5–80% Co, and 10–45% Fe.

J. S. G. THOMAS.

Manufacture of cores for electromagnets or the like. GEN. ELECTRIC CO., LTD., W. SINGLETON, and G. C. MARRIS (B.P. 309,394, 9.1.28).—Cores for electromagnets, loading coils, etc. are made of magnetic material, *e.g.*, powdered or laminated iron or nickel-iron alloy, and coated with silicon or material containing silicon. Thus the magnetic material may be heated in a stream of an inert gas, *e.g.*, nitrogen, and silicon tetrachloride.

J. S. G. THOMAS.

Treatment of substances used for impregnation of cables. FELTEN & GUILLEAUME CARLSWERK A.-G. (B.P. 308,748, 5.6.28. Ger., 30.3.28).—The impregnating substance is treated under vacuum or in a chemically inert atmosphere, e.g., nitrogen or carbon dioxide, during the mixing, boiling, and impregnation so that an airtight covering preventing oxidation of the impregnator is formed on the cable. J. S. G. THOMAS.

Non-corrodible battery terminal. C. T. HIXSON, Assr. to W. M. CAMPBELL and H. E. PISCHEL (U.S.P. 1,715,599, 4.6.29. Appl., 5.7.27).—Powdered glass is stirred into a molten mass of lead and antimony at red heat, and the whole is moulded. F. G. CROSSE.

Manufacture of moulded bodies [dynamo brushes] from carbon or mixtures of carbon and metal, with metal insertions. K. F. and H. VON SIEMENS and A. FRANKE (GEER. SIEMENS & Co.) (B.P. 285,081, 7.2.28. Ger., 11.2.27).

Nickel-iron alloys (B.P. 312,411). Coating of metal (U.S.P. 1,710,747). Electrolytic production of metals (B.P. 285,824, 312,395, and 312,403).—See X.

XII.—FATS; OILS; WAXES.

The butyro-refractometer. F. BOLM (Z. Unters. Lebensm., 1929, 57, 91—93).—The liquids supplied for standardisation of the Zeiss butyro-refractometer vary in refractive index, and care must be taken to use the table referring to the particular standard liquid used. The n value of the liquid varies with age, so that a standard is not usable after a period of a year. The value of n in absolute Zeiss degrees can readily be found by means of the special thermometer. A table for converting Zeiss degrees into n values, and a formula for calculating n for any given temperature are provided. W. J. BOYD.

"Values" of fat from preserved-milk products and its mixtures with cacao butter. H. FINCKE (Z. Unters. Lebensm., 1929, 57, 9—13).—The "values" of mixtures of cacao butter with milk fat, i.e., the saponification and iodine values and the m.p. of the fats and fatty acids, agree sufficiently with the values calculated from the actual milk fat contents and from the milk fat contents derived from the Reichert-Meissl values to permit conclusions to be drawn as to the presence or absence of foreign fats. W. J. BOYD.

Luminescence of sound Dutch lard in ultra-violet light. A. VAN DRUTEN (Z. Unters. Lebensm., 1929, 57, 60—62).—Contrary to hitherto accepted explanations of luminescence phenomena in lard it is shown that perfectly sound Dutch lard rendered on the water-bath at a temperature not above 60° may show blue or blue-violet fluorescence. It is therefore not permissible to draw definite conclusions from such phenomena as to the nature, origin, and method of manufacture of the product. W. J. BOYD.

Determination of detergency of soap products. L. T. HOWELLS (Oil & Fat Ind., 1929, 6, [6], 23—29).—The Detergents Sub-Committee of the American Oil Chemists' Society report on the experimental machines designed for laboratory testing of the detergency of soaps.

The standard washing tests proposed proved not altogether satisfactory, the readings being less consistent and the end-points (number of washings required for complete removal of soil) more drawn-out than in practical laundry tests. It is clearly indicated that the last traces of soil are the most difficult to remove; testing procedure must be modified to accomplish this in a reasonable time. E. LEWKOWITSCH.

Application of the hydrogen value to unsaturated fatty acids. H. I. WATERMAN, S. II. BERTRAM, and H. A. VAN WESTEN (J.S.C.I., 1929, 48, 50—51 r).—The hydrogen values of elaidic, linoleic, and stearolic acids were determined by the method described previously (B., 1929, 102); the results obtained from the first-named are considered as standard and confirm the accuracy of the method. Δ^9 -¹²-Linoleic acid was shown to contain two ethylenic linkings, 2 mols. of hydrogen being absorbed in the conversion into stearic acid. Stearolic acid, which behaves towards thiocyanogen as though fully saturated, and absorbs 1 mol. of iodine, also absorbs 2 mols. of hydrogen to yield stearic acid, proving the presence of a triple linking. E. LEWKOWITSCH.

Composition of α -elæostearic acid, the most important component of Chinese wood (tung) oil. J. BÖESEKEN (J.S.C.I., 1929, 48, 71—72 r).—The author criticises the quotations of the work of himself and others on the constitution of elæostearic acid made by Steger and van Loon (B., 1929, 103). It is noted that Böeseken and Hoogland (A., 1928, 1169) demonstrated that the ethyl ester absorbed exactly 3 mols. of hydrogen, and the work establishing the constitution of elæostearic acid as $\text{CH}_3 \cdot [\text{CH}_2]_3 \cdot [\text{CH}:\text{CH}]_3 \cdot [\text{CH}_2]_7 \cdot \text{CO}_2\text{H}$ is reviewed. E. LEWKOWITSCH.

Unsaturated fatty acids of chrysalis oil. W. KIMURA (Chem. Umschau, 1929, 36, 185—190).—The oily bromides, insoluble in light petroleum, obtained from the unsaturated fatty acids (separated by the lead salt-alcohol method) of chrysalis oil are shown to consist principally of tetrabromolinoleic acid, admixed with small amounts of liquid bromides of linolenic and oleic acids: not more than 60% of hexabromostearic acid was obtained. The composition of the liquid unsaturated fatty acids of chrysalis oil is computed as 21.3% of linolenic, 48.9% of linoleic, and 29.8% of oleic acids. Oxidation with alkaline permanganate yielded linusic (m.p. 204°), isolinusic (m.p. 174°), dihydroxystearic (m.p. 132°), and two sativic (m.p. 156°, 173°) acids. E. LEWKOWITSCH.

Preparation of neutralised olive oil. M. MALMY (J. Pharm. Chim., 1929, [viii], 9, 521—524).—The difficulties occasioned in the technical production of neutral olive oil and suggested modifications of the official specifications are outlined. E. H. SHARPLES.

Fachini's reaction for detection of "residue" olive oils. R. MARCILLE (Ann. Falsif., 1929, 22, 163—166).—Fachini's reaction with acetic anhydride (B., 1926, 592) gives a strong cherry-red colour with olive residue oils; with the finer oils the colour is paler, and it is shown that a dark coloration may be due to the quality of the oil being tested, and may not necessarily indicate admixture with residue oil. Assuming that all

olive oils give some colour, and that kernel oils do not, Fachini's reaction is useful for identifying olive oil in mixtures, and also for determining whether or not olive oils of low acidity contain refined inferior oils.

D. G. HEWER.

Palm oil from the Belgian Congo. G. S. JAMIESON and R. S. MCKINNEY (Oil & Fat Ind., 1929, 6, [6], 15—17).—The oil, on analysis by the lead salt-ether separation method, followed by fractionation of the methyl esters, had d_{25}^{25} 0.9146, n_D^{20} 1.4578, acid value 20.65, saponif. value 197.9, unsaponif. matter 0.39%, iodine value (Hanus) 53.7, acetyl value (André-Cook) 15.27, Reichert-Meissl value 0.10, Polenske value 0.29, saturated acids (corr.) 44.3%, unsaturated acids (corr.) 55.6%, iodine value of unsaturated acids 99.9. It contained the glycerides of oleic (47.2%), linoleic (5.6%), myristic (0.5%), palmitic (40.8%), stearic (5.2%), and lignoceric (0.1%) acids, and 0.39% of unsaponifiable matter. Lignoceric acid is thus reported as a constituent of palm oil for the first time. E. LEWKOWITSCH.

Cacao oil. BODINUS (Pharm. Ztg., 1929, 74, 647—648).—Samples of fat extracted from cocoa dust, a by-product in the manufacture of cocoa and chocolate, and sold as pure cacao butter, have inferior colour and odour, a lower m.p., and higher iodine value and refractive index than normal cacao butter. The differences are due to the presence of about 1.6% of the fat extracted from the pods, which yield about 2% of a pasty fat of unpleasant odour, and very high acidity, refractive index, and iodine value. S. I. LEVY.

Oil from seeds of *Erucastrum elongatum*. N. BELIAIEV (Oil Fat Ind. [Russia], 1928, No. 8, 26—28; Chem. Zentr., 1929, i, 166—167).—The oil has acid value 4.52, iodine value (Hübl) 116.3, saponif. value 176.02. It is a semi-drying oil. A. A. ELDRIDGE.

Hydrogenation of oils. H. I. WATERMAN and S. H. BERTRAM (J.S.C.I., 1929, 48, 79—80 T).—Soya-bean oil was hydrogenated in the presence of a nickel-kieselguhr catalyst; in contrast to the results of similar experiments by Kaufmann and Hansen-Schmidt (B., 1927, 226) on sunflower-seed and arachis oils, it was found that the decrease in iodine value (76%) was accompanied by a decrease (35%) in the thiocyanogen value, although the hydrogenation of those unsaturated compounds, which are not saturated by thiocyanogen, but are saturated by iodine chloride, predominates. E. LEWKOWITSCH.

Influence of temperature on hydrogenation [of oils]. A. MARKMAN and V. VASSILIEV (Oil Fat Ind. [Russia], 1928, No. 8, 23—24; Chem. Zentr., 1929, i, 166).—The hydrogenation of oils is reversible. For sunflower oil the optimum temperature is 250—270°.

A. A. ELDRIDGE.

Tung oil. Possibilities of production within the British Empire, with a bibliography. L. A. JORDAN (J. Oil & Col. Chem. Assoc., 1929, 12, 113—153).

Colloid-chemical changes in fatty oils. AUER.—See XIV.

PATENTS.

Production of neutral fats and oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,523, 2.7.28).—Fats and oils containing free fatty acids are treated at

100—200° with ethylene oxide or its homologues. The process is accelerated by working under pressure or by the use of catalysts, e.g., metal acetates or oxides such as titanium oxide. E. LEWKOWITSCH.

Manufacture of water-soluble or emulsifiable products from wool fat. I. G. FARBENIND. A.-G. (B.P. 286,252, 29.2.28. Ger., 1.3.27).—Semi-solid, water-soluble or emulsifiable products are obtained by sulphonating wool fat (or its fatty acids) in the presence of a phenol. Emulsions of these compounds are stable even in acid solution, form clear solutions on the addition of alkali, but are salted out by the addition of neutral salts. E. LEWKOWITSCH.

Manufacture of soap and saponaceous materials. J. Y. JOHNSON. From FARBENIND. A.-G. (B.P. 312,405, 8.6.28).—The oxidation products of paraffin hydrocarbons are saponified with about the theoretical amount of concentrated caustic alkali and the solution is cooled to 10—5°, causing the unsaponifiable matter (about 20%) to separate. E. LEWKOWITSCH.

Extraction of oils. J. W. BECKMAN (U.S.P. 1,698,294, 8.1.29. Appl., 19.5.24).—Animal or vegetable material containing oil is macerated with water and treated with a culture of lactic acid bacteria at 50°, preferably in darkness or shadow and with at least partial exclusion of air. The lactic acid produced is periodically neutralised, and when the cellular structure is destroyed the mixture is diluted with water and the oil separated. Addition of 10% of sodium chloride solution accelerates the decomposition. E.g., 1000 pts. of macerated coconut (copra) are treated for 100—125 hrs. with 1 pt. of malt and 1 pt. of magnesium carbonate at 50°, neutrality being maintained by further additions of magnesium carbonate as required. R. BRIGHTMAN.

Extraction of oil from fish liver. W. P. WILLIAMS. From SOC. FRANÇ. DES PROD. ALIMENTAIRES AZOTÉS Soc. ANON. (B.P. 312,768, 20.4.28).—Fresh or salted fish livers are pulverised and subjected to autolysis for 10—24 hrs. at 42—45° until a semi-liquid mass is obtained from which the bulk of the oil, which is pure and of good odour, may be decanted; the remaining oil is removed by filtration or centrifuging, the residue constituting a rich degreas for leather dressing.

E. LEWKOWITSCH.

Manufacture of emulsions. E. C. R. MARKS. From CHEM. FABR. STOCKHAUSEN & Co. (B.P. 312,799, 24.5.28).—Stable oil emulsions are produced by the aid of the sulphuric acid compounds of fats or fatty acids described in B.P. 293,480 and 293,717 (B., 1928, 678, 718) as emulsifying agents. E. LEWKOWITSCH.

Manufacture of hair toilet oil. Y. SHIMURA and K. TAKAGI (B.P. 312,568, 23.10.28).—Non-drying vegetable oils (castor, tea-seed, etc.) are agitated and warmed with equal quantities of ethyl or methyl alcohol and a small quantity of ether for a considerable time (e.g., 7 days); the product on being washed and distilled under reduced pressure yields a pale non-oxidising oil of low viscosity. E. LEWKOWITSCH.

Extraction of candelilla wax. J. T. GARCIA (U.S.P. 1,715,194, 28.5.29. Appl., 15.9.25. Mexico, 30.10.24).—The candelilla plant is crushed and subjected

to a tearing and beating action followed by screening to separate the wax and non-waxy matter from the plant fibres.

E. LEWKOWITSCH.

Wax-like chlorinated hydrocarbons (B.P. 309,421).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Microstructure of paint films and proof of internal stresses. A. V. BLOM (Farben-Ztg., 1929, 34, 2127—2130).—Transverse sections were cut from old paint films and examined under the microscope after suitable preparation—staining, etching with solvents, etc. A distinct laminated microstructure was observed, due to the alternation of layers relatively rich in pigment or fillers with layers rich in resins, but practically devoid of pigment: this segregation of pigment during the drying process cannot be due to gravity settlement as the paints examined were stripped from vertical surfaces. Good or defective adhesion of successive coatings is evidenced by smooth or irregular transverse fractures respectively. The considerable distortion produced in a red lead undercoating by a hard bituminous top-coat gave evidence of the magnitude of the stresses evoked during drying, and the conception of "compressibility" of a paint film is introduced in connexion with the development of stresses (and consequent breakdown) in the film by the shrinkage of the support with falling temperatures.

E. LEWKOWITSCH.

Testing [ageing] of paints. P. NETTMANN (Farben-Ztg., 1929, 34, 2181—2183).—The ageing of paints is discussed from a theoretical point of view on the basis of energy changes in the coating: it is suggested that, during ageing, and as a result of the various influencing factors (irradiation, temperature, etc.), the paint film may accumulate potential energy, which is chiefly responsible for the changes in the inner structure of the film, even after the lapse of years.

E. LEWKOWITSCH.

New accelerated [weathering] test for paints. W. H. DROSTE and M. WERNER (Farben-Ztg., 1929, 34, 2131—2133).—Accelerated durability tests for paints are discussed and criticised.

E. LEWKOWITSCH.

Destructive light sources for use in accelerated weathering systems. F. C. SCHMUTZ and D. L. GAMBLE (Ind. Eng. Chem. [Anal.], 1929, 1, 83—86).—The action of vigorous artificial light sources has been studied with mixtures of lithopones with lacquer or with house paints. The specimens were mounted on porcelain palettes and dried at 50°, and then subjected to rays from carbon and mercury arcs or to sunlight. The carbon arcs were operated so as to give accentuation of different parts of the spectrum, and it was found that intensification of the infra-red and visible part had little effect, but that in the far ultra-violet the differences between the pigments was lessened. Much information is summarised as to the operation of mercury arc lamps, particularly with a view of obtaining a constant total intensity of radiation, and the use of special glass filters is also discussed. The problem of comparison of these sources with natural sunlight is complicated by large variations in the intensity of the latter.

R. H. GRIFFITH.

Examination of pigments in ultra-violet light. M. J. SCHOEN and J. RINSE (Chem. Weekblad, 1929, 26, 321—322).—Pigment value cannot be determined from the fluorescence colours in the cases of lithopone and zinc white. Natural and precipitated chalk can, however, be distinguished, and the presence of zinc oxide (25%) in titanium white changes the violet fluorescence to greenish-yellow. Admixture of organic colouring matter in mineral pigments can usually be detected.

S. I. LEVY.

Pigment and oil. E. KLUMPP (Farben-Ztg., 1929, 34, 2130—2131).—The theoretical conclusion that oil absorption varies inversely with particle size is challenged: pigments that had been very thoroughly pulverised in a mortar gave very much lower values than those obtained for the raw pigment.

E. LEWKOWITSCH.

Determination of oil absorption of pigments. RUCHTI (Farben-Ztg., 1929, 34, 1954).—It is pointed out that oil absorption depends not only on the degree of subdivision or specific surface, but also on the chemical nature of the pigments; the oil absorption should only be taken as an analytical "value" of a pigment and the convenient Wolff method (cf. B., 1929, 103) is recommended to obtain reproducible results in technical practice.

E. LEWKOWITSCH.

Determination of arsenic in antimony oxide pigment. G. SIROIS (Chemist-Analyst, 1929, 18, 14).—Small quantities of arsenic are distilled off as trichloride and weighed as trisulphide. For larger quantities the pigment (1 g.) is treated with concentrated sulphuric acid (15 c.c.) and potassium sulphate (5 g.), the mixture is heated to eliminate sulphur, then cooled, diluted with 20 c.c. of water, heated to dissolve the salts, treated with hydrochloric acid (20 c.c.), and saturated with hydrogen sulphide. Treatment of the arsenic trisulphide with carbon disulphide is recommended.

CHEMICAL ABSTRACTS.

Thickening of oils for use in the varnish industry. C. DORN and J. BURDIN (Oil Fat Ind. [Russia], 1928, No. 7, 29—31; Chem. Zentr., 1928, ii, 2682).—Restricted action of sulphur monochloride on oil affords a suitable product; experiments with aluminium chloride were fruitless.

A. A. ELDRIDGE.

Enrichment of poor gums. M. TOMEO (Anal. Fis. Quím. [tecn.], 1929, 27, 77—106).—The effect of the addition of turpentine during the preparation, decantation, and distillation of various gums has been studied. With several classes of resins considerable advantage is to be gained by addition to the gum of about one half the quantity of turpentine, although the exact proportion varies with the material to be treated.

H. F. GILLBE.

Electrical heating [in varnish manufacture]. CARLETON.—See XI.

PATENTS.

Preparations for printing, painting, coating, or impregnating surfaces. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 311,795, 14.2.28).—The products comprise solutions of cellulose esters or ethers in water-soluble esters of formic acid and polyhydric alcohols, e.g., glycol mono- or di-formate, together with water and, if desired, dyes etc.

L. A. COLES.

Painting process. E. FRENKEL (H. FRENKEL) (B.P. 288,624, 16.3.28. Ger., 14.4.27).—Paints which dry throughout the mass, and thereby permit the application of successive coats before the preceding coats are dry, contain binding material consisting of fatty oils pre-treated with sulphur chloride, with or without the addition of high-boiling hydrocarbons, esters, amines, etc. to retard the time of drying. Ordinary oil paints, varnishes, nitrocellulose lacquers, etc. may be used for the final coat. L. A. COLES.

Manufacture of non-lead face powder or paint. M. FUKUI and T. MIYAGUCHI (B.P. 312,562, 3.10.28).—Zinc oxide, powdered talc, titanium oxide, starch, etc. is treated with a solution in a volatile solvent of transparent material insoluble in water, *e.g.*, nitrocellulose or caoutchouc, the solution being coloured, if desired, and the solvent removed by evaporation. L. A. COLES.

Manufacture of [white] pigments. J. B. PIERCE, JUN. (U.S.P. 1,715,384, 4.6.29. Appl., 10.11.24).—Hydrogen sulphide is passed into a solution containing 200—500 g. of zinc sulphate per litre and having precipitated barium sulphate or blanc fixe suspended in it. F. G. CROSSE.

Production of white lead. METALLBANK & METALLURGISCHES GES. A.-G., and G. SITZ (B.P. 311,986, 17.7.28).—Lead chloride, obtained, *e.g.*, by the digestion of waste plumbiferous material with hot sodium chloride solution and subsequent precipitation by cooling and/or dilution, is treated in aqueous suspension with sodium carbonate, sodium hydroxide and carbon dioxide, or sodium carbonate and hydroxide, these being added gradually so that the solution does not exhibit a definitely alkaline reaction until conversion of the lead chloride into basic carbonate is complete. L. A. COLES.

Preparation of printer's ink. R. and I. M. MACLAURIN (B.P. 312,745, 30.3.28).—The use is claimed of low-temperature or semi-low-temperature tars, and of products derived from them. The tar may be thickened by adding resinous constituents precipitated from other portions of the tar by the addition of petroleum oil and/or dilute acids, or thinned by the addition of the oil remaining after removal of the resins, or it may be treated with ferrous hydroxide and/or mixed with linseed oil, sodium hydroxide, pigments, etc. L. A. COLES.

Composition for lithographing ink. O. E. HARDER (U.S.P. 1,714,166, 21.5.29. Appl., 11.6.27).—A mixture of petrolatum oil, paraffin, and magnesium carbonate is used with printers' ink.

H. ROYAL-DAWSON.

[Non-explosive and fireproof] cellulose lacquers. INTERNAT. FIREPROOF PRODUCTS CORP., Assees. of F. S. VIVAS (B.P. 286,724, 8.3.28. U.S., 11.3.27).—Products are claimed comprising a solution of a gum and boric acid in a mixture of butyl and ethyl alcohol, a solution of nitrocellulose in a mixture of an aromatic hydrocarbon (toluol) and ethyl or butyl acetate, and a mixture of a vegetable oil, *e.g.*, castor oil, with alcoholic calcium chloride solution. A chlorinated hydrocarbon, *e.g.*, carbon tetrachloride, is added to each solution and to the mixture, and a mixture of alcohol and carbon tetrachloride may be used as diluent. L. A. COLES.

Driers for varnishes, lacquers, oil paints, etc.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,716, 14.1.28).—The use is claimed of metal compounds insoluble or sparingly soluble in water of crude or purified oxidation products of paraffin wax etc., alone or mixed with salts of high-molecular acids of natural origin, *e.g.*, resinates, linoleates, stearates, naphthenates, etc. L. A. COLES.

Application of lacquers containing cellulose esters. IMPERIAL CHEM. INDUSTRIES, LTD., N. STRAFORD, E. E. WALKER, and W. J. JENKINS (B.P. 312,204, 14.2.28).—The surfaces to be coated are primed with a lacquer containing a phenol-formaldehyde resole and a resin of the "glyptal" type, with or without the addition of an acid catalyst, *e.g.*, sulphuric acid, before applying the cellulose ester lacquer. L. A. COLES.

Removal of [non-vitreous] enamel. H. O. LANG (U.S.P. 1,714,879, 28.5.29. Appl., 19.5.24).—The enamelled metal article is immersed in a bath of molten material consisting chiefly of an alkali salt of nitric or nitrous acids. H. ROYAL-DAWSON.

Manufacture of pure colophony and other resinous products from resinous plants or woods. D. GARDNER (B.P. 289,774, 23.4.28. Fr., 30.4.27).—Wood etc., after removal of volatile constituents by treatment with steam, preferably at 1 atm. pressure, followed by drying, is treated in a finely-divided form with sodium hydroxide solution, *d* 1.015—1.02, in the presence of a reducing agent, *e.g.*, sodium bisulphite or hyposulphite, and an oleate. The treatment may be effected at about 30° in a colloid mill, or, when the wood is not so finely divided, at 75—80° in closed apparatus. After removal of the cellulose by filtration, the solution is acidified to precipitate colophony, which is removed and treated with a solvent, *e.g.*, turpentine. L. A. COLES.

Refining of rosin. HERCULES POWDER Co., Assees. of A. LANGMEIER (B.P. 298,214, 10.1.28. U.S., 6.10.27).—A high-grade rosin, suitable for soap and varnish making or for white paper sizes, may be prepared from low-grade wood rosin by passing the molten material through a small-bore tubular distiller and then through a similar vaporiser in which it is heated in countercurrent by means of oil to temperatures of 240—260° and 260—302°, respectively. At the same time superheated steam is passed through the molten rosin, and the whole system is maintained at a pressure of 6—127 mm. of mercury. E. HOLMES.

Manufacture of artificial [resin] materials. SOC. CHEM. IND. IN BASLE (B.P. 281,717, 5.12.27. Switz., 4.12.26).—A preliminary condensation product of urea and formaldehyde, condensed beyond the methylol-urea stage, is subjected to the action of an acid or a non-basic salt polymerising agent, in sufficient quantity to cause the whole mass, including the solvent still present, to gelatinise; the resulting material hardens without the application of external heat. The polymerising agent is first neutralised and then washed out, leaving a product of sp. gr. as low as 0.4. Filling materials, plasticisers, and dyes may be incorporated during manufacture. E. HOLMES.

Resin-like hydrocarbons (G.P. 454,307).—See II. **Treatment of fibrous materials** (U.S.P. 1,695,912).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber and its fractions. R. PUMMERER (Kautschuk, 1929, 5, 129—135).—Gel and sol rubber separated from various types of commercial rubber show a difference in the proportion of iodine chloride or benzoyl peroxide with which they react. The gel constituent possesses the greater reactive power, but with iodine chloride yields more hydrogen chloride. This difference in chemical behaviour may be due to cyclic formation in one of the constituents, to *cis-trans* isomerism, or to difference between the structure of the micelles. Sol rubber appears to be convertible partially into the gel type by heating with exclusion of air. A benzene solution of sol rubber on partial crystallisation yields mixed crystals; this renders cryoscopic measurements for rubber in benzene untrustworthy, and may explain the discrepancies between the results of cryoscopic and capillary determinations of the mol. wt.

D. F. TWISS.

Rubbers with low nitrogen content. A. D. CUMMINGS and L. B. SEBRELL (Ind. Eng. Chem., 1929, 21, 553—557).—Purified rubber prepared from latex by treatment with sodium hydroxide by the method of Pummerer and Pahl (A., 1927, 1193) still contained 0.004% N even after intensified treatment. Unlike the rubber from ordinary latex, the rate of vulcanisation of rubber from this purified latex was very little affected by the p_H of the alcohol used as coagulant. Vulcanisation yielded products of good quality comparable with controls containing the normal proportion of nitrogen. Such nitrogen-poor rubber was not easily electro-deposited from the latex. The small proportion of residual nitrogen was not present as protein, but was removable by acetone; the extracted rubber thus obtained vulcanised only slowly and the products were physically less satisfactory.

D. F. TWISS.

Isoprene and rubber. H. STAUDINGER (Kautschuk, 1929, 5, 94—97, 126—129).—A review of the author's investigations of the constitution of rubber mainly by way of hydrocaoutchouc and of polymerisable substances such as vinyl acetate and styrene. The properties of rubber are dependent on a high mol. wt., possibly approaching 100,000. The difference between rubber and gutta-percha or balata rests probably on a different spatial arrangement of the groups at the double linking, i.e., on *cis-trans* isomerism.

D. F. TWISS.

Colloid-chemical changes in rubber and fatty oils. L. AUER (Trans. Inst. Rubber Ind., 1929, 4, 499—520).—The colloidal condition of iso-colloids is discussed. The isolated sol and gel constituents of rubber are regarded as mixtures representing only products containing increased proportions of the respective true constituents. In the iso-colloidal fatty oil system the disperse phase may increase at the expense of the chemically similar dispersion medium by the action of light, heat, and aggregators, e.g., electrolytes and gases such as oxygen. Formic acid acts as a coagulant on the iso-colloidal fatty oil system, effecting separation of the dispersed phase. Gases such as oxygen also have a coagulant effect on lyophobic colloidal systems, similar to that of electrolytes, e.g., on latex or oils. Sulphur resembles oxygen in its

vulcanising action on oils, increasing the concentration of the disperse phase and coagulating the system; if the sulphur is present in sufficient excess it may subsequently combine chemically with the fatty acids, but this is not primarily necessary for the vulcanisation process. The gelation of oils by heat can be accelerated or retarded by the addition of suitable electrolytes. By vulcanising oils which have been solidified with the aid of electrolytes new types of "rubber substitutes" can be prepared. Similarly, by heating rubber containing electrolytes, thermoplastic conversion products are obtainable.

D. F. TWISS.

Ageing tests for sponge rubber. H. P. STEVENS (Trans. Inst. Rubber Ind., 1929, 4, 486—492).—By compressing a piece of sponge rubber between plates in an ageing oven at 70° for 3 days a compact sheet is formed from which rings can be punched for testing in the usual manner. The ageing can be prolonged as desired, but 2 weeks is the useful limit. Tested in this way, and also by hand tests and chemical examination after ageing uncompressed, different samples not only showed wide variations between themselves, but irregularities in vulcanisation could be detected in individual samples.

D. F. TWISS.

Physical characteristics of sponge rubber. H. F. CHURCH (Trans. Inst. Rubber Ind., 1929, 4, 533—542).—A wide range of samples of sponge rubber sheeting has been examined as to general physical properties. "Cellularity," i.e., the ratio of trapped air to the total air space, was estimated by enclosing a sample in a space of known volume and pressure and observing the effect of alterations in pressure on the total volume. Other characteristics examined included weight per unit volume, compressibility, e.g., between two parallel plates or by an indentation instrument, and permanent set. Cellularity had a very marked influence on compressibility; in the case of true sponges with inter-connecting cells, compressibility was mainly dependent on cell size, and was relatively insensitive to alteration in the physical properties of the rubber or its composition.

D. F. TWISS.

Oxidation of rubber mixings. W. C. DAVEY (Trans. Inst. Rubber Ind., 1929, 4, 493—498).—Examination of progressively aged (70°) samples of vulcanised rubber as to increase in weight and acetone-soluble matter before and after extraction with acetone or with acetone and chloroform successively indicates three stages of oxidation: in the first, oxidation is slight and yields acetone-soluble products; in the second, increase in weight is much more rapid than the production of soluble matter; and in the third, the acetone-soluble matter increases after oxygen absorption has ceased. It is suggested that three successive reversible changes occur, viz., vulcanised rubber = depolymerised rubber (chloroform-soluble) = resinous matter (acetone-soluble) = oxidised matter (insoluble). Of these, the middle stage is technically of greatest importance.

D. F. TWISS.

Distribution of ingredients in rubber mixings. H. PAGE (Trans. Inst. Rubber Ind., 1929, 4, 521—525).—Evidence is adduced that, contrary to a recent opinion

(Reiner, B., 1928, 341), compounding ingredients can be uniformly dispersed in rubber by mixing under the ordinary conditions of the factory mixing operation.

D. T. TWISS.

Chemical reactions in rubber compounds.

I. Reactions between pine tar and litharge. W. H. REECE (Trans. Inst. Rubber Ind., 1929, 4, 526—532).—When litharge is added to a rubber "compound" containing pine tar, a hardening soon occurs. That this change is due to a chemical reaction between litharge and pine tar comparable with that which occurs between them when heated without rubber is confirmed, *e.g.*, by the approximate agreement in the amount of water formed under the two sets of conditions. The pine tar used contained 46.9% of acids which reacted with litharge yielding 2.45% of water. D. F. TWISS.

Temperature-recording micropress for studying the course of vulcanisation. J. C. WALTON (Ind. Eng. Chem. [Anal.], 1929, 1, 106—108).—The micropress consists of an electrically heated upper part, a central portion containing a thermocouple, and a container for the sample undergoing vulcanisation; readings obtained on a millivoltmeter are interpreted by measurements with crystals of different organic substances. Continuous observation of specimens at temperatures between 30° and 170°, with the addition of various curing agents, has shown that the changes produced in the rubber can be critically followed in this way. R. H. GRIFFITH.

Compressibility of rubber. R. ARIANO (Nuovo Cim., 1928, 5, 77—101; Chem. Zentr., 1929, i, 153).—A study of the compressibility curves of vulcanised rubber, and of the effect of duration of vulcanisation on the mathematical constants. A. A. ELDRIDGE.

Power consumption in [rubber] crêpeing mills. R. RIEBL (Med. Proefstat. Rubber No. 41; Arch. Rubbercultuur, 1929, 13, 219—238).

PATENTS.

Treatment of rubber. SOC. ITAL. PIRELLI, and U. PESTALOZZA (B.P. 284,608, 30.1.28. Italy, 31.1.27).—Latex is treated with an appropriate proportion of a suitable coagulant, especially salts of bi- or ter-valent metals, *e.g.*, calcium sulphate, so as to render it capable of local thickening and subsequent formation of a compact layer of coagulum when brought into contact with a heated surface, *e.g.*, at 75—95°. Rubber solvents such as benzol or benzine may be used in conjunction with such coagulants. Articles are manufactured from such latex, which may be natural, preserved, concentrated, compounded, or otherwise, by bringing it into contact for a short period with a heated mould or former of the desired shape and size. D. F. TWISS.

Vulcanisation of rubber. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of B. W. NORDLANDER (B.P. 290,602, 15.5.28. U.S., 16.5.27).—"Stabilised amorphous sulphur," for the vulcanisation of rubber, is prepared by fusing sulphur with selenium or tellurium and grinding the brittle product or by allowing hydrogen sulphide to react with selenious acid in a suitable liquid medium; vulcanisation may be aided with an organic accelerator.

The selenium or tellurium maintains the sulphur in an amorphous form which is exceptionally active in vulcanisation. Sulphur so treated may also be mixed colloiddally into rubber latex for electrodeposition purposes. D. F. TWISS.

Preparation of products of latex-like character. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 312,201, 19.12.27 and 1.6.28).—Butadiene and its homologues or analogues are dispersed in aqueous media with emulsifying agents reducing the surface tension of the water; polymerisation is then effected in the presence, if desired, of buffers giving a p_H range of 4—8.5. The addition of compounds supplying oxygen and of agents which combine with acids further simplifies the formation of a latex-like product. D. F. TWISS.

Manufacture of rubber compositions. DUNLOP RUBBER Co., LTD., A. E. T. NEALE, and F. THOMAS (B.P. 311,930, 5.5.28).—Condensation products of an aldehyde and an aminophenol prepared in neutral or acid solution, *e.g.*, of acetaldol or crotonaldehyde and *p*-aminophenol, are employed as antioxidants for rubber. D. F. TWISS.

Incorporation of colloidal substances in rubber. FELTEN & GUILLEAUME CARLSWERK A.-G. (G.P. 453,628, 20.1.23).—Ingredients such as sulphur are dissolved colloiddally in a rubber solvent and then mixed with the rubber. Uniformity is thus ensured. D. F. TWISS.

Method of compounding caoutchouc. R. P. DINSMORE, Assr. to GOODYEAR TIRE and RUBBER Co. (U.S.P. 1,712,333, 7.5.29. Appl., 22.11.24).—An emulsion of rubber is mixed with a suspension of compounding ingredient, the rubber being then coagulated on the latter. The product is freed from water-soluble material by washing, and a rubber softener is added to the still pasty mass, which is then incorporated into a rubber mixture by milling. D. F. TWISS.

Direct production of thread or tubes from concentrated compounded latex. DUNLOP RUBBER Co., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 311,844, 23.2. and 5.9.28).—Concentrated and/or compounded aqueous dispersions of rubber or similar materials are caused to flow through orifices of the desired shape into a bath, such as a solution of common salt (150 pts.), ammonium acetate (150), and water (450), capable of effecting dehydration and setting at least partly by penetrative osmotic action. The cross-sectional area of the threads or tubes can be varied by altering the distance of the orifice below the surface of the bath. If a number of threads, on issuing from the bath, are allowed to come into contact and are then dried, they unite to form a tape with longitudinal grooves on its wide surfaces. D. F. TWISS.

Deodorisation of articles produced by the electrophoresis of latex mixings. DUNLOP RUBBER Co., LTD., and E. W. MADGE (B.P. 312,443, 29.3.28).—Articles produced by electrophoretic deposition from latex are treated with formaldehyde or an aqueous solution of paraformaldehyde, *e.g.*, by immersion for 2—3 hrs. in a 20% solution of the former. D. F. TWISS.

Rubber-bonded abrasive articles (B.P. 311,104).—See VIII.

XV.—LEATHER; GLUE.

Biochemistry of soaking and liming [of animal skins]. III. Influence of gaseous environment on the soaking of heavy hides. E. R. THEIS and J. M. MILLER (J. Amer. Leather Chem. Assoc., 1929, 24, 290—314; B., 1928, 827).—The gases which cause the greatest protein degradation of "domestic" hides are nitrogen and hydrogen, whilst in the case of "Frigorificos," hydrogen causes the greatest decomposition and nitrogen the least. A greater amount of volatile fatty acids is obtained in a nitrogen atmosphere than in one of oxygen; these acids appear to be derived from the hydrolysis of the skin proteins. The greatest hydration of the skin is obtained in atmospheres of carbon dioxide and oxygen, respectively, and the least in nitrogen. The soak water is rendered alkaline by using atmospheres of air or oxygen. D. WOODROFFE.

Hydrolysis of hide powder in saturated sodium chloride solutions at various p_H values. A. W. THOMAS and M. W. KELLY (J. Amer. Leather Chem. Assoc., 1929, 24, 280—282).—Weighed portions of hide powder were treated with saturated solutions of sodium chloride of p_H values 2.0 to 11.0, known volumes of the solutions were withdrawn at intervals up to 224 days, the nitrogen content was determined, and the percentage hydrolysis calculated for each time interval. The p_H values of the solutions were determined at the end of the experiment. The hydrolysis of hide powder in saturated sodium chloride solutions is shown to increase with increasing alkalinity, and the hydrolysis of hide powder as a function of the p_H value is affected by the presence of sodium chloride. The addition of sodium carbonate to preclude salt stains causes a loss of hide substance. D. WOODROFFE.

Action of sodium chloride on hides and on animal tissues. A. PONTE (Boll. Uff. Staz. Sperim. Ind. Pelli, 1929, 7, 97—119).—The differences in the solubilising actions of sodium chloride and sodium sulphate on the derma proteins seem to become accentuated as the degree of alteration undergone by these proteins increases. Experiments on prepared hides ready for tanning and freed from water of capillarity show that dilute sodium chloride solutions (5.8%) effect only slight changes due to a feeble swelling action. Increasing salt concentration is accompanied by a progressive deswelling effect, a saturated or almost saturated solution reducing the moisture content of the hide to about 50%. Similar dehydration doubtless occurs when beef is salted. T. H. POPE.

Measurement of the properties of sole leather and the effects of sulphuric acid. D. BURTON (J. Soc. Leather Trades' Chem., 1929, 13, 178—191).—The various physical properties of sole leather are enumerated and the work which has been done in that connexion is summarised. The usual chemical analysis is considered to have very little relation to the durability of the leather and actual practical requirements. The sp. gr. of leather is important in judging quality and cutting value, but it is difficult to obtain a representative figure since it varies with the position of sampling. The degree of tanning indicates the quality of a leather and the yield likely to be obtained on currying or retanning.

Some leathers which contain 1.1% of free mineral acid as determined by the Procter-Searle method are quite sound after 5 years' keeping; others are entirely disintegrated. Raw hide treated with sulphuric acid will keep indefinitely. Innes' method (B., 1928, 721) fails if oxalic acid and syntans are present in a leather. Deterioration of leather appears to be caused by free, but not combined, sulphuric acid. The yield of sole leather and the amount of tan fixed by the hide is affected by the method and the degree of liming. The greater the hydrolysis of the collagen, the greater is its combining capacity. Methods for sterilising anthrax-infected hides, preventing the formation of deposits from myrobalan extracts, and overcoming the difference between tan liquors prepared from extracts and those made by leaching are considered. D. WOODROFFE.

Influence of hydrogen-ion concentration on the colour of vegetable-tanned leather. R. O. and A. W. PAGE (Ind. Eng. Chem., 1929, 21, 584—585).—Two series of tan liquors ranging in p_H value from 1.0 to 9.0 were prepared from wattle bark and chestnut wood extracts, respectively. Pieces of bated ox hide were brought to the p_H value of the tan liquor with which they were to be tanned, then placed in that tan liquor for 24 hrs., removed, and their colours compared. The tan liquors showed a progressive darkening in colour with increasing p_H value, but the variation in colour of the leather followed closely the swelling curve. It was palest at p_H 5, darkest at p_H 2, brightened toward p_H 1, but with an increased reddish tint, darkened gradually from p_H 5 to p_H 7, and more so at p_H 8—9, especially with the chestnut liquors. Pretreatment of the hide pieces with a buffer solution of p_H 5 followed by tannage first in wattle-bark liquors at p_H 5 and finally in liquors varying in p_H from 1 to 9, gave leather pieces in which there was a regular gradation in colour from the lightest at p_H 1 to the darkest at p_H 9. Pretreatment of the hide pieces with buffer solutions of high or low p_H values caused a darkening in colour of the subsequently tanned leather. It follows that the colour changes on the tanned leathers are due to actual changes in hide structure brought about by variation in the hydrogen-ion concentration. D. WOODROFFE.

Temperature factor in vegetable tannin fixation. A. W. THOMAS and M. W. KELLY (J. Amer. Leather Chem. Assoc., 1929, 24, 282—289).—Portions of hide powder were tanned with wattle bark, oak bark, gambier, and quebracho liquors, respectively, at 4°, 7°, 25°, and 37.5°, and the amount of fixed tannin was determined at intervals up to 117 days. The rate of tanning was markedly greater at 37.5° than at 25°, and then gradually fell with decrease in temperature. Fixation of tannin occurred at low temperatures, but the amount was very small. D. WOODROFFE.

Determination of moisture in tanning extracts. Report of Committee of the American Leather Chemists' Association. H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1929, 24, 314—321).—Comparisons have been made of the direct method of determining moisture, i.e., by drying, and the indirect method, in which it is obtained by difference from the total solids figure in the official method of tannin

analysis, using a solid quebracho extract. Under some conditions identical results were obtained by the two methods, and under others 0.7—1.2% more water was found by the direct method. The effect of time of drying, size of sample, and thickness of the film on the results has been studied, and also the method of drying in both methods. Identical results were obtained by both methods in determining the moisture in wattle and larch extracts. It is recommended that the direct method for determining moisture in tanning extracts be used only on materials which do not give a uniformly turbid solution. D. WOODROFFE.

Qualitative analysis of [vegetable] tanning materials by means of ultra-violet rays. V. KUBELKA and V. NEMEC (J. Soc. Leather Trades' Chem., 1929, 13, 113—117).—Ethyl ether or ethyl acetate (10 c.c.) is shaken for 5 min. with 20 c.c. of an aqueous infusion of the tanning material of analytical strength, and the ethereal or acetate layer is separated and examined by ultra-violet light. The solutions have been obtained for a number of different tanning materials and their fluorescence alone, after rendering alkaline and acid respectively, on cotton wool, and after rendering alkaline and placing on cotton wool have been observed. Extracts of the dry tanning materials with 50% alcohol have been examined in ultra-violet light, then rendered alkaline, the colour change noted, and the colours of each observed on cotton wool. To identify gambier, 5 c.c. of the tannin solution is shaken for 1 min. with 5 c.c. of alcohol; a greyish-violet fluorescence in ultra-violet light is produced. Addition of 1 c.c. of sodium hydroxide solution produces a faint greenish fluorescence. When 10 c.c. of petroleum spirit are added and the mixture is shaken in a separating funnel and allowed to separate, the spirit layer will show a brilliant pale-green fluorescence even in presence of very small quantities of gambier. D. WOODROFFE.

One-bath chrome-tanning process in the light of Werner's co-ordination theory. C. H. SPIERS (J. Amer. Leather Chem. Assoc., 1929, 24, 246—270).—The addition of neutral salts to a chrome-tanning liquor causes an increase in the precipitation figure and diminishes the amount of chromium absorbed by pelt. Boiling diminishes the p_H value of the liquors, increases the precipitation figure, diminishes the amount of chromium absorbed by the pelt, and increases the time required for tannage. A definite time is required for the chrome liquor to reach equilibrium after it has been diluted or boiled, or salts, acids, or alkalis have been added. The precipitation figure and the amount of chromium absorbed by pelt are both diminished by dilution of a chrome liquor. These various effects are attributed to the formation and existence of chromium complexes of the Werner type. Their stability depends on the nature of the central co-ordinating atom and of the co-ordinated groups. Thus hydroxyl groups are more firmly retained than is chlorine and will replace it. Bivalent ions give more stable complexes than univalent ions and will replace them. Units with two co-ordinatable groups, e.g., oxalate ions, form very stable ring structures, hence the extraction of chromium from chrome-tanned leather by oxalic acid. In chrome

tanning the carboxyl of the collagen combines with the chromium by co-ordinating itself with it, but this would be impossible if the complex ions were too stable. Chromium cations should contain water and hydrolyse in order to show good tanning properties. The work of Stiasny and his collaborators is reviewed, and their theories of "ol" and "oxo" compounds, which are most active in tanning, is expounded. The more complicated the "ol" and "oxo" compounds, the larger are the molecules, and the more they approach colloidal dimensions. Dilution promotes hydrolysis with consequent formation of "ol" and "oxo" compounds, thus producing coarser dispersions. Additions of alkali or pelt to a chrome liquor disturbs the equilibrium in it, causing the production of complex ions containing more "hydroxo," "ol," and "oxo" groups, and it may require time to restore equilibrium, hence the subsequent changes on ageing. Stiasny and Gustavson's theories of the mechanism of tanning are reviewed and compared. D. WOODROFFE.

Distribution of chromium in one-bath chrome-tanned leathers. W. SCHINDLER and K. KLANFER (Collegium, 1929, 121—153).—Calf pelts were tanned with different chrome alum solutions rendered basic with sodium carbonate, and the chromium was determined in the different layers of the tanned leather. The chromium content of all layers increased with increase in basicity of the tan liquor. The middle layer contained most chromium for tan liquors of basicity $> 40\%$, and the grain layer for liquors of basicity $> 40\%$. "Ageing" did not affect the chromium distribution for basicities of 22%, neither did variations in the precipitation figure of the tan liquors used. The grain of leathers tanned with fresh tan liquors of 45% basicity contained more chromium than the middle layer, whereas the reverse was found with "aged" liquors. The composition of chrome-tanned leathers was affected more by the composition of liquors of 45% basicity than by that of tan liquors of lower basicity. The grain contained more chromium than the middle layer when the precipitation figure of a chrome-tanning liquor fell below an "isostratic" limit (about 2.0—3.0) which depended on the kind of tan liquor used and the pre-treatment of the pelt. A smaller amount of chromium was fixed by the grain than by the middle layers with increased amount of sulphuric acid in the pickle used on the pelts before chrome tanning, whilst there was decreased fixation of chromium in the middle layers when a similar increased amount of formic acid was used. Small variations in the concentration of the chrome-tanning liquor, within narrow limits, did not affect the fixation of chromium. At 11° and 26° the middle layer fixed more chromium than the grain, but at 35° the grain fixed slightly more than the middle layer. Large additions of sodium carbonate to the chrome-tanning liquors towards the end of the tannage considerably increased the chromium absorbed by the grain, but had little effect on the flesh and middle layers. D. WOODROFFE.

Proposed official method for analysis of one-bath chrome-[tanning] liquors. Report of Commission of the Society of Leather Trades' Chemists. R. F. INNES (J. Soc. Leather Trades' Chem., 1929, 13,

111—112).—An aliquot portion of the liquor is diluted to 150 c.c., an excess (10 c.c.) of *N*-sodium hydroxide and 10 c.c. of 10-vol. hydrogen peroxide are added, and the mixture is boiled in contact with a clean piece of iron and either filtered or diluted to 400 c.c., then mixed with 5 c.c. of cold saturated potassium permanganate solution, made up to 500 c.c., and filtered; the chromium is determined iodometrically using a 5 c.c. excess of concentrated hydrochloric acid and 15 c.c. of 20% potassium iodide solution. The acidity is determined as before (cf. *ibid.*, 1924, 504) and the p_H value obtained electrometrically.

D. WOODROFFE.

Production of tannin extracts from crude tanning materials. P. JAKIMOV (J. Chem. Ind. [Russia], 1928, 5, 507—509; Chem. Zentr., 1928, ii, 2767).—The process (of Smetkin and Jakimov) depends on the fact that the colloidal tannic acid solutions diffuse more slowly through a membrane than the other water-soluble substances. The cell walls of the extracted material serve as the dialysing membrane.

A. A. ELDRIDGE.

Alkaloid test for tannins. C. M. FEAR (Analyst, 1929, 54, 316—318). The addition of 2 c.c. of 1% gallo-tannin solution to a large number of alkaloid hydrochlorides showed that the only ones giving appreciable precipitates were brucine, caffeine, cinchonine, cinchonidine, quinine, and strychnine. The reaction appears not to be due to precipitates of unchanged alkaloid hydrochlorides, since the intensity of the precipitates does not vary from 1% to 10% solutions, but is due rather to an interaction, either physical or chemical, of alkaloid and tannin.

D. G. HEWER.

Glue testing. II. Report of the Commission of the German Association for testing technical materials. O. GERNGROSS (Collegium, 1929, 119—121; 191—207; cf. B., 1928, 496).—The water content, ash, p_H value, viscosity, jelly strength, "adhesibility" values of Rudeloff and of Bechhold, and glutin content were determined by various members of the Commission for 15 different glues and gelatins, of which five were bone glues. The m.p. and setting point were determined for gelatins. A glue recovered from chrome leather showed high jelly strength, glutin content, m.p., and setting point. The above short method of testing gave a false valuation for bone glues which did not coincide with their adhesive properties. Bone glues of the same viscosity as hide glues possessed a considerably higher "adhesibility" (Rudeloff). Attempts to evaluate glues and gelatins by precipitation with sulphasalicylic acid were unsuccessful.

D. WOODROFFE.

PATENTS.

Leather oil and its manufacture. A. E. BECKER and A. B. BOEHM, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,715,892, 4.6.29. Appl., 27.7.25).—A mineral oil (lubricating grade) and an oil-soluble sulphonate containing free alkali, which is neutralised by a saponifiable fish oil, are compounded.

E. LEWKOWITSCHE.

Waterproofing vegetable protein-base glue. G. H. OSGOOD (U.S.P. 1,706,674, 26.3.29. Appl., 11.8.26).—A solution of relatively small quantities of gum dammar, asphalt, and sulphur in carbon disulphide is mixed with an aqueous solution of vegetable meal with

a high protein content, calcium hydroxide, sodium carbonate, and sodium silicate.

L. A. COLES.

Compressed laminated products and their manufacture. SPRUCOLITE CORP., Assees. of L. J. OLLESHEIMER (B.P. 299,441, 29.2.28. U.S., 28.10.27).—Sheets of wood are bound together under compression by a glue containing water, e.g., an aqueous solution containing casein, lime, sodium silicate, and a metal salt, the original wood or the finished block being dried to such an extent that the moisture content of the final product, including the glue, is less than that of ordinary dry wood.

L. A. COLES.

Tanning material. O. SPENGLER and A. THURM (U.S.P. 1,698,659, 8.1.29. Appl., 25.2.27. Ger., 23.2.26).—See B.P. 266,697; B., 1928, 206.

Leather oil (B.P. 312,768).—See XII.

XVL—AGRICULTURE.

Soil microbiology. IV. Degradation of cellulose in soil. S. WINOGRADSKY (Ann. Inst. Pasteur, 1929, 43, 549—633).—The organisms concerned in the decomposition of cellulose in soil are classified and their characteristics described. Cellulose undergoes a rapid oxidation in soil and the product closely resembles oxycellulose. The biological oxidation of cellulose is differentiated from the purely chemical process in that the product does not reduce Fehling's solution. Assimilable nitrogen, preferably inorganic, is essential to this process, the consumption amounting to approx. 2 pts. of nitrogen per 100 pts. of cellulose decomposed. The consumed nitrogen is transformed into organic combination, but there is evidence of some partial reduction to ammonia even with unrestricted access to the air. The optimum reaction for cellulose decomposition is p_H 7. Slight changes of reaction occur during the process depending on the form in which the nitrogen is supplied. Colloidal acids affect the reaction of the medium to a small but definite extent. There is no production of fatty acids or of volatile matter, and the presence of these substances may be taken as an indication of the presence of anaerobic organisms.

A. G. POLLARD.

Soil actinomycetes. I. Introduction. K. SUBRAHMANYAN and R. V. NORRIS (J. Indian Inst. Sci., 1929, 12A, 53—56). **II. Mode of occurrence in soil.** K. SUBRAHMANYAN (*Ibid.*, 57—68).—I. The literature of the subject is reviewed.

II. Actinomycetes occur in soil only in the conidial form. The usual micro-organic nutrients and stimulants do not accelerate the vegetation of actinomycetes in soil.

A. G. POLLARD.

Colorimetric determination of nitrates in soil and water. L. U. DE NARDO (Giorn. Chim. Ind. Appl., 1929, 11, 107—109).—Use is made of a 2.5% pyrogallolsulphonic acid solution, prepared by dissolving 5 g. of pyrogallol in 10 c.c. of concentrated sulphuric acid, heating for a few moments at 80—90°, allowing the acid formed to crystallise, and dissolving in water to 200 c.c. A sample of the fresh soil (100 g.) is shaken for 6 hrs. with 200 c.c. of water, and 80 c.c. of the filtered liquid are shaken and heated to boiling in a 100-c.c. flask with 1—3 c.c. of saturated baryta solution. When

the precipitate has settled, 0.5—1 c.c. of 50% basic lead acetate solution is added, excess of lead and any barium being precipitated after 2—3 min. with about 5 c.c. of saturated sodium sulphate solution, and the cold liquid made up to 100 c.c. and filtered. Ten c.c. of the filtrate are placed in a flat-bottomed porcelain dish (about 50 c.c.) and, if the soil contains nitrites in excess of 0.1 mg. of N_2O_3 per kg., are treated with a drop of saturated carbamide solution and, with stirring, with about 1 c.c. of concentrated sulphuric acid. After 10 min., 0.5 c.c. of the pyrogallolsulphonic acid solution is added and also slowly and with stirring, 20 c.c. of concentrated sulphuric acid. After the lapse of 1 hr. the coloration is compared with those obtained similarly with nitrate solutions of known strengths. If the 10 c.c. of solution tested contains more than 0.1 mg. of KNO_3 , pyrogallol should be used instead of the sulphonic acid. If the latter fails to detect nitrate, smaller proportions may be tested for by suitably concentrating the alkaline defecated solution before applying the colour test. The method may be applied also to aqueous vegetable extracts, foodstuffs, etc.

T. H. POPE.

Dependence of soil reaction on fertilisation and season. K. BORESCHI and R. KREYZI (Fortschr. Landw., 1928, 3, 963—968; Chem. Zentr., 1929, i, 284).—In pot experiments with potatoes, stable manure and calcium cyanamide reduced the faintly acid reaction of the soil; carbamide, ammonium sulphate-saltpetre, and ammonium sulphate increased it considerably, whilst sodium nitrate was almost without effect. Variations in the hydrogen-ion concentration of the aqueous extract of the soil represent its varying carbon dioxide content.

A. A. ELDRIDGE.

[Fertilising] action of "ammonia-superphosphate" compared with ammonium sulphate and superphosphate. DENSCH, HUNNIUS, and STEINFATT (Landw. Jahrb., 1928, 68, Suppl. I, 21—22; Chem. Zentr., 1929, i, 130).—"Ammonia-superphosphate" undergoes conversion from ammonium sulphate and superphosphate into ammonium phosphate and calcium sulphate, giving a favourable soil reaction for plant growth.

A. A. ELDRIDGE.

Rhenania phosphate [fertiliser]. DENSCH, HUNNIUS, and STEINFATT (Landw. Jahrb., 1928, 68, Suppl. I, 20—21; Chem. Zentr., 1929, i, 130).—Comparative experiments with basic slag and superphosphate are described.

A. A. ELDRIDGE.

Solubility of phosphorites at different hydrogen-ion concentrations, and their assimilability by plants. S. N. ROSANOV (Landw. Jahrb., 1928, 68, 559—581; Bied. Zentr., 1929, 58, 253—255).—The solubility in the usual solvents of Russian phosphorites was compared with that of apatite and of basic slag. The solubility in various buffer solutions of all materials examined increased with acidity over the range p_{H} 3.0—8.0. Differences in the solubility of phosphorites due to fineness of grinding were examined by shaking with buffer solutions for 2 hrs. Such differences were only apparent in acid solutions, and these disappeared when the extraction period was increased to 2 weeks and over. Pot experiments with buckwheat indicated a

similar order of assimilability of the phosphorites by plants as was shown by extraction with buffer solutions.

A. G. POLLARD.

Effect of nitrates on the growth of flax. V. ILLUVIEV and K. GALUNOVA (Ber. landw. Versuchsstat. Engelhardt, 1928, No. 3, 59 pp.; Chem. Zentr., 1929, i, 283—284).—In a loess soil sodium nitrate retards the development of flax; the cultural state of the soil, and particularly its nitrogen content, influence this effect, as also does the time of application. Sodium dihydrogen phosphate is somewhat compensatory. The p_{H} of the soil is raised by sodium nitrate and phosphate, and lowered by ammonium nitrate and potassium sulphate. Variations in the p_{H} of the soil caused by the flax were followed.

A. A. ELDRIDGE.

Effect of nitrogen in increasing yield [of plants]. GERLACH and SEIDEL (Landw. Jahrb., 1928, 68, Suppl. I, 299; Chem. Zentr., 1929, i, 130).—The effect of a given quantity of nitrogen in a particular fertiliser depends on the amount of water it receives, and on the kind of plant treated.

A. A. ELDRIDGE.

Cause of the sensitiveness to lime of yellow lupins. DENSCH (Landw. Jahrb., 1928, 68, Suppl. I, 33—34; Chem. Zentr., 1929, i, 131).—The action is specific, and is more marked with calcium sulphate than with the carbonate. The availability of the phosphate appears to be diminished, an arrest of protein formation and an accumulation of sugars resulting.

A. A. ELDRIDGE.

Stimulation of mustard seeds with carbon disulphide. MUTH and VOIGT (Landw. Jahrb., 1928, 68, Suppl. I, 428—429; Chem. Zentr., 1929, i, 132).—Stimulation of growth was observed.

A. A. ELDRIDGE.

Composition of mangolds grown in Mid-Wales. T. W. FAGAN and J. E. WATKIN (Welsh J. Agric., 1928, 4, 102—113).—Five varieties were compared. Nitrogenous fertilisers increased the protein in the dry matter. Application of ammonium chloride or heavy dressings of ammonium sulphate reduced the percentage of dry matter and sugar. Potash narrowed the ratio of true to crude protein. Storage of mangolds in clamps reduced the dry matter and sugar content, but largely increased the percentage of true protein in the dry matter. When mangolds were allowed to run to seed the dry matter and sugar content were reduced. Mangold leaves contain: crude protein 21.64, silica-free ash 19.00, CaO 2.19, P_2O_5 0.98, K_2O 5.61%.

CHEMICAL ABSTRACTS.

Laboratory method for measuring relative adhesive qualities of fungicidal dusts. R. C. WILLIAMS (Ind. Eng. Chem. [Anal.], 1929, 1, 81—82).—Microscope slides are coated with shellac, placed under a bell jar, and treated with fungicide dust from an atomiser. The amount of dust on each is determined by weighing, and its resistance to washing and drying is measured by moistening in steam, drying in air, and dipping ten times into water; the loss from this treatment is shown by another weighing. Similar tests can be made with apple leaves of standard size, and the results are in general agreement with those of field experiments.

R. H. GRIFFITH.

Petroleum products as insecticides. INMAN.—See II.

PATENT.

Plant disinfectant and its manufacture. R. ZELLMANN and D. LAMMERING, Assrs. to CHEM. FABR. VON HEYDEN (U.S.P. 1,694,497, 11.12.28. Appl., 26.12.25. Ger., 4.10.24).—Metallic copper is distributed on a finely-divided carrier; e.g., lime water is added to a solution of ferrous and copper sulphates to give reduced copper on a ferric hydroxide-gypsum carrier.

C. HOLLINS.

XVII.—SUGARS; STARCHES; GUMS.

Diffusion experiments with dried beet slices. V. STANEK and K. SANDERA (Z. Zuckerind. Czechoslov., 1929, 53, 525—534).—Beet slices dried at Eynsham by the Oxford process were extracted in a laboratory diffusion battery comprising 16 water-jacketed cells, designed to reproduce the conditions of large-scale working. The slices were about 1 in. long on the average, and contained 6.9% of moisture, 66% of sucrose (polarisation), and 1.01% of invert sugar. The diffusion operation presented no great technical difficulties. The charge of dry slices per cell was 200 g. (corresponding to 80 kg. of fresh slices per hectolitre), and successive cells were charged at intervals of 8 min., so that the slices were in battery for about 128 min. Circulation of juice was maintained by pressure, the fresh water being introduced at 85°, whereby the temperature of the end five cells was kept at 80—83°. With a juice draw-off of 180 c.c. per cell the density attained was 52.9° Brix. The purity quotient of the juice was 88.7—88.9%. Its composition, calculated on 100 pts. of sucrose, showed no striking difference from that of ordinary diffusion juice in respect of ash, total nitrogen, or nitrogen as ammonia, amides, or betaine. The amount of protein nitrogen was relatively low, representing about 3—5% of that in the original slices, whilst in the diffusion of fresh slices about 20% of the total passes into the juice. The juices from the dried slices were rather acid, 100 c.c. requiring 5.2 c.c. of *N*-sodium hydroxide, and consequently some inversion occurred during diffusion so that the final juices contained about 2 pts. of invert sugar per 100 pts. of sucrose. About 4% of the sucrose remained in the exhausted slices, but this abnormally high loss may be attributable to the thickness of the slices, which in the swollen state ranged up to 7 mm.

J. H. LANE.

Treatment of beets by De Vecchis' process. J. PROCHÁZKA (Z. Zuckerind. Czechoslov., 1929, 53, 453—456).—Juices obtained by extraction of dried beets are not of higher purity than those from fresh beets. In climates where beets deteriorate rapidly in the ground after a certain stage, desiccation of the beets at this stage might avoid serious losses of sugar, but the necessity of drying the whole crop within a short time involves a heavy outlay in drying plant, which would probably outweigh the advantages of the process in other directions.

J. H. LANE.

Spontaneous decomposition of sugar-cane molasses. C. A. BROWNE (Ind. Eng. Chem., 1929, 21, 600—606).—Periodical analyses of two samples of sugar-

cane molasses, which had undergone spontaneous deterioration during 14 years, showed progressive lowering of polarisation, constant decrease in sucrose, slow irregular increase in invert sugar, constant decrease in total sugars after inversion, and constant increase in organic non-sugars. Considerable quantities of formic and acetic acids and of organic colloidal substances both nitrogenous and non-nitrogenous were present. These organic colloids had a carbon content of approx. 52%. No micro-organisms could be detected in the molasses and the spontaneous decomposition is believed to be a retarded "froth" or "hot-room" fermentation purely chemical in nature. The chief type of reaction appears to be the formation of unstable compounds (glucic acid etc.) by the action of the lime used in clarification on the reducing sugars of the juice, but the reaction of reducing sugars with the amino-acids present undoubtedly also plays a part.

W. J. BOYD.

Syrup filtration in making white sugar using kieselguhr. T. VAN DER LINDEN (Archief Suikerind. Nederl.-Indië, 1928, Af. No. 4, 170—175).—Syrup from the evaporators was heated to 85—90° and passed through Kroog filter-presses the cloths of which had been pre-coated with "Hyflo-supercel" grade of kieselguhr. A clear filtrate, lighter in colour than that obtained by the Bach process, was obtained; the consumption of kieselguhr was about 0.6% of the cane.

J. P. OGILVIE.

Effect of activated carbon and of sulphur dioxide combined on juices etc. G. MEZZADROLI and E. VARETON (Ind. Sacch. Ital., 1928, 21, 491—501, 545—554).—Heavy sulphitation of cane and beet juices even at 85° followed by the addition of "Norit" does not cause inversion. Such combined treatment is advantageous, especially with dilute juices, the surface tension, e.g., being diminished to a greater extent than when they are used separately. Decolorisation is permanent, and is not diminished when the juice is made alkaline.

J. P. OGILVIE.

[Cane] milling-diffusion process in Egypt. F. MAXWELL (Internat. Sugar J., 1929, 31, 186—189).—Juice extracted by two 3-roller mills is measured, tempered, and heated; it then passes up through a "meichage" cell filled with bagasse from the mills, and from thence into the Naudet battery of diffusion cells, which it finally leaves in a clear condition ready for the evaporators. Bagasse discharged from the final cells is further expressed in mills, and finally sent to the furnaces. An extraction of juice from the cane of about 98 is obtained in this process employing only four 3-roller mills.

J. P. OGILVIE.

Keeping quality of white sugars. P. HONIG (Internat. Sugar J., 1929, 31, 214).—Sugar should leave the centrifugals with at most 0.6% of water, otherwise it must be passed through a dryer. On leaving the granulators, it should be allowed to cool in a sieving installation so that it comes into contact with the atmosphere at its prevailing water content. Drying and sieving plants should be installed apart from the factory, not in an atmosphere saturated with water. In this department the control of the moisture of the air is as important in the store. The effect of micro-organisms in sugar

deterioration has been over-emphasised, the contents in water and invert sugar, and the p_H being the three main factors.

J. P. OGILVIE.

PATENTS.

Purification of sugar juice. AKTIEROLAGET SEPARATOR (B.P. 287,526, 16.3.28. Swed., 23.3.27).—The sugar juice is clarified in the usual manner (heat and chemical precipitation) and then most of the sediment is allowed to separate by gravity settling. Clarification of the juice is completed by treatment in a centrifuge of the non-perforated bowl type, action being discontinued at intervals for the removal of sediment from the bowl.

E. B. HUGHES.

Removal of hydrochloric acid from sugar solutions. A. CLASSEN (B.P. 309,896, 17.1.28).—For the removal of hydrochloric acid, particularly residual traces, from sugar solutions such as those obtained in the acid saccharification of wood, the solutions are distilled, preferably under reduced pressure, in presence of solid grains or pieces of quartz or other natural siliceous matter, so that by the end of the distillation the syrupy liquid is distributed over the surface of the grains.

J. H. LANE.

Treatment of saccharate scums. RAFFINERIE TIRLEMONTAISE SOC. ANON. (B.P. 290,265, 21.1.28. Ger., 12.5.27).—Carbonatation scums obtained in ordinary sugar manufacture or saccharate scums obtained in the desaccharification of molasses are separated from their respective liquors by centrifuges in which the centrifugal force is increased to 1500 or more times the force of gravitation. The moisture content can thus be reduced to 15–20%, compared with 50% attainable by means of filter presses.

J. H. LANE.

XVIII.—FERMENTATION INDUSTRIES.

Phosphatases of malt. H. LÜERS and L. MALSCH (Woch. Brau., 1929, 46, 143–146, 153–157).—Four phosphatases are present in barley, and during malting increase about tenfold in amount. Glycerophosphatase and phytase are markedly, and saccharophosphatase slightly, restricted during kilning, nucleotidase being little affected. Their temperature and reaction optima are: glycerophosphatase 36°, p_H 5.2; nucleotidase 49°, p_H 5.6; phytase 48°, p_H 5.2–5.3; saccharophosphatase 41.5°, p_H 6.0. Only in the case of phytase and at low concentration is the velocity of hydrolysis proportional to the amount of enzyme. Relatively long flooring at low temperature increases the phosphatase content of malt; high floor temperatures have the reverse effect. The enzymes may be separated by fractional adsorption on alumina.

F. E. DAY.

Biological condition of "bottich"-sediment yeast. Usefulness of pumping-off wort from the "bottich." F. WINDISCH (Woch. Brau., 1929, 46, 183–187, 197–200).—The practice of some bottom-fermentation breweries of removing the fermenting wort from the yeast which first settles, on account of the supposed impurity and lack of vitality of the yeast, is shown to be without justification. In a test, 44% of the yeast present at pumping-off after 1 day was in the sediment and would be rejected. Comparison, with several races, of the sediment-yeast with that centrifuged

from suspension in the fermenting wort showed that the former fermented cane sugar or wort more rapidly than the latter, and gave a slightly higher degree of fermentation. Both small-scale and brewery fermentation was checked by pumping-off the wort, and the final degree of fermentation was lowered. The bearing of wort turbidity and pumping-off on fermentation is discussed.

F. E. DAY.

Importance of hop-tannin for wort and beer. H. BILGRAM (Woch. Brau., 1929, 46, 137–141).—Three brewings, employing hops, hop extract, and tannin-free hop extract, respectively, are compared. It is concluded that the tannin assists and increases precipitation of the more complex proteins and imparts a distinctive hard bitterness which is preferred by some. The addition of hop-tannin to beer precipitates a considerable amount of protein, but though 0.05 g./litre reduced the nitrogen in a beer by 10.9 mg./100 c.c., it did not reduce the liability to turbidity on pasteurisation.

F. E. DAY.

Artificial acidification of mash and worts. F. EMSLANDER (Woch. Brau., 1929, 46, 157–159).—Laboratory mash and fermentations confirm the conclusions of Windisch, Kolbach, and Banholzer (B., 1929, 69) that increase in acidity during mashing leads to higher nitrogen in the resulting beer. The technical application is discussed.

F. E. DAY.

Phosphoric acid and iron contents of Rheingau musts and wines. MUTH and VOIGT (Landw. Jahrb., 1928, 68, Suppl. I, 422–426; Chem. Zentr., 1929, i, 160).—The iron content of the musts was 10.2–20.9 (average 13.93) mg. per litre, falling after 6 weeks to 11.12 and after 12 weeks to 7.85 mg. per litre. The phosphoric acid content of the must and wine, about 20% of the total ash, was 0.48–0.7 (average 0.62) g. per litre. The decrease during the principal fermentation was 17%, and after 12 weeks 20.7%.

A. A. ELDRIDGE.

Occurrence of invertase in must and wine. C. VON DER HEIDE and H. MÄNDLEN (Z. Unters. Lebensm., 1929, 57, 13–36).—Small quantities of invertase occur in grapes and must. The invertase produced by the yeast passes into the wine, which contains relatively much invertase when new. The invertase content decreases gradually and is usually absent from wine over five years old.

W. J. BOYD.

Sweet musts. KOCHS (Landw. Jahrb., 1928, 68, Suppl. I, 364–366; Chem. Zentr., 1929, i, 160).—Apple must contains alcohol 0.16, extract 11.89, sugar 8.71, sugar-free extract 3.28, malic acid 0.714%. Grape must contains alcohol 0.19, extract 17.13, sugar 13.53, sugar-free extract 3.66, acid 0.873%. The musts contain more acid and sugar-free extract than the wines.

A. A. ELDRIDGE.

Use of alkali sulphites in wine manufacture. J. H. FABRE and E. BRÉMOND (Ann. Falsif., 1929, 22, 262–271).—Fermentations were conducted on musts prepared from dried grapes to which were added (1) sulphurous acid alone, (2) ammonium bisulphite, (3) sulphurous acid and diammonium phosphate, (4) sulphurous acid and disodium phosphate. The quantities added corresponded to 25 g. of sulphurous acid, 6.02 g. of ammoniacal nitrogen, and 6.65 g. of phosphorus per

hectolitre. Those with added ammonia (samples 2 and 3) fermented most rapidly, the yeast utilising the added ammonia but not the phosphate. With fresh musts fermented on an industrial scale, with similar additions, except that the amount of phosphorus was doubled by the use of monophosphates, there was little difference in the fermentation rates, sample 2 fermenting most slowly. As before, the added nitrogen was utilised but not the phosphorus. It is concluded that ammonium sulphite is suitable for use in wine manufacture, being without ill effect on yeast or wine. Phosphates appear to be of no value as yeast nutrients, and in this case appeared to favour the development of "casse ferrique" which subsequently appeared in (3) and (4) of the second series, which contained about 0.04 g./litre of iron.

F. E. DAY.

General action of very hard frosts on the chemical composition of wines. ASTRUC and CASTEL (Ann. Falsif., 1929, 22, 272—275).—Wines which were partially frozen during the severe frost of the early part of this year have suffered an appreciable loss of acidity and extract, presumably by abnormal separation of potassium bitartrate. Analyses, of which three sets are quoted, show that this occurred mainly after December 26, 1928, and attention is drawn to the resulting raising of Blarez' and the alcohol/extract ratios and lowering of the Halphen and Roos ratios. Since these ratios are used to form an opinion on the genuine or other character of wines, the possibility of their alteration by freezing must not be overlooked.

F. E. DAY.

Test for cider and other fruit juice in wine. J. WERDER (Ann. Falsif., 1929, 22, 260—261). **Conversion of dibenzylidenesorbitol into sorbitol hexaacetate.** C. ZACH (*Ibid.*, 261—262).—Sorbitol is present in most fruits of the natural order *Rosaceae* and absent from grapes. The suspected wine is fermented as completely as possible, treated with animal charcoal, concentrated to a syrup, and a few drops of benzaldehyde and 1 c.c. of 1:1 sulphuric acid are added. After well shaking, the test is stood aside for at least 10 hrs., when dibenzylidenesorbitol separates if cider is present, and on dilution remains insoluble as a white flocculent precipitate. For further identification this may be hydrolysed by sulphuric acid, the benzaldehyde removed by ether and the sulphuric acid by baryta. The sorbitol obtained by evaporation is treated with 0.5 c.c. of acetic anhydride and a drop of pyridine for 1 hr. on the boiling water bath, and the hexa-acetyl derivative crystallised from hot water. If necessary, it may be purified by washing its solution in ether with 10% soda and water.

F. E. DAY.

Examination of damaged wines. L. ROOS (Ann. Falsif., 1929, 22, 166—169).—Wines attacked by *Mycoderma vini* may exhibit the characteristics of watered wine, for both the alcohol content and acidity are lowered. A red wine with a surface of 30 cm.²/litre was inoculated with a trace of *Mycoderma vini* and on analysis for alcohol content, and for acidity on the original wine and before and after 7 and 11 days, respectively, had alcohol 8.6, 7.7, and 6.65; total acidity 5.0, 4.8, 4.7; volatile acidity 1.28, 1.18, and 1.18.

D. G. HEWER.

Detection of isopropylalcohol in brandy by Griebel's micro-beaker method. F. WEISS (Z. Unters. Lebensm., 1929, 57, 45—48).—The alcohol is distilled from 1—5 c.c. of the brandy and 0.1 c.c. of the distillate is placed in a flask containing 5 c.c. of water and 8—10 drops of 50% chromic acid solution. 3 c.c. of the liquid are slowly distilled into a Reichert-Meißl flask containing 2.5 g. of 50% silver nitrate solution and 1 g. of a 30% sodium hydroxide solution in 10 c.c. of water. The mixture is boiled for 4 hrs. under reflux, cooled, and kept for 2—3 hrs. Then 1 c.c. of the liquid is distilled off and tested for acetone by Griebel's micro-beaker method (cf. B., 1929, 121), using *m*-nitrophenylhydrazine. If the brandy is free from isopropyl alcohol no crystals are formed in the drop of reagent unless the last traces of acetaldehyde have not been removed by the silver oxide, but the acetaldehyde derivative is easily distinguishable from the acetone compound by its crystalline form. If 5% or more of isopropyl alcohol is present in the brandy the final distillate must be diluted in order that the characteristic form of the crystals may be seen. No genuine brandy was found to give a positive test for isopropyl alcohol, 1% of which can be satisfactorily detected by this method.

W. J. BOYD.

Composition of fusel oils which have been separated as vapour. B. LAMPE (Z. Spiritusind., 1929, 52, 190).—The percentages of amyl alcohol obtained by fractional distillation from seven samples of fusel oil are quoted, and comparison of these with the results of rough commercial tests shows the latter to be misleading. Since two samples which had been separated as vapour contained 42.6 and 46.6% of amyl alcohol, against 50—63.75% in five samples separated as liquid, there appears to be no ground for supposing that the former separation yields fusel oil richer in amyl alcohol.

F. E. DAY.

Determination of nitrogen. LUNDIN and ELLBURG.—See VII. **Inoculation of milk.** WOLFF.—See XIX.

PATENTS.

Recovery and drying of solid matter from distillery wash and other liquids. B.W.A. and J. L. WATNEY (B.P. 312,433, 22.3.28).—The solid matter is separated from the liquid by a horizontally inclined oscillating or revolving sieve and discharged into a tank in which it is agitated with water. The sludge is pumped into a filter press and the separated solid matter conveyed to a steam-heated dryer consisting of a number of horizontally inclined units which are alternately oppositely inclined, and enclosed in a perforated metal or wire-gauze cover.

C. RANKEN.

Separation of gases (U.S.P. 1,706,707).—See VII. **Extraction of oils (U.S.P. 1,698,294).**—See XII.

XIX.—FOODS.

Colorimetric determination of ergot in flour. F. S. OKOLOV (Z. Unters. Lebensm., 1929, 57, 63—71).—The colorimetric method described is based on that of Hofmann (Arch. Pharm., 1885, 223, 828). The disturbing effect of the colouring matter of the flour is avoided by concentrating the ergot by flotation on a mixture of 500 c.c. of chloroform and 60 c.c.

of alcohol. The colouring matter is extracted from the ergot fraction by shaking it with a mixture of ether and 35% sulphuric acid, and again removed from the ether by shaking with sodium bicarbonate solution. The coloured solution so obtained is compared with standard carmine solutions in a Walpole comparator (cf. Michaelis, "Praktikum der physikal. Chem."), using tubes containing alkaline methyl-orange solution to compensate for the varying quantities of yellow pigment derived from the flour. In the case of wheaten flour concentration by flotation on chloroform is inapplicable, but a modified method is described in which the flour is extracted with a larger quantity of sulphuric acid present. The standard carmine solutions must be specially prepared for the particular method used or corrections must be applied. Prolonged storage of the flour does not influence the results to an important extent.

W. J. BOYD.

Serological determination of ergot in flour. F. S. OKOLOV and I. G. AKINOV (Z. Unters. Lebensm., 1929, 57, 72—76).—Rabbits were immunised to aqueous ergot extracts prepared by extracting 1 pt. of ergot with 10 pts. of normal saline. An antigen without marked poisonous properties was prepared by extracting the material first with water for 24 hrs., drying it, and then extracting it with alkaline normal saline for 24 hrs., the extract being finally neutralised and filtered. By the method of Uhlenhuth slightly modified a titre of 1:2000 was obtained which was further raised to 1:20,000 by Raishi's method of repeating the immunisation after two months or more. The precipitin reaction was applied by means of the ring test, using 0.2 c.c. of immune serum to which 0.5 c.c. of the extract under examination was added. The tubes were incubated for 25 min. at 37° and 35 min. at room temperature. The extract of the flour was prepared by extracting 20 g. of the material with 100 c.c. of normal saline for 24 hrs., a control extract being made at the same time from ergot-free flour. The specificity of the reaction in relation to the seeds of various weeds was studied, but although non-specific reactions were obtained with certain of these they were not detectable with flour containing only 3% of the seeds. The development of the reaction as a quantitative method is being continued.

W. J. BOYD.

Detection of oxygenated salts in flour. NEUMANN and SCHEYER (Landw. Jahrb., 1928, 68, Suppl. I, 330—331; Chem. Zentr., 1929, i, 164).—For the detection of per-salts the benzidine reaction is employed; for bromates, reduction to bromide and treatment with chlorine; and for oxidising agents generally the oxidation of hydrogen iodide.

A. A. ELDRIDGE.

Distinguishing wheaten flour and bread from those of rye. M. WAGENAAR (Z. Unters. Lebensm., 1929, 57, 37—45).—Microscopical methods involving the use of polarised light are described.

W. J. BOYD.

Rice husks in bran and sharps. A. J. AMOS (Analyst, 1929, 54, 332—333).—Rice husks (the glumes and palea of the fruit) are light brown, stiff, and hard, with dull outer and shiny inner surfaces. A normal bran has a fibre content of 7—10% and sharps 4.5—6.5, but rice husks contain about 40% of fibre. Rice

husks may readily be detected under the microscope after boiling the sample with chloral hydrate solution, for the cells of the outer epidermis are arranged in longitudinal rows, are square in general outline, and have very sinuous side-walls. Dagger-like hairs or hair scars are also to be seen.

D. G. HEWER.

Milk of East Friesian milch sheep during lactation. A. BURR (Landw. Jahrb., 1928, 68, Suppl. I, 178—179; Chem. Zentr., 1929, i, 163).—The milk of a 4-year-old sheep contained 6.44%, and of a 1-year-old sheep 5.35%, of fat. The milk was high in dry matter; the acidity, density, and refractive index of the fresh milk were higher than for cows' milk. The lactose was at first 5%, but fell to 4% shortly before drying-off.

A. A. ELDRIDGE.

Relation of food to the composition of milk. R. O. DAVIES and A. L. PROVAN (Welsh J. Agric., 1928, 4, 114—121).—Grazing generally increases the total protein, casein, total phosphorus, inorganic phosphorus, and calcium in the milk; the changes depend on the previous winter feeding. The flow of milk increased simultaneously.

CHEMICAL ABSTRACTS.

Inoculation of pasteurised milk [with lactic acid bacteria]. A. WOLFF (Milch. Zentr., 1928, 57, 277—283, 293—297, 341—346, 357—364; 58, 105—110, 157—160).—The bacterial flora of pasteurised milk can be favourably influenced by addition of lactic acid bacteria before or after pasteurisation. Pasteurised milk in practice contains abundant lactic acid bacteria, but pure lactic acid fermentation seldom develops as the surviving strains are attenuated. Addition of raw, sour milk cultures before, or of heated cultures after, pasteurisation produces a suppression of the spore formers, but the best results are obtained by breeding a resistant strain of the common lactic acid bacteria and adding it in pure culture to the milk which is to be pasteurised. Such a strain must be of proved purity and resistance to temperature changes in milk fermentations of definite age. Addition of raw cultures after pasteurisation is dangerous, as the bacteria develop too quickly.

W. J. BOYD.

Gerber's acid-butyrometry as a scientific method of examination of milk. G. KOESTLER and W. LOERTSCHER (Z. Unters. Lebensm., 1929, 57, 48—59).—The values obtained by means of Gerber's acid butyrometer agree much better with the values obtained by exact methods such as the Gottlieb-Röse and Schmid-Bondzynski methods, when the reading is assumed to be g. of fat/100 c.c. of milk instead of g. of fat/100 g. of milk. Corrections for use when the latter reading is adopted are given. The method of operation is described in detail.

W. J. BOYD.

Hoyberg's method for determining fat in milk and cream. A. BURR (Landw. Jahrb., 1928, 68, Suppl. I, 179; Chem. Zentr., 1929, i, 164).—The advantages over Gerber's more rapid method are discussed. The accuracy attainable is: for milk 0.1%, for cream 0.5%.

A. A. ELDRIDGE.

Refraction of milks low in solids-not-fat. G. D. ELSDON and J. R. STUBBS (Analyst, 1929, 54, 318—320).—A continuation of the work on milk analysis (cf. B., 1928, 346) shows that of 2850 samples examined in 1928, in every case except 2 (which were sour milks) a low

value for solids-not-fat corresponds with a low refraction, so that all such low values are due (a) to watering, (b) to the fact that milks naturally low in solids-not-fat do not give a normal refraction of 38 or above, or (c) to a combination of the two causes. D. G. HEWER.

Water content of cheese. A. BURR (Landw. Jahrb., 1928, 68, Suppl. I, 172—173; Chem. Zentr., 1929, i, 164).—Values (%) for fresh and ripe cheese, respectively, are: Limburg 48.47—61.22, 48.90—60.93; Romadur 46.53—63.02, 54.82—61.58; Camembert 56.00—69.55, 54.30—68.12; Brie 42.80—69.82, —; Tilsit 43.73—58.70, 35.90—55.08; Gouda 46.97—56.83, 33.58—56.27; Edam 44.80—56.53, 38.00—45.17; Holstein 35.80—58.00. A. A. ELDRIDGE.

Analytical differences between acid and rennet caseins. C. PORCHER and J. BRIGANDO (Ann. Falsif., 1929, 22, 153—163; cf. B., 1928, 543).—The large number of "acid caseins" found in commerce are produced owing to deviations from the method of preparing pure casein which calls for a slow precipitation to bring about controlled but total solubility of the di- and tri-calcium phosphates and of the casein-calcium, a low temperature, and the use of very dilute acid to keep the reaction at p_H 4.6. Lactic caseins result from acidity induced by lactic acid, and acid caseins by the use of mineral acids. Rennet casein is a double calcium salt—calcium paracasein with di- and tri-calcium phosphates—the mineral ash of which should, in practice, be kept at 7.2—7.5%. It results from the action of ferments on the calcium caseinate, the paracaseinate formed carrying down at the same time the insoluble calcium phosphates. D. G. HEWER.

Formation of acid and alcohol in fruit residues and its significance in the nutrition of utility animals. E. CRASEMANN (Mitt. Lebensm. Hyg., 1928, 19, 371—378; Chem. Zentr., 1929, i, 455).—The formation of acetic acid is wasteful, that of lactic acid desirable; the formation of alcohol, for which the material is suited, is not undesirable. The residues are best preserved with sodium benzoate. A. A. ELDRIDGE.

Detection of regenerated preserved peas. O. LÜNING and E. BEYER (Z. Unters. Lebensm., 1929, 57, 76—85).—Fresh green peas which have been preserved directly by heat sterilisation in closed containers can be distinguished from dried fully-ripe peas which have been swelled by soaking in water before being introduced into the containers, by their moisture content. The moisture content of the former lies between 79.0% and 85.6%; that of the latter between 72.7% and 77.6%. The observation of Froidevaux (B., 1927, 91) that the former have a lower content of water-soluble nitrogenous substance calculated on the dry weight was not confirmed. W. J. BOYD.

Determination of the degree of ripeness of peas. E. LASAUSSE, B. GUÉRITHAULT, and PELLERIN (Bull. Sci. pharmacol., 1928, 35, 575—579; Chem. Zentr., 1929, i, 455).—An attempt to discover chemical differences in the preserved products. A. A. ELDRIDGE.

Examination of marzipan. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1929, 57, 85—90).—Different kinds of almond oil give the Bellier and the Kreis reactions in different degrees. The intensities of these reactions diminish with ageing of the material, a

negative result being obtained for both after six months. The Kreis reaction is, however, useful in detecting apricot-kernel oil in marzipan. The latter should be considered adulterated when the Kreis reaction is equal in intensity to that given by a mixture of 15% of apricot-kernel oil in an almond oil which gives a negative result by itself. W. J. BOYD.

Detection of prohibited vegetable and coal-tar colours in foodstuffs. J. R. NICHOLLS (Analyst, 1929, 54, 335; cf. B., 1927, 922).—Test 16 has been found to be fallacious and should be deleted, and test 17 has been modified so that the excess of permanganate is reduced after the oxidation by the addition, drop by drop, of sodium sulphite solution. D. G. HEWER.

Packaging and quick-freezing perishable flesh products. II. Packaging flesh products for quick freezing. C. BIRDSEYE (Ind. Eng. Chem., 1929, 21, 573—576).—Causes of deterioration of flesh products during cold storage, requirements of the manufacturer, distributor, and consumer, and relative merits of various kinds of packaging materials and shipping cases are discussed. The advantages of packaging flesh goods before freezing are emphasised. W. J. BOYD.

Relative vitamin-A content of four oriental foods. Relative content of water-soluble vitamin-B in thirty oriental foods. Relative water-soluble vitamin-C content of nine oriental fruits and vegetables. Certain proteins added to mung beans, or to white or red *Sorghum vulgare*, increase the fertility of mice. H. E. SHERMAN (Philippine J. Sci., 1929, 38, 1—7, 9—36, 37—46, 47—67).

Chemical analysis of thirty-seven oriental foods. Calcium, iron, and magnesium content of sixteen Chinese foods. H. E. SHERMAN and T. C. WANG (Philippine J. Sci., 1929, 38, 69—79, 81—82).

Preserved-milk products etc. FINCKE.—See XII. **Composition of mangolds.** FAGAN and WATKIN.—See XVI.

PATENT.

Production of frozen food compositions. HOM-FREZE CORP. (B.P. 302,883, 7.6.28. U.S., 23.12.27).—In the ice-cream mix (particularly water-ices) a small quantity of agar-agar is used in order that overrun may be obtained on freezing. The agar is boiled in about 200 times its weight of water until it darkens, is then mixed with the flavour and a boiling solution of sugar, and the mixture is maintained at about 82° for some time. E. B. HUGHES.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Nitrobenzaldehyde as reagent for organic medicinal substances. H. W. VAN URK (Pharm. Weekblad, 1929, 66, 429—435).—The colorations obtained by evaporating mixtures of the commoner organic medicinal substances with alcoholic solutions of *o*-, *m*-, and *p*-nitrobenzaldehydes in presence of sulphuric acid are tabulated. The three isomerides show little difference in behaviour in general, though in some cases only the *o*-compound reacts. S. I. LEVY.

Preparation of alcoholic potash solution and a test for aldehyde in chloroform using potassium hydroxide. N. VALTASCHKO (Pharm. J. [Russia],

1928, 34—35; Chem. Zentr., 1929, i, 457).—The development of a yellow colour by a particle of potassium hydroxide may be due to the presence of alcohols in the chloroform. A. A. ELDRIDGE.

Detection of carbon tetrachloride in chloroform. J. SIVADJIAN (J. Pharm. Chim., 1929, [viii], 9, 434—437).—Two c.c. of chloroform and 0.5 c.c. of concentrated sodium hydrate solution are carefully added to a few c.c. of a 1% alcoholic solution of pyrocatechol. A pinch of copper powder is added and the mixture is heated rapidly and boiled for 8—10 sec. It is then cooled, 1 c.c. of concentrated hydrochloric acid and 1 c.c. of water are added, and, after shaking, the liquid is filtered. If the chloroform contains not more than 0.25% of carbon tetrachloride the colour of the filtrate is pale yellow to yellowish-brown, with 0.25—0.50% yellowish-orange, and with more than 0.50% it is reddish-purple. Pure chloroform gives no colour.

E. H. SHARPLES.

Colour reactions of sesamin. H. KREIS (Mitt. Lebensm. Hyg., 1928, 19, 385; Chem. Zentr., 1929, i, 458).—Sesamin (0.1 g. in 100 c.c. of petroleum, b.p. 50—100°) gave the following colorations: nitric acid, with or without sulphuric acid, yellow; acetic anhydride and sulphuric acid (Bömer and Winter), red, then green; pyrogallol and hydrochloric acid (Tocher), violet; vanadium and sulphuric acid (Bellier), dark green; formaldehyde and sulphuric acid (Bellier), violet; stannous chloride solution (Soltsien), red; sulphuric acid and hydrogen peroxide (Kreis), green.

A. A. ELDRIDGE.

Preparation and probable constitution of dermatol. I. TELETOV (Pharm. J. [Russia], 1928, 19—23; Chem. Zentr., 1928, ii, 2353—2354).—Bismuth hydroxynitrate is employed for the preparation of dermatol; the constitution proposed by Fischer and Grützner (A., 1894, i, 416) is confirmed. A. A. ELDRIDGE.

Macedonian opium. A. VRGOČ (Arch. Pharm., 1929, 267, 352—370).—The culture, harvesting, and marketing of Macedonian opium is considered in detail. Analyses of representative samples from twelve districts indicate that the morphine content is usually above 10% and may reach 16%. A few samples contained adulterants consisting of dried, baked, and ground-up bread, the fleshy parts of apricots or melons, egg shells, boiled starch, or, less frequently, ground dates.

S. COFFEY.

Essential oil of *Salvia officinalis*, L. V. MORANI (Nuovi Ann. Agric., 1928, 7, 25—40).—The oil of *Salvia officinalis* grown in the littoral regions of the Mediterranean exhibits marked differences in composition, according as it is obtained in spring and summer from the flower heads alone or from the whole flowering plant, or in winter from the non-flowering plant. The oil from the flowering plant contains cineole 32—35, sesquiterpenes about 30, borneol 9—14, camphor and thujone 5—10, esters 2%, whereas the winter oil shows cineole 13—20, sesquiterpenes about 20, borneol 7.5—12, camphor and thujone 20—32, esters 2.2—3.7, terpenic hydrocarbons about 15%. These oils differ from those of other origins by their comparatively low solubility in alcohol and their higher refractive indices. As with the oil from the Eastern Mediterranean attributed

to *S. triloba*, this Italian oil is laevorotatory, and thus differs from the oil of *S. officinalis* from Dalmatia, Spain, France, Germany, etc.; the camphor in the Italian oil is optically inactive. During the winter the oil changes little in composition or properties, but the yield is less in the morning than in the evening and at the same hour is greater in clear than in cloudy weather.

T. H. POPE.

Essential oils of *Magnolia grandiflora*, L. G. TOMMASI (Riv. Ital. Ess. Prof., 1928, 10, 156—160; Chem. Zentr., 1929, i, 401).—Oil from the leaves (two specimens), flowers, young fruiting plants, and bark gave the following values: d_{4}^{20} 0.915, 0.92, 0.902, —, —; $[\alpha]_D^{20} + 1^\circ 32'$, $+ 1^\circ 46'$, $+ 3^\circ 20'$, $+ 1^\circ 35'$, $+ 1^\circ 42'$; $[\alpha]_D$ after acetylation $+ 5^\circ 16'$, $+ 5^\circ 36'$, $+ 6^\circ 50'$, —, —; n_D^{20} 1.5014, 1.5020, 1.5250, 1.5080, 1.5060; f.p. -16° , -16° , -16° , —, —; b.p. 170—265°, 165—275°, 155—260°, —, —; acid value 2.10, 1.90, 1.20, 1.75, 1.80; ester value 30.24, 32.20, 54.20, 32.65, 32.55; acetyl-saponif. value 51.00, 55.50, 75.50, 53.70, 52.20.

A. A. ELDRIDGE.

Report of the Essential Oil Sub-Committee to the Standing Committee on Uniformity of Analytical Methods. II. Physical constants (Analyst, 1929, 54, 335—338).—Apparatus and methods of procedure for f.p. and m.p. are given, with a special method for otto of roses. For determining the b.p. the use of a standardised apparatus and conditions is considered necessary. The apparatus is illustrated and dimensions are given, together with formulæ for temperature corrections. (Cf. B., 1927, 506.) D. G. HEWER.

Adulterations of essential oils, perfumes, and drugs. SCHIMMEL & Co. (Ber. Schimmel, 1928, 39, 41, 45, 50, 55, 61, 71, 83, 104, 113, 114, 118, 129; Chem. Zentr., 1929, i, 311—312).

Alkaloid test for tannins. FEAR.—See XV.

PATENTS.

Preparation of monoalkyl ethers of proto-catechuic aldehyde in addition to vanillin. F. BOEDECKER (B.P. 309,929, 15.12.27. Cf. B.P. 285,156; B., 1928, 360).—*iso*Chavibetol, obtained with vanillin from safrole according to the process of the prior patent, is ethylated, and the resulting 4-methoxy-3-ethoxypropenylbenzene, m.p. 50—51°, b.p. 145—148°/14 mm., is demethylated by heating with methyl-alcoholic or alcoholic sodium hydroxide at 140—150° in a closed vessel. The product is mainly 4-hydroxy-3-ethoxypropenylbenzene, b.p. 145—150°/13 mm. (benzoate, m.p. 89°), which is oxidised in the usual way to the aldehyde. C. HOLLINS.

Preparation of 1-phenyl-2-methylaminopropanol-1 [β -methylamino- α -phenyl-*n*-propyl alcohol]. W., K., L., W., and F. MERCK (E. MERCK) (B.P. 284,644, 12.1.28. Ger., 2.2.27).— α -Bromopropiophenone reacts with aqueous methylamine solution, and the resulting α -methylaminopropiophenone, isolated as hydrochloride, m.p. 188°, or hydrobromide, m.p. 144°, is catalytically reduced to the secondary alcohol (hydrochloride, m.p. 188—189°; hydrobromide, m.p. 200°).

C. HOLLINS.

Manufacture of basic derivatives of substituted quinolinecarboxylic acids [anæsthetics]. Soc.

CHEM. IND. IN BASLE, and K. MIESCHER (B.P. 310,074, 22.12.27. Addn. to B.P. 270,339; B., 1928, 730).—The following compounds, prepared by the process of the prior patent, show increased anæsthetic properties: (a) ethers of 2-hydroxyquinoline-4-carboxylic β -diethylaminoethylamide: cyclohexyl, m.p. 19°; p-methoxyphenyl, m.p. 108°; n-butyl, m.p. 64°; n-propyl, m.p. 63°; allyl, m.p. 57°; n-amyl, m.p. 72°; isoamyl, m.p. 35°; n-heptyl, m.p. 66°; benzyl, m.p. 119°; β -phenylethyl, m.p. 90°; (b) ethers of 2-hydroxyquinoline-4-carboxylic *N*-ethyl-*N*-(β -diethylaminoethyl)amide (prepared from the 2-chloro-compound, b.p. 165°/0.015 mm.); ethyl, b.p. 158–160°/0.02 mm.; isoamyl, b.p. 165–168°/0.01 mm.; methyl, b.p. 150°/0.008 mm.; n-propyl, b.p. 155°/0.008 mm.; n-butyl, b.p. 163°/0.01 mm.; n-amyl, b.p. 175°/0.02 mm.; cyclohexyl, b.p. 185°/0.015 mm.; benzyl, b.p. 192°/0.01 mm.; (c) ethers of the 4'-methylpiperazide (from 2-chloroquinoline-4-carboxylic 4'-methylpiperazide, m.p. 208°): ethyl, m.p. 183°; n-butyl, m.p. 145°. From 2-chloroquinoline-4-carboxylic bis-(β -diethylaminoethyl)amide, b.p. 165–170°/0.01 mm., prepared from the carboxylic chloride and bis-(β -diethylaminoethyl)amine, b.p. 105–110°/8 mm., there are obtained the 2-n-butoxy- and 2-ethoxy-compounds, b.p. 172°/0.008 mm. and 165°/0.01 mm., respectively. 2-Chloroquinoline-4-carboxylic ϵ -diethylaminoethylamide, m.p. 55°, gives a 2-ethoxy-compound, m.p. 74°; 2-chloroquinoline-4-carboxylic β -N-piperidylethylamide, m.p. 147°, gives a 2-n-butoxy-compound, m.p. 93°.

C. HOLLINS.

Agar product. J. BECKER (U.S.P. 1,712,785, 14.5.29. Appl. 7,12.26).—The liquor obtained by cooking with water at constant temperature and pressure previously soaked and washed seaweed (1 lb. per gal. of water) is filtered, agitated, concentrated to remove excess water, and congealed at about 15°; the product is sized and frozen, after which the ice is melted, excess water is drawn off by suction, and the residue is dehydrated.

L. A. COLES.

Reducing the nicotine content of raw tobaccos, semi-manufactured and finished tobacco products.

R. E. ELLIS. From GENERAL-DIREKTION DER OESTERR. TABAKREGIE (B.P. 290,204, 2.5.28).—Previously dried tobacco is heated rapidly to and maintained at above 150° until the required quantity of nicotine has been expelled from it. Apparatus is described.

L. A. COLES.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Method of increasing the printing speed of dichromated gelatin. F. J. TRITTON (Phot. J., 1929, 69, 281–285).—The sensitisation produced by the addition of certain metallic salts to the sensitising baths used in conjunction with dichromate-colloid mixtures is due to the tanning action of the insoluble metal hydroxide or chromate formed in the colloid film, and is therefore largely controlled by the p_H at which the metal hydroxide or chromate is precipitated and the p_H of the colloid. The best results are obtained with a metal, the chromate or hydroxide of which is precipitated at a p_H value slightly above that of the colloid, since under these conditions no general precipitation and

consequent hardening of the colloid film can take place. Practical applications, especially with regard to the use of cerium salts, are described.

J. W. GLASSETT.

Hypersensitising for extreme speed of exposure. K. JACOBSON (Brit. J. Phot., 1929, 76, 314–315).—Methods of hypersensitising and its practical limitations are outlined (cf. B., 1928, 212).

J. W. GLASSETT.

Physical development and nature of the latent image. R. E. OWEN (Phot. J., 1929, 69, 278–280).—The results of A. and L. Lumière and Seyewetz on post-fixation physical development (cf. B., 1924, 579, 732) are confirmed, but their deduction that the latent image cannot therefore consist of metallic silver is shown to be invalid, since these results may be explained by the fact that during fixation the latent-image silver becomes coated with silver sulphide formed from the silver-sodium thiosulphate complex, and will therefore react as silver sulphide in any later treatment. Experiments in confirmation of this showed that the conditions necessary for the deposition of silver upon a fine suspension of silver sulphide in gelatin were the same as those required for post-fixation physical development of the latent image.

J. W. GLASSETT.

Developer fog from metal of tanks. J. F. ROSS and J. I. CRABTREE (Brit. J. Phot., 1929, 76, 346–349).—The fogging properties of three tank developers, one of which contained sodium bisulphite, were studied after storage in contact with a number of metals and alloys under the following conditions: in open containers; in closed containers; in open containers with the metals coupled with metallic silver. Metallic zinc differed from the other metals causing fog in that a fogging effect was only produced with the bisulphite developer and was probably caused by the formation of sodium hyposulphite by reaction between zinc and the bisulphite. Copper and its alloys caused strong aerial fogging properties, whilst tin and its alloys caused strong chemical fog. The fogging effects were minimised when closed containers were used, and coupling the metals with silver produced no effect.

J. W. GLASSETT.

Magnesium light as a standard source in photographic sensimetry and its relation to the international sunlight standard. J. M. EDER (Z. physikal. Chem., 1929, 141, 321–331).—Light from burning magnesium leaf filtered through solutions of cobalt and copper salts is recommended for photographic sensimetry in the case of both negatives and positives. When the solutions are made up in the proportions given, such light possesses the same spectral energy distribution as that of a full radiator at a "black body" temperature of 5000° Abs. A pure carbon arc closely approximates to magnesium as regards its black-body temperature, and is in practice more convenient. A table is given showing the relation of the colour range of other light sources of different temperatures to that of burning magnesium: this comparison is of importance in determining times of exposure for panchromatic plates and in three-colour photography. Figures are given to show the relative colour sensitivity of a number of different types of paper.

F. L. USHER.

Tentative hypothesis of the latent image. II. A. P. H. TRIVELLI (Z. wiss. Phot., 1929, 26, 381–392).—See B., 1928, 212.

PATENTS.

Manufacture of light-sensitive materials. S. SOKAL. From KALLE & Co. (B.P. 311,196, 7.1.28).—An aqueous solution of methyl cellulose is used as the vehicle for light-sensitive substances of the diazo-type. A slightly acid solution of the diazotised amine together with a coupling agent or a diazo compound which bleaches in light is admixed with the aqueous solution of methyl cellulose and coated on a suitable base. In the first case gaseous ammonia may be used as the developer, and in the second an alkaline alcoholic solution of a coupling agent. J. W. GLASSETT.

Photomechanical printing. F. II. HAUSLEITER (B.P. 280,220, 3.11.27. Ger., 4.11.26).—By using a hardening developer the dots or lines of the screen negative are embedded in hardened gelatin, thereby controlling the action of the subsequently applied photographic reducer which is used to etch the dots or lines for correction of tone or colour value. Alternatively, a positive is made by general blackening of the dry plate, resensitisation with potassium dichromate, exposure behind the screen negative, and, finally, treatment with the reducing agent, the action of which is modified as in the first case. J. W. GLASSETT.

Production of natural-colour [photographic] pictures. R. GSCHÖPF (B.P. 311,833, 27.4.28. Addn. to B.P. 279,381; B., 1928, 833).—An ordinary positive of the first part-negative is produced in an emulsion coated on a non-permeable, white, opaque celluloid film, and is converted into any suitable mordant and dyed. The second colour-image is superimposed by the iron-silver process, and the third added in a similar manner. Alternatively, the third image may be produced on a separate stripping emulsion, weakly dyed, transferred in register on to the others, and then further dyed until correct colouring is obtained. J. W. GLASSETT.

Colour photograph and its production. W. V. D. KELLY, Assr. to M. B. DU PONT VITACOLOR CORP. (U.S.P. 1,712,439, 7.5.29. Appl., 24.5.20).—A dye-absorbing image and a reduced silver image are formed in the same colloid film; the former is then dyed and the latter tinted in different colours. J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Permitted explosives in coal mines. NATHAN.—See II.

PATENT.

Quick-burning delay powder. W. T. INGRAHAM (U.S.P. 1,716,313, 4.6.29. Appl., 8.11.23).—The powder contains a mixture (9:1) of mercury fulminate and black powder. H. ROYAL-DAWSON.

XXIII.—SANITATION; WATER PURIFICATION.

Changes in sulphur compounds during sewage treatment. A. L. ELDER and A. M. BUSWELL (Ind. Eng. Chem., 1929, 21, 560—562).—The sulphur present in sewage is shown to be mainly of non-fæcal origin. During treatment under aerobic conditions there is little change in the total sulphur content, though the immediate oxygen demand observed during experiments on pure compounds indicates that sulphides and thio-sulphates are completely oxidised to sulphates within five days. Under anaerobic conditions sulphates are

reduced to hydrogen sulphide, with possibility of aerial nuisance unless the sewage is kept alkaline and at as low a temperature as possible. C. JEPSON.

Activated carbons and their use in removing objectionable tastes and odours from water. J. R. BAYLIS (J. Amer. Water Works' Assoc., 1929, 21, 787—814).—Chlorine and phenolic substances, or both, may be adsorbed by activated carbon, and filtration through this material is suggested as a final treatment for drinking water in order to prevent the occurrence of any objectionable tastes or odours, of which they are a common cause. The cost of such treatment is estimated to be about \$1.5 per million gallons. Very highly polluted water may be rendered potable by treatment which would include super-chlorination and removal of residual chlorine by carbon, in those regions where, owing to scarcity of supply, the cost of treatment is not a vital factor. C. JEPSON.

Copper from mine waters. EBBUTT and SELNES.—See X. **Determination of nitrates in water.** DE NARDO.—See XVI.

PATENTS.

Sewage-disposal apparatus. M. B. TARK, Assr. to LINK-BELT Co. (U.S.P. 1,714,739, 28.5.29. Appl., 30.3.27).—A settlement tank is divided into a series of narrow compartments, the dividing walls of which are submerged and tapered at the top to facilitate collection of solid matter on the floor of the tank, whence it is removed by a travelling scraper into a hopper situated at the end of each compartment. C. JEPSON.

Sewage-treatment plant. J. T. TRAVERS, Assr. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,715,438, 4.6.29. Appl., 25.3.27).—The plant consists of detritus and sedimentation tanks, the latter being suitable for chemical precipitation, and drainage beds for the disposal of the sludge produced. C. JEPSON.

Manufacture of [veterinary] disinfectants. E. A. GARBE (B.P. 311,920, 25.4.28).—Tobacco is extracted with salt solution, preferably in the presence of sulphur, and, after fermenting for 8—14 days, the extract is mixed with glacial acetic acid and sodium cresolate solution, turpentine oil etc. being added, if desired. The product, diluted with 5—7 pts. of water, is sprayed, alone or together with sulphur dioxide, into stables to prevent the spreading of anthrax, glanders, whitlow, etc. L. A. COLES.

Destruction of vermin and their brood, also injurious germs, parasites, etc. A. VONDRAN (B.P. 311,668, 8.2.28).—Verminous garments, hides, foodstuffs, etc. are treated with a humid atmosphere at a temperature about that of human blood heat, and subsequently with dry air or other gases at 80°.

L. A. COLES.

[Boiler] scale solvent and scaling composition. J. M. COUGHLIN, Assr. to SCALE SOLVENT AND PRODUCTS Co. (U.S.P. 1,715,337, 28.5.29. Appl., 21.1.27).—A solution (*d* 1.40—1.52) containing soluble glass, coffee extract, and a diluent is used.

H. ROYAL-DAWSON.

Oxides of sulphur for disinfecting (B.P. 312,746).—See VII. **Germicides etc.** (B.P. 311,218 and 311,253).—See XI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 16, 1929.

I.—GENERAL; PLANT; MACHINERY.

Mass circulation in carbon dioxide refrigerating machines. H. MAWSON (Inst. Mech. Eng., June, 1929. Advance copy. 33 pp.).—The relative advantages of various compression cycles in carbon dioxide refrigerating machines are discussed. It is shown that both compound compression and multiple-effect compression cycles give greater refrigerating effects than is given by the ordinary single-acting cycle operating with the same temperature of the cooling water and between the same limits of temperature. It is concluded that this is mainly due to the greater mass circulation to the condenser and only slightly to the effects of gas cooling. The coefficient of performance is also higher, but the refrigeration per unit of power can only be increased by making more expensive machines than the ordinary double-acting compressor giving the same refrigeration.

C. J. SMITHELLS.

Jena glass filtering crucibles. R. H. SIMON (J. Assoc. Off. Agric. Chem., 1929, 12, 209—211).—Glass filtering crucibles with fixed plate of porosity less than $4-5\mu$ have been used successfully in determinations of phosphorus and potassium. Ammonium phosphomolybdate precipitated by the method of Ames and Bollz (cf. Ohio Agric. Exp. Stat. Bull. No. 285, 1915, 206) can be collected rapidly with little suction and readily redissolved for reprecipitation or titration. Larger porosity has been used for potassium chloroplatinate. The crucibles can be readily cleaned and restored for use.

W. J. BOYD.

See also A., July, 774, **Topochemical reactions** (KOHLSCHÜTTER). 785, **Viscosimeter** (VAN LINGE).

PATENTS.

Furnaces. AMER. ENGINEERING Co. (B.P. 297,094, 2.4.28. U.S., 15.9.27).—Those portions of the side walls of a mechanical stoker that are subjected to the abrasion and clinkering action of the fuel are constructed of water-cooled refractory blocks. B. M. VENABLES.

Furnaces. A. SMALLWOOD and J. FALLON (B.P. 312,391, 25.2.28).—The heating gases after leaving the work chamber (or flues round the muffle, if the furnace is of that type) pass through one of two alternative paths. In the stage of heating up they are sent upwards through flues in the hollow arch of the furnace, thus obtaining quick heating, and afterwards downwards to recuperators below the furnace, thus obtaining fuel economy. These paths are controlled by dampers in separate exit flues. B. M. VENABLES.

Utilisation of pulverulent or powdered carbonaceous fuel in boiler furnaces. J. J. C. BRAND and B. LAING (B.P. 313,225, 19.3.28).—A tubular air heater

is placed in the return flue of a Lancashire or Cornish boiler, and air blown through this by a fan is supplied as primary and/or secondary air to the same boiler. The primary air may go to the burner or to the pulveriser. B. M. VENABLES.

Preventing foaming and entrainment of liquid in steam generators. "EUKAMA" EIS- U. KÜHLMASCHINEN GES.M.B.H. (B.P. 291,343, 29.5.28. Ger., 28.5.27).—Steam formed in a steam generator is raised to a slightly higher pressure (e.g., by a small turbo-blower), and caused to blow upon the boiling liquid in the same boiler, approximately evenly over the whole surface thereof. If a group of boilers is available, it is preferable to work one of them at a slightly higher pressure than the others and use its steam for the foam-preventing blast. B. M. VENABLES.

Non-heat-conducting coverings for steam pipes etc. WÄRME- U. KÄLTESCHUTZ G.M.B.H. ALTHOFF & SCHOENAU, and P. ALTHOFF (B.P. 313,364, 13.8.28).—Heat-insulating material of known composition is attached to pipes etc. by a skeleton comprising strip-iron brackets supporting iron rings, which in turn support wire netting. A concrete sheath may be formed on the skeleton, and other better insulators placed in the space between the concrete and the pipe. B. M. VENABLES.

Heat accumulators. RUTHSACCUMULATOR AKTIEBOLAGET (B.P. 298,601, 15.9.28. Ger., 12.10.27).—A tall steam accumulator is provided with an auxiliary circulation, for use when discharging steam, which prevents the difference in temperature due to the static head of the column of water in the accumulator. A gaseous or vaporous medium, preferably a minor quantity of steam, is injected through a number of fine jets at the bottom of a circulation guiding pipe. This fitment is preferably separate from the main injection device which is used when charging the accumulator. B. M. VENABLES.

Heat exchangers. W. H. OWEN (B.P. 312,702 and 313,114, 6.3.28).—(A) Cells for use in the construction of heat exchangers are formed from two metal sheets only slightly separated and joined round their edges. The major part of one or both sheets is corrugated, but an uncorrugated border is left all round for use in forming the inlet and outlet passages and for the attachments necessary to build up the complete interchanger. If both plates are corrugated the peaks of the corrugations may be opposite each other or staggered as desired. (B) In a heat exchanger as above, in which the fluids move in opposite directions, the corrugations on adjacent plates are transverse to each other, and the ridges may touch at the points of intersection. B. M. VENABLES.

Apparatus for treating materials, particularly for the heating and drying of pasty masses. M. MCGUINNESS (B.P. 312,699, 11.2., 9.3., and 25.9.28).—A number of polygonal (say four-sided) drums with concave faces are arranged in a row with parallel axes so that the edges of one drum when rotated 90° will scrape the concave faces of its two neighbours which are held stationary. Transfer of material is produced by alternately moving the odd-numbered and even-numbered drums of a row. The row of drums need not be in a straight line, but may turn at right angles as often as desired. The containing casing may either have an undulating form closely following the edges of the drums or be of simpler form with large clearances. In the former case escape of evolved gases may be permitted through slots cut in the ends of the drums, the slots being kept clear by corresponding fixed studs.

B. M. VENABLES.

Drying devices. N. TESTRUP, O. SODERLUND, T. GRAM, and TECHNO-CHEM. LABS., LTD. (B.P. 312,964, 1.3.28).—A dryer for material which is suspended in air or in a gas and is dried by heat transmitted from surfaces over which it passes is constructed so that the material continuously impinges on the heating surface throughout its length of travel. The stream of air and material may pass through tubes which are provided with "rifling," so that the material travels in close proximity to the walls owing to centrifugal force. The "rifling" is preferably formed so that the thickness of the tube is not increased anywhere, *e.g.*, by drawing the tubes in non-circular shapes and at the same time twisting them. It is considered to be insufficient to whirl the stream merely at the beginning of its travel.

B. M. VENABLES.

Pulveriser. J. MOLZ, Assr. to BABCOCK & WILCOX Co. (U.S.P. 1,714,132, 21.5.29. Appl., 30.6.23. Ger., 14.8.22).—The pulveriser comprises one or more pulverising units and a double fan on one shaft. One part of the fan draws air and ground material through the pulveriser, the other part creates a partial vacuum at the end of the casing, so that every point within the casing is below atmospheric pressure and no dust can escape. Incidentally the two fans have a common outlet so that there is an excess of air to effect transport of the material.

B. M. VENABLES.

Pulverising mill. S. DUVALL (U.S.P. 1,713,957, 21.5.29. Appl., 20.6.28).—An impact pulveriser has two staggered groups of hammers each side axially of a set of radial arms, and all being on the same shaft and in the same casing. The radial arms are twisted alternately in opposite ways so as to form propellers which force the material alternately towards each group of hammers.

B. M. VENABLES.

Beaters for impact pulverising mills. HARTSTOFF-METALL A.-G. HAMETAG (B.P. 305,469, 29.1.29. Ger., 4.2.28).—The beaters are of special toothed form, with the object of preventing useless sliding of material along them and cushioning of the blows. B. M. VENABLES.

Centrifugal air-float pulveriser. O. OLSTON (U.S.P. 1,713,297, 14.5.29. Appl., 26.3.28).—In a pulveriser comprising a stationary breaker-ring with co-operating impeller rotated by a vertical shaft, the

breaker-ring is spaced apart from the containing casing, leaving passages for currents of air-borne ground material, and the feed material is guided to the centre of the impeller by means of a hopper-shaped diaphragm above, which also acts as a baffle to create the exhaust draught. A number of grinding units, each comprising the above-mentioned three parts, may be superposed in one casing. B. M. VENABLES.

Impact mills. DEUTS. BABCOCK & WILCOX DAMPKESSEL-WERKE A.-G., and L. KOLLBOHM (B.P. 306,848, 25.2.29. Ger., 23.2.28).—In a pulveriser where the material is blown in by compressed air, this being the sole means of causing impact, the apparatus is maintained under partial vacuum (say 50–80 mm. of water) by means of a suction blower. B. M. VENABLES.

Disintegrating apparatus. J. E. KENNEDY (U.S.P. 1,714,312, 21.5.29. Appl., 31.3.24).—A main body of air is kept in circulation through a revolving drum (*e.g.*, a ball mill), a fan, a cyclone separator, and back to the drum. Excess air, due to leakage, is drawn off between the separator and the mill through a chamber with flexible screening walls, from which the dust may be dislodged by shaking. B. M. VENABLES.

Grinding or crushing mills. M.A.G. MASCHINEN-FABR. A.-G. GEISLINGEN (B.P. 300,213, 8.11.28. Ger., 8.11.27).—A disintegrator is provided with a revolving grinding member which also acts as a fan. The air-borne ground material passes over deflectors to a screen where oversize is taken out; the undersize passes up an ascending pipe provided with annular baffles which cause eddies by which another size of product is removed. The dusty air is exhausted through a filter sleeve surrounding the ascending pipe, and there is also a passage from inside the filter leading back to the disintegrator, to relieve the pressure on the filter. B. M. VENABLES.

Separation of the powder from granular or like material. K. ORTMANN (B.P. 309,361, 13.8.28).—The comminuted material falls in thin streams over the surfaces of deflectors arranged in staggered rows. The deflectors are of Λ -shape in section, but longitudinally the lower edges are cut away and provided with closely adjacent under plates. Suction draughts are produced underneath the ridges, the air and dust exhausting through passages in the walls of the casing at the ends of the ridges. B. M. VENABLES.

Centrifugal concentrator. R. M. WENDEL (U.S.P. 1,712,184, 7.5.29. Appl., 19.12.27. Swed., 7.10.26).—A centrifugal separator for solids suspended in a liquid comprises a vertical, tapering, rotary drum the small end of which is downwards, has a central opening, and dips into the feed pulp. The conicity of the drum causes the material to rise as well as stratify, and each stratum is drawn off at the top by conical dividing plates which are within and parallel to, and rotate with, the drum. One form of the apparatus may have two concentric rotary drums, the outer one receiving partly separated product from the inner.

B. M. VENABLES.

Separation of impurities from circulating air, gas, or vapour [by "electret" filter]. PLATEN-MUNTERS REFRIGERATING SYSTEM ARTIEBOLAG (B.P.

292,479, 18.6.28. Ger., 18.6.27).—The gas is passed through filters or over contact surfaces made of insulating substances (e.g., carnauba wax) which have been melted and solidified while in a strong electric field, whereby they obtain a charge which is retained for long periods. The "electrets" may be divided into grains or powder and attached to fabric or other supports if desired, and may be washed or disinfected without losing their charge.

B. M. VENABLES.

Apparatus for centrifugal separation. C. E. FAWKES (U.S.P. 1,700,928, 5.2.29. Appl., 25.8.22).—The mixture, e.g., liquid air, is charged through Venturi nozzles into the middle compartment (A) of medium centrifugal pressure in a series of separating chambers mounted vertically and containing rapidly rotating discs. The heavier component, e.g., oxygen, is thrown out into a collecting chamber connected with the region of medium centrifugal pressure in the next lower separating chamber (B), while the lighter components, e.g., nitrogen, are drawn off from their axial position to the region of medium pressure in the next higher separating chamber (C). Lighter components separated in C are drawn back upwards into A with fresh air supply through the Venturi nozzle, while heavier (oxygen) components from B are similarly returned downwards, relatively pure nitrogen being ultimately delivered at the top and oxygen at the bottom of the apparatus.

R. BRIGHTMAN.

Continuous filter. J. D. COMPAIN (U.S.P. 1,712,258, 7.5.29. Appl., 28.11.27. Cuba, 21.12.26).—A rotating cylindrical strainer or filter, to the interior of which the prefilter is supplied, is provided with radial lifting vanes on the interior circumference; the vanes are hinged about lines intermediate their radial depth, and work in conjunction with fixed circular guides, which, during part of a rotation, bend the vanes to form buckets which fall open at the highest point of their travel, and tip collected refuse into a discharge hopper.

B. M. VENABLES.

[Metallic edge]-filters. J. A. PICKARD (B.P. 312,944, 2.12.27, 28.1. and 1.8.28).—An edge-filter is constructed of non-fibrous plates in such a way that the passages for the flow of liquid become thinner in the direction of travel. The metallic discs or strips are formed with a taper for this purpose, and may be provided with embossed studs or ribs to maintain the correct distance apart. Filter aids may be used. Cleaning by reverse flow is facilitated by the taper of the passages.

B. M. VENABLES.

Filter presses or pressure-filter elements. L. A. J. REVEL (B.P. 292,117, 15.3.28. Fr., 14.6.27).—A form of construction for filter plates is described whereby both inlet and outlet plates are identical in manufacture, but are assembled at right angles to each other.

B. M. VENABLES.

Testing and indicating the condition of filters. E. C. R. MARKS. From A.C. SPARK PLUG CO. (B.P. 313,179, 8.12.27).—Used motor oil or other fluid under pressure is passed in turn through a constriction and the filter. Leads to pressure gauges are taken off before and after the constriction; as long as the filter is

passing oil there will be a difference in the readings of the gauges, but when the filter is completely choked the readings will be equal. Suitable arrangements of gauges are described.

B. M. VENABLES.

Apparatus for determining gas content of solid bodies. C. LORENZ A.-G., Assees. of W. HESSENBRUCH (B.P. 299,303, 22.9.28. Ger., 22.10.27).—A furnace with air-lock device preferably consists of a graphite crucible within a magnesia crucible, the whole being enclosed in a silica container and heated by electrical or other means. The silica container is connected in an air-tight manner, through a valve large enough to pass the material, to a material feeding chamber and to a vacuum pump. In operation, firstly, the whole empty apparatus is subjected to vacuum and the crucibles are heated; after the gas has been removed, the crucible valve is closed, the vacuum released (but not in the crucible owing to the closed valve), the feed chamber charged with material, and the vacuum restored. When fully restored, the crucible valve is opened and the material dropped into the crucible, the gases evolved being collected and assayed by known means.

B. M. VENABLES.

Apparatus for heating liquids by steam or the like. R. MORTON & Co., LTD., and P. ROBINSON (B.P. 313,345, 11.7.28).—When a mass of liquid is heated by a submerged heater, such as a bundle of steam tubes, the heater is surmounted by a hood within which the convection currents are concentrated. The hood may be provided with doors, or may be entirely swung aside or lifted out so that the heater may be cleaned.

B. M. VENABLES.

Distilling liquids by introduction of another liquid into the heated liquid to be distilled. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,254, 10.4.28).—When, for example, peanut oil is heated at 280° *in vacuo*, and it is desired to introduce water (without steam) at 175°, the water is supplied under a pressure of 8 atm. through a device comprising a double concentric tube to the inner of which water is fed, and from the outer tube part of the water is exhausted at the same end. At the further end, where the water turns back, is situated a finely-adjustable valve which permits some of the water to pass out into the liquid to be distilled. The returning water insulates the feed water from the excessive heat of the oil in which the device is submerged.

B. M. VENABLES.

Apparatus for the distillation and other hot treatment of liquids. A. E. BIANCHI and G. GUARDABASSI (B.P. 312,399, 27.2.28).—In apparatus comprising externally-heated retorts provided with stirrers and chain agitators, the shafts of the stirrers are made hollow, and the incoming liquor is passed through them in series; the liquor is thereby preheated and the shafts are kept cool. A loaded valve may be used to regulate the pressure and rate of flow of liquid, which issues in the form of a spray to the interior of the retort (cf. B.P. 277,404 and 278,041; B., 1927, 930).

B. M. VENABLES.

Desiccation of liquids. J. A. REAVELL (B.P. 312,400, 27.2.28).—In the process of desiccation by spraying into heated gas, the bulk of the powder is

recovered from the lower part of the spraying chamber: the exhaust gas, which contains dust in suspension, is scrubbed by the liquor before desiccation, the liquor preferably being kept in comparatively rapid counter-current circulation through a tower, while a smaller proportion (the net feed) is sent forward to the spray. If desired, an intermediate step of ordinary evaporation may be interposed. B. M. VENABLES.

Sterilisation of liquids. C. A. HARTUNG (U.S.P. 1,714,366, 21.5.29. Appl., 8.5.26. Ger., 25.3.26).—The liquid is repeatedly circulated through a heat inter-changer, the heating fluid being maintained slightly hotter than the liquid at all points in its course.

F. G. CLARKE.

Apparatus for spraying or atomising liquids. C. E. GARDNER (B.P. 313,212, 9.3.28).—The apparatus comprises one or more mixing chambers, terminated by one or more spraying nozzles. The liquid is supplied under pressure through a lateral inlet to the first mixing chamber, and air or other fluid under pressure to an opposite lateral inlet, and a plug cock with two separate passages is situated between the three last-mentioned parts.

B. M. VENABLES.

Drying of gases. O. Y. IMRAY. From ALLGEM. GES. F. CHEM. IND.M.B.H. (B.P. 312,457, 18.4.28).—The gases are dried by contact with a liquid such as sulphuric acid. The apparatus comprises a vertical casing in the lower part of which is a bundle of tubes forming a shallow liquid seal in a bath of the absorbent liquor. The gas is blown through the tubes in the reverse way to an ordinary liquid seal, and in consequence gas and finely-divided spray ascend through the tubes to a plain intermediate chamber, and thence to an upper chamber with filling such as Raschig rings, which form a spray trap. Liquor drains back to the base chamber through one or more pipes of large bore forming deep liquid seals, and is circulated by a pump or air lift to the top of the filling. Part of the liquid is removed for reconcentration.

B. M. VENABLES.

Regulating the density of mixed gases. SMOOT ENGINEERING CORP., Assees. of C. H. SMOOT (B.P. 284,647, 23.1.28. U.S., 2.2.27).—This invention is described as used for keeping the carbon dioxide content of boiler gases constant; it is also suitable for other purposes. A single motor drives two equal suction fans which are fed through restricted inlets with air and with flue gases, respectively, after each has passed through a saturator and temperature equaliser; the fans are also water-jacketed as a further precaution. The air may then be taken as a standard of density, and the relative density of the flue gas will only be affected by its chemical composition, *i.e.*, its carbon dioxide content. The exhaust from the fans passes freely to atmosphere, but from points that may be accurately adjusted with regard to their distance from the axes of the fans other pipes lead to above (for air) and below (for gas) a bell floating in liquid, the position of which through other pneumatic mechanism regulates the air and/or fuel supply to the boiler furnace.

B. M. VENABLES.

Treatment of noxious and other fumes. L. S. FRASER and F. L. RICH (B.P. 312,200, 19.12.27).—

Exhaust gas from an engine (or other gas) passes through an ejector device which draws in and mixes a quantity of air or oxygen, and the mixture passes on to an enlarged part of the apparatus where it passes over a catalyst such as copper or other metallic oxide. The apparatus may be heat-insulated, and the air inlets may have non-return valves. B. M. VENABLES.

Apparatus for indicating the presence of gases. B. TREBITSCH (B.P. 313,355, 26.7.28).—The apparatus comprises a vessel with double, porous walls through which gases will diffuse at different rates according to their density. At one end the walls are rendered non-porous, and here is inserted a tube affording communication between the inner vessel and a manometer, the latter of which may be used to operate electrical contacts. Preferably the inner wall has a greater porosity than the outer. B. M. VENABLES.

Catalytic apparatus [for gaseous-phase oxidations, reductions, etc.]. SELDEN Co., and A. O. JAEGER (B.P. 306,519, 27.4.28. U.S., 23.2.28).—The temperature of reaction is controlled by a non-boiling, circulating liquid the temperature of which is controlled by heat-conducting elements which may be metal rods or metal tubes containing a liquid boiling at about the desired reaction temperature. These elements extend beyond the circulating liquid and come into contact with the reacting or reacted gases. A suitable apparatus is figured. C. HOLLINS.

High-pressure drum and its manufacture. W. H. LEWERS. From SUPERHEATER Co. (B.P. 313,222, 15.3.28).—Since the longitudinal stress in a cylinder under pressure is only half the circumferential stress, a high-pressure drum is formed of two concentric cylinders, the larger being pressed on the smaller. One of them, preferably the inner, is left longer and the ends are swaged down in a conical or other shape to form the ends of the drum. The swaging will increase the thickness of the metal, and for this and mathematical reasons connected with the angle of the conical ends, the inner drum may be even less than half the combined thickness. The cylinders may be formed from sheets, rolled and welded longitudinally, the seams of the separate cylinders being placed well apart circumferentially. Holes for tubes may be drilled of equal diameter through both drums and the joints exposed half way through the holes welded up, or the holes in the inner drum may be drilled slightly smaller than those in the outer, and the tubes expanded into the inner drum only. B. M. VENABLES.

Aeration process for drying, carbonising, and oxidising plants and apparatus therefor. R. GLINKA (U.S.P. 1,715,830, 4.6.29. Appl., 13.10.27. Ger., 18.10.26).—See B.P. 303,572; B., 1929, 307.

Centrifugal separator. B. C. CARTER (U.S.P. 1,714,658, 28.5.29. Appl., 10.12.28. U.K., 28.10.27).—See B.P. 310,550; B., 1929, 498.

Clarification of liquids and recovery of solids therefrom. R. J. MARX (U.S.P. 1,717,111, 11.6.29. Appl., 22.6.27. U.K., 2.2.27).—See B.P. 272,130; B., 1927, 591.

Separation and recovery of gases and vapours by solid absorbents. A. GODEL, Assr. to Soc. DE

RECHERCHES & D'EXPLOITATIONS PÉTROLIFÈRES (U.S.P. 1,717,103, 11.6.29. Appl., 23.6.26. Ger., 8.6.26).—See B.P. 267,369; B., 1927, 689.

Doors for furnaces, retorts, etc. S. R. ILLINGWORTH, and ILLINGWORTH CARBONIZATION CO., LTD. (B.P. 313,021, 29.2.28).

Furnace walls [for steam boilers]. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX CO. (B.P. 312,846, 12.7.28).

[Grooved] filling blocks for heat exchange, reaction, and absorption apparatus. J. H. JACK. From STELLAWERK A.-G. VORM. WILISCH & CO. (B.P. 313,226, 19.3.28).

Reversing mechanism for washing and like machines of the rotary-drum type. A. F. TULLIS (B.P. 312,973, 3.4. and 24.8.28).

Fire-extinguishing liquid (U.S.P. 1,716,476).—See VII. Furnace for fusing finely-divided materials (B.P. 294,639).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Carbonisation. IV. Influence of various factors on ignition temperature, reactivities, and structure of coke. V. Temperature, size of coal, blending with inorganic compounds. 22ND AND 23RD REPTS. OF JOINT RES. COMM., INST. GAS ENG. AND UNIV. LEEDS (Gas J., 1929, 186, 766—770, 772—775; cf. B., 1928, 555).—IV. The ignition temperatures, and reactivities to carbon dioxide and steam, of a number of cokes have been measured by passing 10 litres per hr. of (a) oxygen, (b) carbon dioxide, and (c) steam, respectively, over standard quantities of graded coke in three similar types of apparatus. It is found that (1) the size of the coal carbonised has no marked effect on the ignition temperature or the carbon dioxide or steam reactivities of the coke; (2) the ignition temperatures of cokes decrease as their carbonisation temperature is lowered or as their percentage of volatile matter is increased; (3) addition of coke breeze to a Nottinghamshire coal does not influence the ignition temperatures or reactivities of the resulting cokes; (4) addition of sodium carbonate to the coal lowers the ignition point of the coke produced when the mixtures are carbonised above 600°, but below this temperature it has no effect; at carbonisation temperatures of 915° calcium carbonate and iron oxide also lower the coke-ignition temperature, but their effects are not so marked as is that of sodium carbonate; (5) admixture of sodium carbonate, calcium carbonate, and iron oxide, respectively, to the coal prior to carbonisation increases the reactivity of the coke made; (6) for high-temperature cokes a low ignition temperature corresponds with high steam and carbon dioxide reactivities; (7) certain of the conclusions given above must be modified if the charge of coal is not uniformly carbonised; (8) examination of a number of cokes by macro- and micro-inspection of "Rose's" sections does not give any indication of the possibility of correlation of visible structure with ignition temperature and reactivity.

V. The results previously recorded (*loc. cit.*) obtained when a Nottinghamshire moderately coking coal was

carbonised in 30-lb. charges in an experimental gas plant furnished by a highly coking Durham gas coal, carbonised under similar conditions, are compared. The Durham coal was graded into three sizes, $1\frac{1}{4}$ — $\frac{3}{4}$ in., $\frac{1}{2}$ — $\frac{1}{4}$ in., and 8—30-mesh, and examined at carbonisation temperatures of 920° and 815°. It was found that (a) the size of the coal had no effect on the ultimate yields of gas, coke, and therms; the size of the coal, when carbonised at 920°, affected the rate at which gas was evolved, the mixture of sizes and the smallest grade giving the slower rates; (b) reduction of the carbonising temperature from 920° to 815° lowered the thermal yield and altered the composition of the gas; the volume of gas obtained from the 8—30-mesh coal decreased from 12,007 cub. ft. at 920° to 9959 cub. ft. at 815°, the calorific value increased from 597 to 649 B.Th.U. per cub. ft., and the final thermal yield per ton of coal fell from 71.7 to 64.6; (c) temperature of carbonisation had a marked effect on the rate of gas evolution; after 1 hr. the volume of gas evolved from the coal carbonised at 815° was approx. 50% of that obtained at 920°; (d) the yields of tar, ammoniacal liquor, and coke increased as the carbonisation temperature was lowered; (e) addition of coke breeze (10—30%) increased the rate of gas production (per lb. of coal but not per lb. of charge) at both temperatures, this effect being the more marked with Durham than with the Nottinghamshire coal; (f) the results obtained when calcium carbonate, calcium oxide, iron oxide, and sodium carbonate (2% of corresponding oxide) were added to the coal before carbonisation again indicated the marked effect of these substances on the amount and rate of gas evolution, thermal, tar, ammonia, and hydrogen sulphide yields. C. B. MARSON.

Low-temperature carbonisation of blended New Zealand coals. W. G. HUGSON (New Zealand J. Sci. Tech., 1929, 10, 263—274).—Gray-King assays have been carried out on a number of bituminous and brown coals, and on blends of the two. Low-temperature carbonisation of the former yield swollen friable cokes, and of the latter non-coherent powders, but semi-cokes of a satisfactory texture and hardness are obtained from suitable blends of the two. A. B. MANNING.

Factors influencing the reactivity of coke. F. J. DENT and J. W. COBB (Gas J., 1929, 186, 776—782, 811; cf. B., 1928, 555).—Experimental evidence indicates that the combustible portion of coke is not homogeneous, but is composed of constituents with different properties which can be divided into two groups, and are the decomposition products from two corresponding groups of constituents of the original coal, viz., those which fuse, or do not fuse, when the coal is carbonised. On carbonisation the fusible portion of the coal first melts, spreading over the non-fusible constituents, and on further heating decomposes, with loss of volatile matter, gradually becoming more and more viscous until it finally resolidifies. This "binder" is unreactive compared with the residue from the non-fusible components and lowers the reactivity of the coke towards carbon dioxide and steam at high temperatures; the amount of binder in strongly coking coals is comparatively large and accounts for the low

reactivity of their cokes, whereas anthracites and lignites, which contain only small quantities of binder, when carbonised produce more reactive cokes. The influence of heat on the binder is much more pronounced than on the non-binding material; heating for a few hours at 900° converts the binder into still more unreactive graphitic carbon. When a coke is gasified at high temperatures its more reactive components are gasified more rapidly than is the main body of the material, hence its reactivity decreases as gasification proceeds, but other factors, such as graphitisation of the surface, the presence of reducible oxides, etc., may mask this effect. Reactivities and ignition points determined at low temperatures cannot be regarded as a means of forecasting the behaviour of coke at higher temperatures, and it therefore follows that the reactivity of a coke should be determined under conditions corresponding as nearly as possible with those under which the coke is intended to be used in practice. It is concluded that for the examination of the influence of various factors on the reactivity of coke, a knowledge of the nature of "parent" coal is of the greatest importance.

C. B. MARSON.

Reactivity of coke. II. Metallurgical cokes. J. H. JONES, J. G. KING, and F. S. SINNATT (Dept. Sci. Ind. Res., Fuel Res., 1929, Tech. Paper No. 22, 30 pp.).—Fifty samples of metallurgical cokes made from a variety of coals and in different types of ovens were examined by a method previously described (cf. B., 1928, 146) to obtain evidence of the influence of the coal carbonised and of the carbonising conditions on the reactivities (to carbon dioxide) of the coke produced; an attempt was also made to correlate the shatter indices of the cokes with their reactivities. It was found that, in general, high reactivity corresponded with low resistance to shatter; cokes may be placed in the order S. Wales → Durham → Yorkshire for increasing reactivity and decreasing shatter index; cokes with high shatter indices gave the most regular results, when tested for reactivity. No definite relationship between compression of the charge and change in reactivity or shatter index was observed; beehive coke was characterised by little variation in its reactivity as the experiment proceeded, and by a uniformity of quality. Insufficient data are available to enable conclusions to be drawn as to the blast-furnace value of the cokes, but there are indications that low reactivity and regularity of behaviour are desirable. Preliminary experiments are also described in which the influence of the inorganic constituents, especially iron, has been examined.

C. B. MARSON.

Disposal of liquor effluents from gas works. 3RD REP. INST. GAS ENG. LIQUOR EFFLUENTS RES. COMM. (Gas J., 1929, 186, 783—793; cf. B., 1928, 556).—The installation and trial of a Lodge-Cottrell electrostatic tar precipitator attached to a new setting of continuous vertical retorts is described, and it is shown that the apparatus is highly efficient as a tar separator and reduces the higher tar acids in the liquor to a comparatively small amount; the liquor is consequently much paler in colour, but the monohydric phenol content is increased, as is also the total oxygen-absorption value. Observations show that in the

liquor obtained from the new setting of vertical retorts the low oxygen content of the crude gas causes a diminution in the quantities of thiosulphate and thiocyanate in the condensed liquor. Systematic tests on the method of purifying liquor effluents by percolating filter beds (cf. previous report, *loc. cit.*) are described. From August, 1928, the whole of the spent liquor made at a gas works has been treated on the beds, 1 pt. of spent liquor being diluted with 23 pts. of purified effluent and fed to the beds in rotating sprinklers. In the first 12 weeks 3 million gallons of spent liquor were purified, with an efficiency of 93%; the running costs after 1 year's working were £828 per annum, or 10s. 9d. per ton of ammonium sulphate made. C. B. MARSON.

Economies in the recovery of ammonia [from gas-works' liquor] by the indirect process. H. HOLLINGS and S. PEXTON (Gas J., 1929, 186, 793—796).—The aim in indirect ammonia recovery should be to produce as concentrated a liquor as possible; a suitable washing medium to replace water may be obtained by the fractional condensation of the virgin liquor, and such replacement does not adversely affect the quality of the ammonia-still effluent liquor. The partial or complete elimination of water used in the extraction of ammonia from gas is dependent on efficient cooling of the gas and on the provision of washers of adequate capacity. It is concluded that, in general, the capacity of condensing and washing plant could be increased, if necessary by 25%, at an increased cost of 5s. to 6s. per ton of ammonium sulphate made; this increased cost may be offset by the economies resulting from the production of a more concentrated liquor, without any reference to the other advantages associated with the use of an efficient condensing plant.

C. B. MARSON.

Methods of refining sulphurous oils. C. J. WRIGHT (J. Inst. Petroleum Tech., 1929, 15, 214—244).—The cost of removing hydrogen sulphide from straight-run benzine by soda-washing is materially reduced by maintaining the condenser worm end temperature as high as 32—38°. Soda which has been used for absorbing hydrogen sulphide, and which contains sodium hydrogen sulphide, can be partially recovered by the Koppers aeration process, in which hydrogen sulphide is liberated and sodium sulphide formed. Working to about a 5% recovery with a large volume of liquor, hydrogen sulphide can be alternately absorbed and expelled. The "soda" recovered by this process will not dissolve mercaptans. Soda containing sodium mercaptides, but not sodium sulphide or sodium hydrogen sulphide, can be recovered almost quantitatively by boiling off the mercaptans; the economical limit is reached after about eight recoveries of the same lot of soda. If soda were the only alkali available, it would be economical to segregate it into two distinct cycles for removal of hydrogen sulphide and mercaptans, respectively. It is not economical to recover lime which has been used for removal either of mercaptans or of hydrogen sulphide. For the removal of mercaptans, a given weight of sodium hydroxide is the more efficient the greater the dilution; moreover, more dilute alkali is more readily recovered by boiling. Sodium

sulphide, derived from sodium hydroxide and hydrogen sulphide in refinery practice, is of use in removing elementary sulphur from petroleum. In refining straight-run benzene by means of calcium hypochlorite, the latter is employed in a solution containing 0.9—1.1 g. of lime per litre. The use of sodium plumbite, sulphuric acid, liquid sulphur dioxide, and bauxite is discussed.

W. S. NORRIS.

Determination of tar-forming value of transformer and switch oils. E. LOCHER (Chem.-Ztg., 1929, 53, 470).—The oil is heated at 120° for 24 hrs. with sodium hydroxide solution of specified concentration, instead of for 70 hrs. in a stream of oxygen. S. I. LEVY.

Technical destruction methods of crude oil emulsions. A. LOTTERMOSE and N. CALANTAR (Kolloid-Z., 1929, 48, 179—184).—Existing technical methods of destroying oil emulsion are examined. They are grouped as electrical methods, mechanical methods (centrifugal, thermal, filtration), and addition of various agents including adsorption media (charcoal, fuller's earth, kaolin, etc.), electrolytes, colloids, and the by-products of petroleum refining. It is concluded that there is no universal method, each emulsion requiring its own special type of treatment.

E. S. HEDGES.

Explosibility of atmospheres. COWARD.—See XXII.

See also A., July, 803, **Pyrogenic decomposition of perhydrides of fluorene and acenaphthene under pressure of hydrogen** (ORLOV and BELOPOLSKY).

PATENTS.

Coke ovens. J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,714,933—4, 28.5.29. Appl., 13.4.26).—(A) Each heating wall of a coke oven is provided with vertical flues which communicate with a horizontal flue passing through the upper portions thereof. Ducts connect the horizontal flue in one wall with that in another. (B) An oven with a horizontal flue in each heating wall passing through the upper parts of and communicating with the vertical flues is provided with regenerators arranged to serve the vertical flues on one half of each of the heating walls as up-flow flues, and other regenerators arranged to serve the vertical flues on the other half as down-flow flues simultaneously.

A. B. MANNING.

Producing a solid fuel in coarse lumps from finely-granular anthracite or similar lean coals and bituminous coal. CHEM. TECH. GES.M.B.H. (B.P. 288,235, 13.1.28. Ger., 7.4.27).—Mixtures of finely-divided anthracite or other lean coal and a caking bituminous coal are carbonised by external heating at 500—600° in stationary layers of 6—15 cm. thickness, packed as closely and evenly as possible. The coal is packed into the retorts preferably by means of worm-conveyors.

A. B. MANNING.

Preparing and subsequently briquetting non-caking fuels. E. FRIEDLÄNDER & Co., Assees. of P. FUCHS (B.P. 281,326, 26.11.27. Ger., 27.11.26).—Non-caking fuels, with the exception of low-temperature coke, are briquetted with both an inorganic and an organic binding agent. Sorel cement (6—7%), or lime slaked with magnesium sulphate solution, may be used

as the former, and 2—3% of sulphite-liquor pitch, naphthalene, tar oils, etc. as the latter. A. B. MANNING.

Carbonisation of coal and like material. I. F. LAUCKS, Assr. to OLD BEN COAL CORP. (U.S.P. 1,713,840, 21.5.29. Appl., 23.6.20; cf. Laucks, B., 1927, 177).—A coal which becomes plastic on heating is conveyed through an externally heated cylindrical retort by means of a screw. The latter, which is hollow, is also heated in such a manner that at the zone where the coal commences to set the screw is hotter than the retort, and the setting process is completed at the retort while the plastic coal adheres to the wall of the retort.

A. B. MANNING.

Heating substances susceptible to oxidation. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,713,834, 21.5.29. Appl., 6.7.21. Ger., 13.5.28).—By intimate admixture of the substances with not too easily combustible carbon a reducing atmosphere is maintained in their immediate environment while they are being heated or carbonised, and the necessity for using an excess of fuel gas in the furnace is avoided.

A. B. MANNING.

Treatment of combustible material [for use as pulverised fuel]. H. P. PETERSEN (B.P. 312,707, 6.3.28).—The material is simultaneously dried and pulverised by the passage of hot combustion gases through the mill. The gases then convey the pulverised material to a centrifugal or cyclone separator. The blower is so placed that the gases are drawn from the combustion chamber through the mill and separator, and then, issuing from the blower, serve to inject the fuel, which is discharged from the separator, into the combustion chamber. By drawing the gases from different points of the combustion chamber or smoke passage the temperature in the mill can be suitably adjusted.

A. B. MANNING.

Treatment of lamp blacks and other carbon blacks and their industrial applications. COMP. LORRAINE DE CHARBONS POUR L'ÉLECTRICITÉ (B.P. 300,610, 31.10.28. Fr., 16.11.27).—Amounts up to 10% of pine tar, natural asphalt, etc. are incorporated with lamp black in order to convert it into a pulverulent form which is not dusty. If possible the added material is one of the necessary ingredients of the industrial product for the manufacture of which the lamp black is subsequently to be used. Examples are its uses in rubber compounding, and in inks and paints.

A. B. MANNING.

Gas producers. MORGAN CONSTRUCTION Co., Assees. of P. S. HULT (B.P. 303,784, 20.9.28. U.S., 9.1.28).—A rotary producer is described in which the shell is supported by three radial arms from the blast duct which rises through the centre of the ash pan. This arrangement allows free passage of the ash from the shell into the pan and the free working of an ash plough therein. The radial arms are cooled by the water seal between the ash pan and shell.

J. A. SUGDEN.

Gas producers having means for removal of residues therefrom. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,709, 30.4.28).—The stationary grate is provided with an adjustable rotating stirrer which discharges the ash through one or more

central ash holes. The system is specially suitable for use in conjunction with the process described in B.P. 214,544 (B., 1924, 549). J. A. SUGDEN.

Gas retort. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,716,643, 11.6.29. Appl., 7.7.21. Ger., 1.9.19).—The flue and regenerator system for a horizontal bed is described. J. A. SUGDEN.

Gas absorber for products of combustion. J. BILSKY (J. G. BILLINGS) (U.S.P. 1,716,479 and 1,716,480, 11.6.29. Appl., [A] 9.2.27, [B] 18.5.27).—(A) The absorbents comprise separate layers of active carbon, cuprous and palladium chlorides, hexamethylenetetramine, and an oxidising catalyst. (B) The absorption chamber is provided with a trap to separate liquids from the gas before passing through the absorbent baffle plates, and with a small pump to maintain reduced pressure within the chamber in order to reduce back-pressure. J. A. SUGDEN.

Gas manufacture. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,716,673, 11.6.29. Appl., 8.3.22).—Comminuted coal (with oil as a bond) is compressed into a mass having a number of channels throughout its length. While the mass is in a suitable retort hot gases are passed through the channels, thereby gasifying the volatile products, which are collected in admixture with the hot gases. J. A. SUGDEN.

Manufacture of coal gas. E. REES and G. GARDINER (B.P. 312,727, 19.3.28).—Means are provided for controlling and varying in any desired manner the vacuum or pressure to which each retort in a setting is subjected during the period of carbonisation.

A. B. MANNING.

Continuous oil-gas process. W. S. YARD and E. N. PERCY (U.S.P. 1,703,506, 26.2.29. Appl., 1.12.24).—Air is blown through a deep bed of fuel in a gas producer, at the top of which are oil inlets so arranged that the fuel bed may be continuously and uniformly replenished by the deposition of carbon formed by the cracking of the oil introduced at suitable rates through the inlets. A. B. MANNING.

Manufacture of mixed water-gas and oil gas. W. D. WILCOX (U.S.P. 1,716,676, 11.6.29. Appl., 31.12.24).—A fuel bed is blasted from the top and the gases pass on to be burned in a regenerator. The fuel bed is then steamed from the bottom and the water-gas is collected. Finally, steam and oil, vaporised by passing through the regenerator, are passed through the fuel bed to generate mixed water-gas and oil gas. J. A. SUGDEN.

Manufacture of fixed gases and condensable hydrocarbon fluids from bituminous materials by fractional disintegration. A. G. MEEZE (B.P. 312,533, 10.7.28).—The fuel bed of a water-gas generator is first blown to incandescence with air enriched with pulverised bituminous coal (cf. B., 1912, 1172), the air blast being followed by a run of superheated steam which, instead of being cut off when the production of carbon monoxide falls off, is continued, the gas, however, being then diverted into a storage vessel and subsequently again passed through the fuel bed of the same or another generator which has been reblown to incandescence.

During the second stage of the steam run pulverised bituminous material may be injected into the generator, the gas being then passed through a condensing system for the recovery of liquid hydrocarbons therefrom before being collected for retreatment in the generator.

A. B. MANNING.

Recovery of gases or vapours taken up by absorbents. N. V. NORITVEREENIGING VERKOPF CENTRALE (B.P. 283,508, 11.1.28. Ger., 11.1.27).—The absorbed substance is removed by circulating the scavenging gas at a rate greater than that used during the absorption process. The scavenging gas may be the original gaseous mixture or an indifferent gas charged with steam. A suitable closed system of absorption and condensation vessels is described which gives a greatly increased efficiency and economy. J. A. SUGDEN.

Purifying gases [from distillation of wood]. E. PIRON, Assr. to PIRON COAL DISTILLATION SYSTEMS, INC. (U.S.P. 1,701,054, 5.2.29. Appl., 7.1.25).—The gases are scrubbed first with, e.g., "green liquor" or crude pyroligneous acid, and then at above 100° with sulphuric acid, *d* 1.263, affording on condensation tar-free pyroligneous acid. The residual gases are returned through an exhaustor to heat the retort, a portion being by-passed through a preheater into the sulphuric acid scrubber to maintain the temperature above 100°.

R. BRIGHTMAN.

Purifying the exhaust gases of internal-combustion engines. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 287,106, 28.2.28. Ger., 15.3.27).—The first discharge of exhaust gases is passed through an adsorptive filter (such as active carbon or silica gel, which is active only at low temperatures) for the removal of hydrocarbons. The remaining carbon monoxide is oxidised to carbon dioxide by its passage through a layer of a catalyst such as "hopcalite"—a prepared mixture of oxides of copper, manganese, cobalt, and silver—which oxidises only carbon monoxide at low temperatures, but hydrocarbons as well at high temperatures. As the heat of the gases warms up the adsorptive filter the adsorbed hydrocarbons are driven off from it and pass along with the exhaust gases. This filter is automatically cut out and, when cool, is again in an active state to deal with the first discharge of gases. Meanwhile the "hopcalite" has been heated (by the heat of reaction and of the gases) to the temperature at which it is capable of oxidising both the carbon monoxide and the hydrocarbons. J. A. SUGDEN.

Purifying the exhaust gases from internal-combustion engines. J. BILSKY (J. G. BILLINGS) (U.S.P. 1,716,481, 11.6.29. Appl., 13.7.27).—Heavy-metal compounds are removed by passing the gases through masses containing sodium chromate.

J. A. SUGDEN.

Desulphurisation of gases. J. Y. JOHNSON. From I. G. FARBERIND, A.-G. (B.P. 312,769, 26.4.28).—Industrial gases which are laden with flue dust, combustion residues, or unconsumed particles of coal etc. are freed from sulphur compounds by adding oxygen, or gases containing oxygen, and ammonia, if sufficient is not already present, and heating the gases at 100–200° until the sulphur compounds are oxidised to sulphur,

which is then removed with the dust by any convenient mechanical or electrical device. Gases containing dust in excess of that necessary to catalyse the oxidation and take up the sulphur may be freed from the excess before undergoing treatment.

A. B. MANNING.

Separation of tar from gases. KOPPERS Co., Assees. of J. BECKER (B.P. 289,768, 20.2.28. U.S., 30.4.27).—The cooled gas is "wire-drawn" through a number of perforated baffles in order to convert the very finely-divided tar into a suspension of relatively large particles. These are condensed by passage of the gas through a conduit provided with vanes shaped to give it a whirling motion. The gas is finally reheated by means of a steam coil before its passage to the saturator for recovery of the ammonia. In a preferred form of apparatus, the perforated baffles are arranged in an annular chamber in the upper part of a cylindrical tower, and the steam heating coils in a corresponding chamber below, the centre of the tower being occupied by the conduit on the surface of which the tar globules are deposited by centrifugal force.

A. B. MANNING.

Distillation of tar. BARRETT Co., Assees. of F. OSBORNE (B.P. 285,907, 18.2.28. U.S., 26.2.27).—Tar is sprayed into the uptake pipes of a section of the coke oven, and the gases are passed through a short collector main where the heavy oils are separated. The enriched gases are then mixed with the gases from the rest of the battery, and passed through the ordinary condensing system. In this way the accumulation of heavy oils in the tar is avoided. A suitable system of collector mains and condensers is described.

J. A. SUGDEN.

Distillation of tar and the like. BARRETT Co., Assees. of S. P. MILLER (B.P. 282,826, 30.12.27. U.S., 31.12.26).—Distillation is effected by bringing the tar directly into contact with hot coke-oven gases as described in B.P. 282,367 (B., 1929, 507). A limited number of ovens in the battery are provided with "uptake pipe stills," and with their own collecting, cooling, and condensing system, so that the gases and vapours from the distillation are kept separate from those from the main portion of the battery. The tar from the latter may be condensed in two separate fractions, and one or both of these then subjected to distillation as above. Mixed tars may be distilled in this manner in order to produce composite pitch residues and distillates of any desired properties.

A. B. MANNING.

Distillation of tar, oils, etc. W. B. DAVIDSON, A. C. MICHIE, and E. W. MUDDIMAN (B.P. 312,722, 14.3.28. Addn. to B.P. 255,919; B., 1926, 815).—Enriched wash oil is fed into a primary still in which the removal of any water and a portion of the light oil takes place. The hot oil leaving the still passes to an annexe and over bubble trays, where it is subjected to the scrubbing action of upward-passing steam jets. Light oils and vapours thereby removed pass through an aperture into the primary still at a position above the level of the oil therein and below the upper steam pipes. By such means any froth is broken up and prevented from contaminating the distillate leaving the still.

H. S. GARLICK.

Tar products [for road making]. J. J. V. ARMSTRONG. From G. PLAUSON (B.P. 312,372, 18.11.27).—Tar is hardened by dispersing 10–50% of hard pitch, hard bitumen, or hard natural resin therein, using a synthetic resin as a dispersing agent. Sulphur may also be incorporated with the tar if desired. The ingredients are preferably mixed in a colloid mill. The synthetic resins may be formed *in situ*, e.g., by the action of formaldehyde on the phenols in the tar, in the presence of a condensing agent, or by the addition of solvent naphtha and the production of coumarone resin by heating with or without a condensing agent. Lime, baryta, etc. or pulverised brown coal may be added to the product to neutralise or absorb any free acidity.

A. B. MANNING.

Manufacture of asphaltic products. L. KIRSCHBRAUN, Assr. to F. L. BELKNAP (U.S.P. 1,715,069, 28.5.29. Appl., 27.1.23).—Petroleum residuum is mixed with air and gradually heated as it is passed, by a long route, to a bulk receptacle. After agitation therein the liquid oxidised asphalt is discharged.

F. G. CLARKE.

Waterproofing composition. W. B. VAN ARSDEL, Assr. to BROWN Co. (U.S.P. 1,714,702, 28.5.29. Appl., 3.9.26).—Petroleum asphalt, a mineral wax, and finely-divided inert material are mixed. The product becomes hard, non-sticky, and wax-like, and melts to a thin fluid at 177°.

F. G. CLARKE.

Separation from solid residues of oils obtained in the destructive hydrogenation of coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,657, 20.2.28).—The hot sludge from the hydrogenator passes through a heat interchanger where the solvent is distilled off from the oil-laden solvent for recirculation through the apparatus. The sludge is then allowed to fall down a long extraction chamber (provided with stirrers) in which it meets an upward-flowing stream of solvent and is discharged at the bottom through a worm-feed. Solvent adhering to the discharged solid is recovered by distillation. The stream of solvent when it reaches the top of the chamber passes into a wider settling chamber where fine solid particles are deposited, and then flows into the heat interchanger for the separation of solvent and extracted oil.

J. A. SUGDEN.

Manufacture of apparatus for use in distillation and cracking of oils. C. ARNOLD. From STANDARD OIL DEVELOPMENT Co. (B.P. 313,057, 31.12.27).—The interior of pressure stills, digestion chambers, soaking drums, and other apparatus of large volume used at high temperatures in the commercial refining of petroleum is protected from corrosion by fusing by means of an electric arc a ribbon or plate of a relatively non-corrodible metal, e.g., a chromium-iron or chromium-steel alloy such as that containing Fe 70–73%, Cr 16–18%, Si 1.25–1.75%, and C about 0.1%, and depositing the molten metal upon the surface to be protected at a temperature sufficient to cause bonding by fusion.

H. S. GARLICK.

Manufacture of hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,717, 10.3.28).—High continuous yields of hydrocarbons (etc.) containing

more than one carbon atom may be obtained from mixtures of oxides of carbon and hydrogenous gases if the catalyst (oxides of iron, cobalt, nickel, etc.) contains 0.4–0.6% of alkali, calculated as metals. Such catalysts, composed of single or mixed metals, may be prepared either by precipitating the appropriate solution with ammonia, drying the precipitate, and subsequently adding the necessary amount of a solution of the alkali salt, or by precipitating the solution with alkali hydroxide or carbonate, and washing the precipitate until the necessary amount of adsorbed alkali remains.

J. A. SUGDEN.

Obtaining gaseous and low-boiling olefines and diolefines from bituminous coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 313,756, 23.6.28. Addn. to B.P. 268,599; B., 1927, 100).—The raw materials are subjected to the action of an electrically heated zone in which granular, electrically conductive materials, which may themselves be carbonaceous or non-conductive materials rendered conductive by impregnation with electrolytes, are kept in a state of agitation by means of gases or vapours, *e.g.*, hydrogen or hydrocarbon vapours, blown through them at a sufficiently high rate, and which must consist of hydrocarbon vapours in case the granular material is non-bituminous. Suitable apparatus for the production of mixtures of olefines rich in butadiene is described.

H. S. GARLICK.

Catalytic conversion of mixtures of carbon monoxide and hydrogen into valuable organic compounds containing more than one carbon atom in the molecule. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 313,467, 9.2.28. Addn. to B.P. 266,405; B., 1927, 316).—The gaseous mixture contains at least 40% of an inert gas. As the products of the reaction are removed, fresh gases, free from inert gas, are introduced into the circulatory system. In this way the deposition of carbon on the catalyst is avoided. The injurious effect of the heat of reaction is reduced by bringing about a heat interchange between the catalyst and the incoming cold gases.

J. A. SUGDEN.

Production of lubricating oil from gases containing hydrocarbons. C. EPNER (B.P. 294,099, 8.6.28. Ger., 16.7.27).—Gaseous hydrocarbons of low mol. wt. are subjected to the prolonged action of an alternating current of high frequency and high tension, *e.g.*, by passage through one or more Siemens tubes at 10,000 cycles and 8000 volts. The resulting viscous product may be hydrogenated in order to obtain greater chemical constancy.

H. S. GARLICK.

Manufacture of white petrolatum. E. B. COBB and A. HOLMES, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,707,187, 26.3.29. Appl., 27.10.25).—A naphtha or other solution of crude petrolatum, after treatment with fuming sulphuric acid, is extracted with a solvent, *e.g.*, isopropyl alcohol, to remove the sulphonated compounds. The solvents are recovered by distillation and the petrolatum is passed through a decolorising agent, *e.g.*, clay.

F. G. CLARKE.

Petroleum distillates and treatment of same with respect to discoloration. C. H. JOHNSON. From STANDARD OIL Co. (B.P. 312, 774, 30.4.28).—Discoloration due

to auto-oxidation is removed from, or prevented from forming in, kerosene and gasoline by the addition of an aromatic or aliphatic dihydroxy-compound, aromatic amine, phenol, urea or urea derivative, or an alkaloid, in the proportion of not more than 1 pt. per 10,000 pts. of petroleum distillate. Suitable compounds are quinol, pyrogallol, resorcinol, pyrocatechol, thiourea, etc. Where the treating agent is a solid, it is preferably dissolved in an aliphatic or aromatic alcohol (*e.g.*, butyl or benzyl alcohol) before being added to the oil.

H. S. GARLICK.

Purification of hydrocarbon oil. L. M. JOHNSTON and J. L. FARRELL (U.S.P. 1,706,614, 26.3.29. Appl., 22.8.25).—The treating medium, *e.g.*, fuller's earth, bentonite, is made into a thin paste and charged through steam-jacketed nozzles by means of a mud press into the top of a tower up which the hot hydrocarbon vapours ascend. The mud threads are partly dried by the vapours and form a hard column through which the vapours continuously pass to a condenser. The spent material is removed continuously through a shaking grate and periodically extracted or distilled to recover entrained oil.

R. BRIGHTMAN.

Removal of wax from hydrocarbon oils. R. S. LANE, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,706,120, 19.3.29. Appl., 2.1.26).—Cylinder stock or lubricating oil distillate is mixed with 50–75% of a diluent containing 35–60% of naphtha, 25–35% of butyl alcohol, and 12–35% of acetone, this mixture cooled to –7° or lower, and the petrolatum removed by settling or centrifuging.

R. BRIGHTMAN.

Refining of mineral oil. H. BLUMENBERG, JUN. (U.S.P. 1,716,103, 4.6.29. Appl., 12.1.27).—The oil is treated with aluminium sulphite, the mixture is heated to 71°, and the volatile constituents are then removed by distillation.

F. G. CLARKE.

Handling heavy oil residues. O. E. BRANSKY and F. M. ROGERS, Assrs. to STANDARD OIL Co. (U.S.P. 1,698,471, 8.1.29. Appl., 18.1.23).—Crude oil residues from the non-cracking distillation of asphalt-bearing petroleum is diluted with light hydrocarbon oil, *e.g.*, naphtha, b.p. 88–165°, the undissolved asphaltenes are separated, and the naphtha solution is treated, *e.g.*, with sulphuric acid, *d* 1.84; filtered through fuller's earth etc., and chilled to remove wax etc. if desired, before distilling off the naphtha.

R. BRIGHTMAN.

Pitch-coking process. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,715,240, 28.5.29. Appl., 31.8.21).—Liquid pitch is gradually introduced at the top of one end of a hot retort of oval section until full. The coking is then continued until completed, the retort emptied, and the process repeated, the retort being heated continuously. By avoiding large changes in temperature, contamination of the carbon product with silica, from the joints and lining, is avoided.

F. G. CLARKE.

Revivifying fuller's earth [from treatment of hydrocarbon oils]. F. W. HALL, Assr. to TEXAS Co. (U.S.P. 1,709,261, 16.4.29. Appl., 20.4.21. Renewed 15.1.25).—Occluded asphalt is removed by washing with a mixture of petrol and alcohol.

W. G. CAREY.

Apparatus for drying, charring, and otherwise treating loose material. O. DOBBELSTEIN (U.S.P. 1,714,853, 28.5.29. Appl., 29.3.26. Ger., 4.4.25).—See B.P. 250,224; B., 1926, 904.

Benzene safety lamp for indicating fire-damp. H. FLEISSNER (U.S.P. 1,716,374, 11.6.29. Appl., 31.8.26. Ger., 12.9.25).—See B.P. 443,269; B., 1928, 44.

[Oil] filters (B.P. 313,179). Distilling liquids (B.P. 313,254).—See I. Oil-soluble sulphonates (U.S.P. 1,707,506).—See III. Bituminous emulsions (U.S.P. 1,707,809). Materials for road construction (B.P. 313,824).—See IX. Artificial shellac (B.P. 309,384). Coumarone-indene resins (U.S.P. 1,705,857).—See XIII. Synthetic rubber (B.P. 312,741).—See XIV. Materials for toilet baths (B.P. 285,356).—See XXIII.

III.—ORGANIC INTERMEDIATES.

Methyl- and ethyl-protocatechuic aldehydes. L. KLOTZ (Amer. J. Pharm., 1929, 101, 442–447).—The colour reactions given by methylprotocatechuic aldehyde (vanillin) and 4-hydroxy-3-ethoxybenzaldehyde are due to either the hydroxyl or aldehyde groups, and in the twelve tests examined marked similarity is observed. *p*-Hydroxybenzaldehyde also gives the vanillin reactions, indicating that a substituent in the *meta*-position has little or no effect on the coloration produced. The similarities in the above colour reactions for the protocatechuic aldehydes are interpreted as being due to the presence of the same number of chromophoric groupings, and the probable uniformity of the absorption spectra of methoxyl and ethoxyl groups.

II. BURTON.

[Preparation of] diphenylamine from chlorobenzene and aniline. P. P. KARPUKHIN (J. Chem. Ind. Moscow, 1928, 5, 1106–1107).—When aniline (46.5 g.), chlorobenzene (66 g.), and soda-lime (40 g.) are heated for 7 hrs. at 300°, the yield of diphenylamine is 12.3% of the theoretical. When the quantities are 37 g., 44 g., and 40 g., respectively, the yields are: 320°, 23–25%; 360°, 32%; 34 hrs. at 360–370°, 41% of the theoretical. On substitution of glass tubes for the steel autoclave, the results were: 300°, 60%; 320°, 72%.

CHEMICAL ABSTRACTS.

Anthracene. I. Methods of analysis. H. A. J. PIETERS and T. H. KOENEN (Chem. Weekblad, 1929, 26, 222–224).—The modification of Luck's anthraquinone method of analysis of crude anthracene by Sielisch (Z. angew. Chem., 1928, 41, 231) does not give accurate results, since phenanthrene is partly converted into phenanthraquinone under the same conditions. The treatment with sulphuric acid, the drying, and the sublimation each gives rise to loss, which by trial with pure anthracene was found to amount to 10.7% in all. The method is therefore modified by first treating the crude anthracene with 4*N* sulphuric acid to remove bases, and extracting the residue with toluene (100 c.c. to 50 g. sample) to remove phenanthrene; 1 g. of the residue is then oxidised and weighed as anthraquinone, which is dried at 90° and neither treated with sulphuric acid nor resublimed.

S. I. LEVY.

Absolute alcohol. SZILAGYI.—See XVIII.

See also A., July, 757, Adsorption from aqueous solution by silica (BARTELL and FU). 775, Electro-synthesis of hydrocarbons (BERMEJO and BLAS). 803, Perylene and its derivatives (ZINKE and SCHNIDERSCHITSCH). 805, Introduction of triphenylmethyl group into phenols (HARDY). 823, Thionaphthens (FRIES and HEMMECKE). 826, Arylamides of aromatic carboxylic and sulphonic acids (HELLER).

PATENTS.

Process and catalyst for synthesis of methyl alcohol. II. II. STORCH, Assr. to ROESSLER & HASS-LACHER CHEM. Co. (U.S.P. 1,707,331, 2.4.29. Appl., 2.8.27).—Catalysts consisting of 10–50% of reduced copper and a fluoride of the cerium group are used for the synthesis of methyl alcohol from hydrogen and carbon monoxide, at, e.g., 300–400°, and 200–300 atm. Thus the precipitate obtained on adding a solution of potassium fluoride and hydroxide to a solution of copper nitrate and cerium, thorium, or neodymium nitrate is dried, granulated, and reduced at 150–200° with a mixture of hydrogen and methyl alcohol in nitrogen.

R. BRIGHTMAN.

Manufacture of esters of vinyl alcohol. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 285,095, 11.2.28. Ger., 12.2.27).—Acetylene and organic (acetic) acid vapour are preheated and passed rapidly (4–5 m.³ of acetylene and 5 kg. of acetic acid per hour) over a catalyst (active carbon or silica gel impregnated with zinc acetate or cadmium acetate), the rate being such that about 0.5–0.7 kg. of vinyl acetate per hour is produced.

C. HOLLINS.

Manufacture of higher alcohols. W. G. DAVIS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,623, 21.3.28).—Crude liquor from the hydrogenation of carbon monoxide is distilled until the residue contains 75% of higher alcohols. The residue is extracted with brine and distilled to give isobutyl alcohol etc. A continuous process is described.

C. HOLLINS.

Distillation of glycerol. E. I. DU PONT DE NEMOURS & Co. (B.P. 300,254, 9.6.28. U.S., 11.11.27).—The spray-still for concentration of glycerol slop is lined with copper or other non-ferrous non-catalysing material at all parts above 225°.

C. HOLLINS.

Manufacture of organic oxygen compounds [ketones, acids]. J. Y. JOHNSON. From I. G. FARBERIND A.-G. (B.P. 310,438, 23.1. and 24.8.28).—Carbon monoxide under pressure reacts with non-aromatic hydrocarbons or their derivatives in presence of aluminium chloride at 30–60° or boron fluoride or chloride at 180°, in absence of air. *n*-Petane gives ethyl isopropyl ketone, b.p. 114–116°, mixed higher ketones, b.p. 116–250°, having a menthol odour, and hexoic and other acids. *cyclo*Hexane yields an oil, b.p. 100–150°, and products are similarly obtained from *n*-butane, light petroleum, heavy benzene, petroleum of b.p. 150–300°, butyl chloride, ethyl chloride, and methyl or ethyl ether, the last two compounds giving acetic and propionic acid, respectively.

C. HOLLINS.

Manufacture of aliphatic acid [acetic] anhydrides. BRIT. CELANESE, LTD., S. J. GREEN, and R. R. WIDDOWSON (B.P. 310,863, 28.1.28).—The vapours from the

thermal anhydriation of acetic acid in presence or absence of catalysts are met by a liquid stream, preferably at 0–20°, of solvent, especially a mixture of ether and light petroleum. The solution of acetic anhydride is separated from the water layer and distilled.

C. HOLLINS.

Manufacture of maleic acid and maleic anhydride. A. BOEHRINGER (B.P. 285,426, 3.2.28. Ger., 16.2.27).—Crude furfuraldehyde vapour is passed with diluted oxygen over an oxidising catalyst (zinc vanadate on a granular aluminium carrier) at 300–350°.

C. HOLLINS.

Oxidation of aliphatic compounds of high mol. wt. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 310,069, 17.12.27).—Paraffin wax is oxidised with air etc. at 80–180° in presence of an organic salt of an alkaline-earth metal, magnesium, or aluminium (e.g., barium stearate, calcium cinnamate) in conjunction with an inert filler (e.g., aluminium packing rings) to give almost colourless acid products. C. HOLLINS.

Manufacture of reduction products of vat dyes and other reducible organic compounds. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 310,437, 21.1.28. Addn. to B.P. 299,899; B., 1929, 12).—Vat dyes and other reducible quinones are reduced to leuco-compounds, suitable for direct conversion into sulphuric esters, by treatment with metals (copper, zinc) in dry pyridine or other organic base containing 20% of its weight of sulphur dioxide. If iron, aluminium, etc. be used the presence of a little water is advantageous.

C. HOLLINS.

Manufacture of oil-soluble sulphonates. H. E. BUC, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,707,506, 2.4.29. Appl., 26.9.27).—Crude sodium sulphonates obtained from petroleum distillates are heated at 150–220°, e.g., at 200° for 2 hrs., in the presence of about 3% of sodium hydroxide, or other alkali, and the product is dissolved in an organic solvent (88% isopropyl alcohol), settled, and the solvent evaporated.

R. BRIGHTMAN.

Dye intermediates [dry leuco-compounds]. R. S. BARNES, J. E. G. HARRIS, B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 309,970, 17.1.28).—Pastes of leuco-compounds of vat dyes are dried by mixing with an organic liquid (chlorobenzene, dimethylaniline, nitrobenzene) and distilling off water and the liquid at suitable pressure (e.g., in a vacuum). The product is suitable for conversion into sulphuric esters. C. HOLLINS.

Manufacture of [nuclear] alkylated phenols. SCHERING-KAHLBAUM A.-G. (B.P. 294,238, 15.6.28. Ger., 21.7.27).—Phenol ethers are heated under pressure, especially in presence of a surface catalyst. Phenetole at 280–320°/50–60 atm. gives *p*-hydroxyethylbenzene; *m*-tolyl isopropyl ether, b.p. 193–194°, yields at 320–350°/60 atm. a product containing thymol.

C. HOLLINS.

Condensation product from *p*-halogenated phenols and aldehydes. M. WEILER, B. WENK, and H. STÖTTER, ASSRS. to I. G. FARBERIND. A.-G. (U.S.P. 1,707,181, 26.3.29. Appl., 13.3.28. Ger., 25.3.27).—*p*-Halogenated phenols containing at least one free *ortho*-position to the hydroxyl group are condensed

with aldehydes in presence of, e.g., sulphuric acid, hydrochloric acid and glacial acetic acid, zinc chloride at 15–70° to give products, $X \cdot CH(R' \cdot OH) \cdot R'' \cdot OH$, where R' and R'' are residues of *p*-halogenophenols, and X = hydrogen, alkyl, or aryl, the hydroxyl groups being *ortho* to the CHX group, which are effective for proofing material against moths etc. The following are described: 3 : 3' : 5 : 5'-tetrachloro-2 : 2'-dihydroxy-diphenylmethane, 5 : 5' - dichloro - 2 : 2' - dihydroxy-diphenylmethane, 4 : 5' : 5''-trichloro-2 : 2'-dihydroxy-triphenylmethane, and 5 : 5'-dichloro-2 : 2'-dihydroxytriphenylmethane-2''-sulphonic acid. R. BRIGHTMAN.

Manufacture of condensation products from [phenols and] unsaturated higher fatty acids or their glyceryl esters. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 310,562, 28.1.28).—Phenols (phenol, resorcinol) are condensed at 100° in presence of acid (50% sulphuric acid) with unsaturated fatty acids or fats, e.g., tung oil, linseed oil. Some of the products are sulphonated with 100% sulphuric acid to give auxiliary materials for the textile industry.

C. HOLLINS.

Manufacture of 3-methyl-6-isopropenylphenol [4-isopropenyl-*m*-cresol]. SCHERING-KAHLBAUM A.-G. (B.P. 293,863, 3.7.28. Ger., 14.7.27. Addn. to B.P. 273,686; B., 1929, 236).—The condensation product of *m*-cresol with acetone (B.P. 308,741; B., 1929, 467) is heated at 250° in liquid or vapour form with or without an inert, porous contact mass.

C. HOLLINS.

Treatment [reduction] of aromatic nitroso-compounds [and indophenols etc.]. R. VIDAL (B.P. 283,467, 9.1.28. Fr., 8.1.27).—For the isolation of aminophenols, leucoindophenols, etc. formed by reduction of nitroso-compounds or mixtures of nitroso-compounds and phenols or amines by means of sodium sulphide, ammonium chloride or sulphate is added.

C. HOLLINS.

Manufacture of aromatic acid chlorides. B. W. HENDERSON, J. G. PERCIVAL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,909 and 310,910, 2.2.28).—Benzoyl chloride is obtained by the action (A) of chlorine or chlorine and hydrochloric acid on benzyl alcohol or hydrolysed crude benzyl chloride, (B) of benzoic acid on benzylidene chloride and subsequent treatment with chlorine. The process is also applicable to the preparation of *o*- and *p*-chlorobenzoyl chlorides from corresponding starting materials.

C. HOLLINS.

Alkoxy-esters of polybasic organic acids. R. H. VAN SCHAAK, JUN., and R. CALVERT, ASSRS. to VAN SCHAAK BROS. CHEM. WORKS, INC. (U.S.P. 1,706,639, 26.3.29. Appl., 31.10.27).—Esters of polycarboxylic acids, e.g., phthalic, citric, tartaric, succinic, phenylmalonic, or trimesic acid, in which at least one carboxylic hydrogen is replaced by an alkoxyalkyl radical containing at least four carbon atoms, are used as solvents and plasticisers for pyroxylin. Examples are β -ethoxyethyl phthalate, b.p. 200–202°/4 mm. (from phthalic anhydride and glycol monoethyl ether), and β -ethoxyethyl butyl phthalate, b.p. 182–185°/5 mm., d_{20}^{20} 1.08.

R. BRIGHTMAN.

Manufacture of borneol. G. AUSTERWEIL (B.P. 311,085, 2.5.28).—Nopinene in an inert diluent (toluene,

xylene, pinene) is heated, *e.g.*, at 145°, with phthalic anhydride, and absolute alcohol is added drop by drop, so as to generate ethyl hydrogen phthalate, which reacts with nopinene (but not with pinene) to form bornyl ethyl phthalate, which gives a 70% yield of borneol on hydrolysis. The reaction gives better results under pressure.

C. HOLLINS.

Salts of β -naphtholsulphonic acids and their manufacture and separation. M. L. CROSSLEY and G. S. SIMPSON, Assrs. to CALCO CHEM. CO. (U.S.P. 1,701,259, 5.2.29. Appl., 14.10.22).—Solutions containing β -naphtholdisulphonic acids are neutralised with an alkaline-earth compound and the neutral salts are converted into basic salts by treatment with an alkaline hydroxide. The sparingly soluble salt, *e.g.*, R-salt, is crystallised out and the mother-liquor treated with an ammonium salt which gives an insoluble salt with an alkaline-earth metal. The insoluble salt is filtered off, the solution concentrated, and the ammonium salt of, *e.g.*, the β -naphtholdisulphonic acid crystallised, the mother-liquors being returned to a subsequent batch. The following salts are described, the figures denoting solubility in 100 c.c. of water at 25°: R-salts (β -naphthol-3:6-disulphonates):—*ammonium hydrogen*, 73.4; *ammonium*, 64.7; *sodium potassium*, yellow, 23; *cupric*, greyish-white, 41.4; *nickel*, light green, 15.4; *ferrous*, bluish-grey, 17.5; *cobaltous*, pink, 17.4; *tricalcium*, 2.5; *potassium disodium*, yellow, 24.6; *sodium dipotassium*, 31; *dipotassium dicalcium*, 0.77; *disodium dicalcium*, 2.8; *diammonium dicalcium*, 3; *calcium tetrasodium*, 7.4; *trisodium*, yellow, 21; *tripotassium*, golden-yellow, 34.6; *triammonium*, 69; G-salts (β -naphthol-6:8-disulphonates):—*ammonium hydrogen*, 30.1; *ammonium*, 30.2; *triammonium*, 69; *tricalcium*, 22.6; *trisodium*, 59; *tripotassium*, 23.5; *ammonium calcium*, 35.4; *potassium calcium*, 27.4.

R. BRIGHTMAN.

Purification of anthraquinone. H. F. LEWIS, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,701,186, 5.2.29. Appl., 27.1.22).—The crude anthraquinone is heated to 275–350° by superheated steam and the resulting vapours are cooled first at 175–275° to separate most of the anthraquinone, and finally at below 100°.

R. BRIGHTMAN.

Manufacture of condensation products of the anthraquinone series [1:1'-dihydroxy-2:2'-dianthraquinonyl]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 310,353, 24.1.28).— α -Hydroxy-anthraquinone is heated at 100–130° with aluminium chloride in dry pyridine to give 1:1'-dihydroxy-2:2'-dianthraquinonyl.

C. HOLLINS.

Manufacture of condensation products of the anthraquinone series [dibenzpyrenequinones]. I. G. FARBENIND. A.-G. (B.P. 294,550, 25.7.28. Ger., 25.7.27. Addn. to B.P. 205,502; B., 1924, 627).—Aryl α -naphthyl ketones (*e.g.*, 1-benzoylnaphthalene) are converted into 3:4:8:9-dibenzpyrene-5:10-quinones by heating at 120–130° with an aroyl chloride (*e.g.*, benzoyl chloride) and aluminium chloride or sodium aluminium chloride in a current of air or oxygen.

C. HOLLINS.

Production of mercaptans of the furfuryl series. H. STAUDINGER and T. REICHSTEIN, Assrs. to INTERNAT.

NÄHRUNG- & GENUSSMITTEL A.-G. (U.S.P. 1,715,795, 4.6.29. Appl., 22.7.27. Ger., 16.12.26).—See B.P. 236,152; B., 1928, 327.

Benzantrones substituted in the Bz nucleus and their manufacture. G. KRÄNZLEIN, H. VOLLMANN, H. GREUNE, and A. WOLFRAM, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,705,868, 19.3.29. Appl., 31.3.27. Ger., 3.4.26).—See B.P. 268,830; B., 1928, 666.

Catalytic apparatus (B.P. 306,519).—See I. **Organic compounds from gases** (B.P. 313,467). **Olefines and diolefines** (B.P. 313,756).—See II. **Polymerisation products of diolefines** (B.P. 312,949 and 313,188).—See XIV. **Pentaerythritol tetranitrate** (U.S.P. 1,705,669).—See XXII.

IV.—DYESTUFFS.

See A., July, 762, **Peptisation of dyes by neutral salts** (OSTWALD). 804, **Azo dyes of dinaphthyl series** (KUHN and GOLDFINGER). 822, **Fluorescein derivatives** (ORNDORFF and WILLARD). 823, **Colouring matter from alkekengi** (*Physalis alkekengi* and *P. franchetti*) (KUHN and WIEGAND).

PATENTS.

Manufacture of derivatives of the anthraquinone series containing nitrogen [dyes for acetate silk]. I. G. FARBENIND. A.-G. (B.P. 284,242, 24.1.28. Ger., 25.1.27).—Anthraquinones containing at least two *para*-hydroxyl groups (quinizarin, purpurin, 1:4:8-trihydroxy- and 1:2:4:5:8-pentahydroxy-anthraquinones) are condensed with ethylenediamine to give nitrogenous products, which are in many cases dyes for acetate silk.

C. HOLLINS.

Manufacture of [nitro]-dyes [for wool]. I. G. FARBENIND. A.-G. (B.P. 285,504, 18.2.28. Ger., 18.2.27).—Diaminodiphenyls are condensed with halogenated, nitrated benzenesulphonic acids (2 mols.) to give wool dyes. The condensation of 1-chloro-2:6-dinitrobenzene-4-sulphonic acid with benzidine (orange), diphenylene (golden-yellow), dianisidine (reddish-brown), 2-nitrobenzidine (orange), of 1-chloro-2:4-dinitrobenzene-6-sulphonic acid with dianisidine (reddish-brown), and of 1-chloro-2-nitrobenzene-4-sulphonic acid with benzidine (yellowish-brown), dianisidine (reddish-brown), and 2-nitrobenzidine (brownish-yellow) is described.

C. HOLLINS.

Manufacture of cotton [azo] dyes [and pigments]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 310,354, 24.1.28).—Dehydrothiotoluidinesulphonic acid is diazotised and coupled in substance or on a substrate (blanc fixe) with an acetoacetic sulphaorylamide, or with an acetoacetic arylamide with subsequent sulphonation. Pigments may also be prepared from solutions of the dyes in the usual manner.

C. HOLLINS.

Manufacture of water-insoluble azo dyes. I. G. FARBENIND. A.-G. (B.P. 284,247, 25.1.28. Ger., 25.1.27).—2:3-Hydroxynaphthoic arylamides are coupled in substance or on the fibre with diazotised *m*-4-xylydines containing in position 6 a group 'NRR' in which R is alkyl or hydrogen, and R' is a benzoyl or substituted benzoyl group. 6-Benzamido-4-*m*-xylylidine, m.p. 175°.

prepared by reduction of the nitration product, m.p. 184—185°, of benz-4-m-xylylide, m.p. 190—192°, gives with 2:3-hydroxynaphthoic α -naphthylanide a bluish-red. 6-Benzmethylamido-4-m-xylylidine, m.p. 177°, and 6-op-dichlorobenzamido-4-m-xylylidine, m.p. 194—195°, give similar shades with 2:3-hydroxynaphthoic 5-chloro-o-toluidide. C. HOLLINS.

Manufacture of complex chromium compounds of azo dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,343, 24.10.27).—Dyes obtained by coupling *m*-phenylenediamine-5-sulphonic acid with diazotised *o*-aminophenols containing two or more negative substituents (picramic acid, 4-nitro-*o*-aminophenol-6-sulphonic acid) are prechromed by the usual methods. [Stat. ref.] C. HOLLINS.

Vat dyes of pyridazine-anthraquinone series. A. JOB, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,786, 6.11.28. Appl., 17.8.27. Ger., 25.8.26).—See B.P. 300,683; B., 1929, 124.

Reduction products of vat dyes (B.P. 310,437).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Flax retting. I. Aerobic spore-bearing bacteria isolated from retted flax fibre. II. Pure culture flax retting. III. Acidity in retting flax. A. TREVETHICK, B. B. ROBINSON, and R. M. SNYDER (Mich. Agric. Exp. Sta. Tech. Bull., 1928, 95, 1—26, 24—37, 38—49).—The principal organisms concerned are clostridia similar to *C. butyricum*. Certain members of the *mesentericus-megatherium* group ret flax. Organic substances, colouring matter, and by-products produced in retting retard the process. The optimum temperature is 30°; neutrality is preferable. The control of acidity is discussed. CHEMICAL ABSTRACTS.

Hydration, paper-formation, and strength. G. PORRVIK (Papier-Fabr., 1929, 27, 372—383).—The degree of greasiness (which is considered synonymous with "degree of hydration") is determined by means of the water content of pulp. The water content is divided into three kinds: external capillary water, internal capillary water, and fibre-wall water, and their importance during grinding is discussed. The combining together of fibres to form paper is really not a mechanical felting process, but a colloid-chemical sizing phenomenon. The importance of grinding lies in increasing the degree of swelling of the fibre wall by mechanical means, whereby the latter is rendered partly more adhesive and partly more plastic. The fibre wall consists of several layers. By swelling the outer one the adhesive capacity of the fibres, and therefore the strength of the paper, is increased, whereas if the sub-layer swells, the fibre is split up into fibrils, and a weaker paper results. Grinding is not a uniformly continuous process, but takes place in three stages with which distinctly different processes are associated. B. P. RIDGE.

Determination of chlorine-consumption number (Sieber number) of sulphite-celluloses. W. HUMM (Papier-Fabr., 1929, 27, 387—389).—The Sieber number is the ratio of chlorine consumed to that originally

present (expressed as a percentage) when 5 g. of dry cellulose are treated with a bleaching powder solution of standard chlorine and alkali concentrations under controlled conditions (cf. B., 1921, 382 A; 1922, 409 A). Within narrow limits (e.g., 4.8—5.3 g.), the weight of cellulose taken does not materially affect the Sieber number, but beyond these limits too small a weight gives too high, and too large a weight too low, values. The suitability of the measurement for classifying sulphite-celluloses is discussed. B. P. RIDGE.

See also A., July, 796, **Sulphite-cellulose process** (MARUSAWA and others). 856, **Pine-wood lignin** (HÄGG-LUND and URBAN).

PATENTS.

Production of fibre. A. TINGLE (U.S.P. 1,714,831, 28.5.29. Appl., 25.1.24).—After digesting with aqueous lime and sulphur, fibrous vegetable material is treated with a lime-eliminating agent, e.g., aqueous mineral acid or sulphite-cellulose waste liquor, and washed to remove colouring matter. F. G. CLARKE.

Softening of vegetable fibres. S. D. WELLS, Assr. to PAPER MILL LABS., INC. (U.S.P. 1,714,459, 21.5.29. Appl., 25.9.26. Cf. B.P. 285,277; B., 1928, 330).—Liquors containing sodium carbonate and sulphur compounds are used, so that the waste liquors from pulping processes employing caustic soda are available without first causticising. F. G. CLARKE.

Treatment of [oil-soaked] hemp waste. C. C. BEETH (U.S.P. 1,710,914, 30.4.29. Appl., 23.1.28).—The material is soaked in sodium hydroxide solution and then thoroughly cooked by the introduction of steam. After removal of the liquid, from which the oil is recovered, the product is pressed into sheets. F. R. ENNOS.

Treatment of outer covers for aircraft. GOOD-YEAR-ZEPPELIN CORP., Assees. of K. HURTLE (B.P. 296,342, 22.5.28. U.S., 29.8.27).—The interstices between the fibres are closed by application of a moisture-repelling substance, e.g., paraffin, aluminium oxide, to the inside surface of the cover, which is then secured to the framework of the airship and its outer surface coated with one or more layers of protective dope. In this way, the fabric is tautened and the necessity of a mechanical smoothing process is obviated. F. R. ENNOS.

Production of chemical wood pulp. D. B. BRADNER, Assr. to CHAMPION COATED PAPER CO. (U.S.P. 1,711,584, 7.5.29. Appl., 24.1.27).—Sulphite-pulp obtained by the acid digestion of raw cellulosic material with sodium sulphite is digested in an alkaline liquor containing sodium sulphide, thus giving a pulp rich in α -cellulose. The waste alkaline liquor obtained together with some fresh alkaline liquor is used for the digestion of a further quantity of cellulose to yield sulphate-pulp and waste sulphate liquor; the latter, after concentration, is smelted to recover the sodium compounds and causticised to produce an alkaline cooking liquor for the treatment of more sulphite-pulp. F. R. ENNOS.

Manufacture of cellulose esters. VER. F. CHEM. IND. A.-G. (B.P. 303,485, 8.2.28. Ger., 5.1.28).—To

ensure thorough mixing during esterification in a rotatable reaction drum, it is provided with bars or beams traversing the interior and fixed at both ends to the inner wall of acid-resisting material, so as to exert a cutting action on the heart of the reacting mixture.

F. R. ENNOS.

Manufacture of threads, filaments, bands, or films of a polished and transparent nature, composed of plastic substances. Z. LANDUCCI, Assr. to DU PONT-PATHÉ FILM MANUFG. CORP. (U.S.P. 1,713,289, 14.5.29. Appl., 18.4.27. Fr., 29.4.26).—The solution of the cellulose derivative in an organic liquid is given the desired shape and coagulated in an aqueous bath, whereby a more or less opaque product is obtained, which is then subjected, before any other treatment, to a hot air-drying at 40–80° for sufficient time to render it transparent and polished.

F. R. ENNOS.

Manufacture of hollow artificial textile threads from viscose. ALSA SOC. ANON. (B.P. 289,796, 30.4.28. Ger., 30.4.27).—Viscose containing about 4% of sodium carbonate is coagulated in an acid bath, which contains a high proportion (10–35%) of zinc sulphate.

F. R. ENNOS.

Manufacture of hollow rayon [artificial silk] fibres. W. O. SNELLING (U.S.P. 1,713,679, 21.5.29. Appl., 21.5.27).—A preliminary fibre which is made from sugar, sulphur, glue, rosin, or casein is covered with a thin film of cellulose acetate, xanthate, or cuprammonium cellulose solution carried on a suitable coating wheel. After coagulation of the coating, the preliminary fibre is dissolved out.

F. R. ENNOS.

Manufacture of films from aqueous cellulose solutions, particularly viscose. FELDMÜHLE PAPIER-F. ZELLSTOFFWERKE A.-G. (B.P. 290,234, 10.5.28. Ger., 10.5.27).—After coagulation, the films are passed through a number of treatment baths, viz., for hardening, washing, bleaching, and dyeing, on cylinders which are so arranged that the same surface of the film is in contact during the whole time of each successive treatment with the corresponding cylinder; each of these has a polished surface and is provided with an adjustable drive in order to allow for shrinkage of the film during treatment.

F. R. ENNOS.

Spinning bath for viscose silk. A. L. MOND. From H. KINDERMANN & Co. (B.P. 311,112, 27.6.28).—An aqueous solution containing at least 80% of free acid and consisting of 35–44% of sulphuric acid and upwards of 36% of an organic acid (e.g., acetic, lactic, or formic acid) is used.

F. R. ENNOS.

Paper making. L. M. BOOTH (U.S.P. 1,713,642, 21.5.29. Appl., 8.2.28).—After adding the size and size precipitant, a metal hydroxide is precipitated in the pulp, at or before delivery to the paper-making machine.

F. G. CLARKE.

Production of cellulose and paper from straw, esparto, reed, and similar raw materials. E. L. RINMAN (U.S.P. 1,716,006, 4.6.29. Appl., 28.3.27. Swed., 7.4.26).—See B.P. 269,154; B., 1927, 873.

Cellulose hydrolysis products (B.P. 313,258).—See XVII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Measurement of colour at lustrous surfaces.

A. KLUGHARDT (Seide, 1928, 33, 333–340; Chem. Zentr., 1929, i, 148).

See also A., July, 777, **Influence of light on bleaching of neocyanine** (BHATTACHARYA and DHAR).

PATENTS.

Manufacture of solutions for dyeing purposes.

A. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 307,777, 13.12.27).—Clear and stable solutions of 2:3-hydroxynaphthoic arylides in an acid, neutral, or alkaline medium can be obtained by mixing them with sulphonated fatty acids derived from wool fat. Such solutions are stable in the presence of calcareous water, and by their use difficultly soluble arylides such as the β -naphthalide can be easily brought into solution.

L. G. LAWRIE.

Wetting-out agents or emulsifiers for use in textile and other industries. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, and E. CHAPMAN (B.P. 311,385, 30.3.28. Addn. to B.P. 274,611; B., 1927, 841).—The process described in the chief patent is simplified, and an improved yield of a lighter coloured product obtained, by adding the alcohol to the sulphonation mixture (which may contain chlorosulphonic acid) and effecting the sulphonation at below 100° (preferably at 60–65°).

A. J. HALL.

Dyeing of cellulose derivatives. A. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 307,813, 13.12.27).—Alkoxyanthraquinones without further substituents in the nucleus or in the alkyl group are suitable dyes for cellulose ethers and esters, and are applied as aqueous dispersions. Examples are: 1:4-dimethoxyanthraquinone (golden-yellow) and 1:4:5-trimethoxyanthraquinone (greenish-yellow).

A. J. HALL.

Production of water-resisting colouring on pellicles of cellulose derivatives. O. Y. IMRAY. From I. G. FARBENIND A.-G. (B.P. 284,999, 7.2.28).—A solution of cellulose ester or ether containing a colouring matter is applied locally to the pellicle of cellulose derivative, which may consist of one of the regenerated celluloses or esters or ethers of cellulose.

F. R. ENNOS.

Washing, dyeing, carbonising, and otherwise treating textiles. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 307,944, 10.9.27).—The addition of cyclohexanone or its homologues to aqueous baths for treating textiles is claimed to increase the efficiency of the process. [Stat. ref.]

L. G. LAWRIE.

Preparing [colouring] foliage. D. S. ANDERSON (U.S.P. 1,714,838, 28.5.29. Appl., 20.1.26. Renewed 24.10.28).—The foliage is impregnated with a solution containing calcium chloride, acetic acid, glycerin, and alcohol, and immersed first in a solution containing the above substances together with an aniline dye, gum arabic, camphor, and water, and then in an aqueous solution of gum arabic, an aniline dye, and the ingredients of the first-named solution.

A. J. HALL.

Improvement of animal or vegetable materials. SOC. CHEM. IND. IN BASLE (B.P. 294,890, 30.7.28. Ger., 28.7.27).—The mono- and di-acylated diamines described

in B.P. 219,304 (B., 1925, 899), and the corresponding quaternary ammonium compounds obtained by treating such products with alkylating or aralkylating agents, are useful wetting-out agents, particularly in acid liquors such as are used in the carbonisation and fulling of wool, dyeing with acid dyes, and the bleaching of straw fibre. If chrome-tanned skins are impregnated with an aqueous solution of such wetting-out agents before drying, they may afterwards be fulled with warm water so as to regain their original properties before drying. Oleyl-diethylenediamine hydrochloride is a satisfactory wetting-out agent; it does not form a precipitate with aluminium chloride under the conditions used in carbonising wool materials. A. J. HALL.

Finishing of fabrics and fibrous materials. H. T. BÖHME A.-G. (B.P. 293,746, 19.6.28. Ger., 11.7.27).—Highly lustrous finishes are obtained by calendering cotton or other fabric impregnated with a filling or weighting agent, such as an inorganic salt, and a lustre-imparting agent, such as paraffin, waxes, and stearic acid, it being possible to apply both these types of agent by means of a stable emulsion containing them, provided it contains a highly sulphonated vegetable oil (the degree of sulphonation must be considerably higher than that which characterises Turkey-red oil) or an aromatic sulphonic acid. A satisfactory homogeneous emulsion consists of 50 kg. of Epsom salts, 20 kg. of dextrin, 150 kg. of water, and 10 kg. of a wax emulsion prepared with dipropylnaphthalenesulphonic acid. A. J. HALL.

Dressing medium for textiles [silk or artificial silk]. CHEM. FABR. JACOBUS GES.M.B.H., and I. BUDOWSKI (B.P. 309,100, 30.12.27).—A dressing suitable for restoring the original lustre and new appearance of used textile materials (*e.g.*, of artificial silk) consists of an emulsion of a fatty acid, a weak organic or inorganic acid, and an emulsifying agent such as casein, an organic sulphonic acid, or saponins. The textile materials are immersed in the dressing diluted with water and then dried. Thus an emulsion of boric, tartaric, and oleic acids with isopropylnaphthylsulphonic acid is stabilised by the addition of paraffin oil. A. J. HALL.

Delustring of artificial silk fibres. O. F. MULLER, ASSR. to NYANZA COLOR & CHEM. CO., INC. (U.S.P. 1,705,490, 19.3.29. Appl., 14.7.28).—The fibres are treated with an emulsion of olive oil soap, glue, and paraffin in water at 30–35°, centrifuged, washed, and treated with an aqueous suspension of 5–10% of infusorial earth or other mineral abrasive, *e.g.*, chalk, fuller's earth, zinc oxide, at 30–35° for 30 min. In place of the paraffin emulsion, a solution of aluminium stearate in carbon tetrachloride may be used. R. BRIGHTMAN.

Mercerising textile materials containing both viscose and cellulose acetate silks. SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 309,280, 2.3.28).—Mercerisation is effected with caustic potash (*d* not less than 1.40, preferably 1.50), and the alkali is removed by washing with water at 10–20°. The period of impregnation, stretching, and washing occupies about 1 min.; saponification of the cellulose acetate silk does not occur and the soft handle and lustre of the viscose silk are unchanged. A. J. HALL.

Treatment of fabrics consisting of or containing acetyl silk. BLEACHERS' ASSOC., LTD., W. KERSHAW, F. L. BARRETT, and R. GAUNT (B.P. 311,306, 9.2.28. Addn. to B.P. 303,286; B., 1929, 204).—The prior patent is modified by impregnating the "blinded" fabric with a solvent for cellulose acetate before embossing or subjecting it to a localised heat-treatment to restore the original lustre in selected areas. Suitable solvents include ethyl acetate, ethyl lactate, lactic and acetic acids, but ethylene glycol monoethyl ether is preferred. The embossed portions of the fabric have an increased affinity for the usual dyes applied to cellulose acetate silk, so that two-tone colour effects may be obtained. A. J. HALL.

Treating the surfaces of hat bodies. V. BÖHM (U.S.P. 1,713,641, 21.5.29. Appl., 2.10.25. Austr., 20.11.24).—The surfaces are treated with a neutral solution of an aldehyde or ketone, *e.g.*, formaldehyde or benzophenone, and are then oxidised preferably with peroxides or permanganate. F. G. CLARKE.

Dyeing process [for animal fibres]. A. ESCAICH and J. P. WORMS (U.S.P. 1,716,218, 4.6.29. Appl., 20.11.23. Ger., 25.11.22).—See B.P. 230,128; B., 1925, 352.

Dyeing and printing. H. MÜLLER, ASSR. to J. R. GEIGY SOC. ANON. (U.S.P. 1,717,242, 11.6.27. Appl., 19.9.25. Ger., 23.7.23).—See G.P. 400,684; B., 1925, 8.

Treatment of vegetable fibrous materials to produce wool-like effects. G. HEBERLEIN, ASSR. to HEBERLEIN PATENT CORP. (U.S.P. 1,717,316, 11.6.29. Appl., 30.10.26. Ger., 19.8.26).—See B.P. 276,352; B., 1928, 260.

Treating cotton fibres to produce wool-like effects. A. BODMER, ASSR. to HEBERLEIN & CO. A.-G. (U.S.P. 1,717,322, 11.6.29. Appl., 8.9.26. Ger., 16.9.25).—See B.P. 258,598; B., 1927, 475.

Treatment of fibres of acetylcellulose to produce wool-like effects. R. CLAVEL (U.S.P. 1,716,423, 11.6.29. Appl., 27.9.23. Fr., 9.11.22).—See B.P. 206,818; B., 1924, 743.

Treatment of yarns and fabrics. Treatment of cellulose derivatives. G. H. ELLIS, ASSR. to CELANESE CORP. OF AMERICA (U.S.P. 1,716,720—1, 11.6.29. Appl., [A] 22.1.26, [B] 17.3.27. U.K., [A] 7.9.25, [B] 10.4.26).—See B.P. 262,506 and 273,819; B., 1927, 105, 650.

[Spraying] apparatus for treating fabrics with liquors. HUNT & MOSCROP, LTD., and E. W. and E. F. HUNT (B.P. 313,378, 19.9.28).

Treatment of textile fabrics with fluids. J. BRANDWOOD (B.P. 313,104, 7.2.28).

Manufacture of yarn from [mixed] artificial and natural silk fibres [suitable for cross-dyeing]. I. G. FARBENIND. A.-G., Assees. of M. F. THOMA (B.P. 286,275, 2.3.28. U.S., 2.3.27).

Baths or vats for use in dyeing, soaping, washing, and other finishing operations [for fabrics]. T. PARKINSON and J. A. O'LOUGHLIN (B.P. 313,243 31.3.28).

Apparatus relating to bleaching, washing, or dyeing of woven fabrics. FÄRBEREI WEIDMANN A.-G. (B.P. 302,598, 17.12.28. Ger., 17.12.27).

Coating, charging, or treating yarn or thread, cord, twine, wire, etc. with solid or liquid substances in being wound on to or into bobbins, skeins, etc. A. SEALEY (B.P. 312,590, 25.2. and 24.12.28).

Staining wood (U.S.P. 1,714,701).—See IX.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Preparation of rubidium compounds from carnallite. G. JANDER and H. FABER (Z. anorg. Chem., 1929, 179, 321—331).—Addition of a solution of sodium silicomolybdate to an acidified solution of recrystallised carnallite precipitates the sparingly soluble rubidium salt, which may be largely freed from potassium by washing with dilute hydrochloric acid. By heating the complex salt in a current of gaseous hydrogen chloride molybdenum oxychloride distils over and may be worked up again as sodium silicomolybdate, whilst the non-volatile material consists only of silica and rubidium chloride, together with some potassium chloride. Various methods may be employed to remove the potassium from the mixed chlorides, such as reprecipitating the silicomolybdate, dissolving it in sodium hydroxide solution, and reprecipitating by acidifying with hydrochloric acid. The sodium silicomolybdate solution is prepared by adding gradually 190 g. of molybdic acid in 550 c.c. of water to a solution of 33 g. of crystallised sodium silicate in 2200 c.c. of water; the molybdic acid which remains undissolved after heating the mixture for a short time is dissolved by adding about 300 c.c. of 2*N*-sodium hydroxide. After adding 200 c.c. of concentrated sulphuric acid and allowing the mixture to settle, the clear solution is decanted and evaporated on the water bath to 700 c.c.

H. F. GILLBE.

Preparation of beryllium chloride from beryl. R. W. WINTERS and L. F. YNTEMA (Amer. Electrochem. Soc., May, 1929. Advance copy. 3 pp.).—An intimate mixture of beryl and carbon is heated at 800° in a graphite chamber and chlorine, carbon tetrachloride vapour, or a mixture of the two is led into the chamber. From the resulting vapour crystals of beryllium chloride, free from aluminium, iron, and silicon, are deposited on passing through a condenser maintained at 375°. The yield of beryllium chloride may be as high as 80%.

H. J. T. ELLINGHAM.

Manufacture of zinc chloride and sulphate from Russian whiting. J. E. ADADUROV and K. U. BRODOVICH (J. Chem. Ind. Moscow, 1928, 5, 1018—1019).—Russian whiting (a mixture of zinc carbonate, oxide, and sulphide, sodium carbonate, thiosulphate, and sulphate, containing 38.8% Zn and 17.7% H₂O) is freed from sodium salts by extraction with water, the residue (46.8% Zn) being dissolved in 30% hydrochloric or sulphuric acid.

CHEMICAL ABSTRACTS.

[Determination of] lead in white arsenic. H. W. JONES (Chemist-Analyst, 1929, 18, No. 2: 11).—The

arsenic is removed by boiling with hydrochloric acid, and the lead is precipitated in neutral solution with sodium sulphide; it is then oxidised to sulphate, dissolved in ammonium acetate solution, and precipitated as chromate.

CHEMICAL ABSTRACTS.

Electrothermic manufacture of yellow phosphorus. J. E. SHIKHUTSKI (J. Chem. Ind. Moscow, 1928, 5, 979—982).—The charge is preferably finely divided and preheated at 200—300°, and the operation is continuous. Amorphous silica from kaolin is preferred to sand.

CHEMICAL ABSTRACTS.

Nitrogen determinations. SPEARS and TERRELL.—See XIX. Collargol. GERASIMOV.—See XX.

See also A., July, 774, **Electrolytic formation of sodium arsenate** (ESSIN). 779, **Antimony phosphate** (HORSCH). **Selenic acid and its salts** (HUFF and McCROSKY). **Molybdates** (ARANDA). **Silicotungstic acid** (SCROGGIE). 782, **Determination of chloride in bromides** (MCALPINE). **Iodo-oxidimetry** (SCHWICKER). **Volumetric determination of sulphate ion** (KÖSZEGI). **Electrolytic analysis of nitrates** (RABINOVITSCH and FOKIN). **Colorimetric determination of phosphoric acid** (BORDEIANU). 783, **Analysis of phosphoric acid** (ISHIBASHI). **Detection and determination of sodium** (KOLTHOFF). **Test for magnesium** (RUGH). **Indirect determination of lead** (FURMAN and WALLACE). **Determination of thallous salts** (JILEK and LUKAS). 784, **Determination of traces of mercury** (STOCK and ZIMMERMANN). 785, **Detection of elements of groups II—IV** (AGOSTINI). **Determination of tungsten in presence of vanadium** (JILEK and LUKAS). **Micro-determination of uranium salts** (TISSIER and BÉNARD). **Volumetric determination of vanadium** (SWIFT and HOEPEL). **Electrode for determination of halogens** (JOASSART and LECLERC).

PATENTS.

Manufacture of sulphuric acid. SOC. GEN. MÉTALL. DE HOBOKEN (B.P. 296,662, 28.6.28. Ger., 3.9.27).—Reaction towers, packed with filling materials, are inserted between the usual lead chambers, and are irrigated with nitrous sulphuric acid in such amount that the nitrous content of the acid discharged from the towers is at least 0.5% (calc. as nitric acid of *d* 1.33), i.e., the same as, or higher than, that of the acid introduced therein.

W. G. CAREY.

Joint production of sulphuric acid and cement. G. J. HARRIS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,661, 6.2.28).—A mixture of sand, clay, and calcium sulphate is ground with water, the slurry is dried, and the powder is fed into a kiln or furnace so that it comes into direct contact with a hot stream of gas, preferably generated by the combustion of powdered fuel in the kiln.

W. G. CAREY.

Conversion of sulphur dioxide into sulphur trioxide. J. G. MELENDY, ASS. to GEN. CHEM. CO. (U.S.P. 1,716,498, 11.6.29. Appl., 26.1.27).—Catalytic oxidation is carried on in adiabatic stages by controlling the temperature of the gases entering the conversion stages by positive cooling, so that the reaction in all but the final stage proceeds only to the point where the

reaction begins to fall off rapidly, and in the final converter is carried to equilibrium. W. G. CAREY.

Production of nitric acid. A. E. MITCHELL, C. C. SMITH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,934, 10.5.28).—Gases containing oxides of nitrogen and oxygen are exposed under pressure to moving, continuous, turbulent films of water or dilute nitric acid produced on one side of a vertical surface, heat being removed from the other side by a turbulent film of water. A nest of tubes, internally cooled by a film of water, may be used, the gases and absorption liquid circulating round the outside of them. W. G. CAREY.

Manufacture of silicic acid sols. I. G. FARBENIND. A.-G. (B.P. 294,264, 21.7.28. Ger., 21.7.27).—A silicic acid gel (10–15% SiO_2), made and purified from soluble constituents by known means, is treated with a small proportion of ammonia and is heated without dilution or evaporation until dissolution takes place.

W. G. CAREY.

Manufacture of silica and like gels. H. WADE. From SILICA GEL CORP. (B.P. 313,242, 30.3.28).—Silica hydrogels prepared as described in B.P. 136,543 and U.S.P. 1,297,724 (B., 1919, 363 A) are purified by successive washing with water, treatment with a strong acid, e.g., sulphuric acid, d 1.71, and a final washing with water. Alternatively, the gel, after the first washing with water, may be treated with sulphuric acid of progressively increasing (d 1.45–1.71) and then of decreasing (d 1.71–1.45) concentration.

L. A. COLES.

Preparation of catalytic gels. SILICA GEL CORP., Assees. of M. LATSHAW and W. L. JUDEFIND (B.P. 286,309, 2.3.28. U.S., 3.3.27).—Silica gels etc. are charged with a reducing gas other than hydrogen, e.g., carbon monoxide, sulphur dioxide, or hydrogen sulphide, and then sprayed with a solution of a compound, e.g., platonic chloride, which, by interaction with the gas, causes deposition of the desired metal within the pores of the gel.

L. A. COLES.

Catalytic and absorbent gel. W. A. PATRICK, Assr. to SILICA GEL CORP. (U.S.P. 1,696,644–5, 25.12.28. Appl., 15.3.26).—Hard absorbent gel having a porous structure similar to that of the silica gel of U.S.P. 1,297,724 (B., 1919, 363 A) and containing (A) copper or copper oxide or (B) nickel or nickel oxide is prepared from a soluble silicate, (A) a copper or (B) a nickel salt, and acid, the p_H after mixing being 0.3–0.9; the hydrogel is subsequently washed and dried.

R. BRIGHTMAN.

Manufacture and use of adsorbing agents containing silica. G. E. VAN NES (U.S.P. 1,715,439, 4.6.29. Appl., 30.4.27. Holl., 26.7.26).—A soluble silicate is added to a liquid to be decolorised, and an acid and a base which react together to give a precipitate are then introduced. Thus, after adding water-glass to molasses, milk of lime, magnesia, or aluminium hydroxide and carbon dioxide, sulphur dioxide, or phosphoric acid are introduced.

F. G. CLARKE.

Manufacture of adsorbing agents. A. ROSENHEIM (B.P. 284,245, 25.1.28. Addn. to B.P. 275,203; B., 1928, 659).—The zeolites etc. are heated, suspended

in water, and treated at raised temperature with sufficient acid to remove only the bases, leaving the silicic acid and the physical structure of the zeolitic material unchanged. The products are bonded together by pressure or by means of a binding agent (water-glass, resins).

W. G. CAREY.

Production of base-exchange materials. F. B. DEHN. From GEN. ZEOLITE CO. (B.P. 313,206, 9.3.28).—Non-friable artificial zeolites are prepared by mixing solutions of alkali silicates and alkali aluminates in the presence of sufficient acid to neutralise all or nearly all the free alkali produced, yielding a mixture alkaline to phenolphthalein or to methyl-orange, and subsequently drying and leaching the gel produced when the mixture sets.

L. A. COLES.

Manufacture of alkali of high concentration. F. BENCKER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,704,909, 12.3.29. Appl., 19.2.26. Ger., 14.5.25).—Amalgams are decomposed with water in presence of an organic nitro-compound; e.g., sodium amalgam decomposed at 80–100° by rapid agitation with a mixture of nitrobenzene and water affords a soda lye from which the hydrate, $3\text{NaOH} \cdot 4\text{H}_2\text{O}$, crystallises out, a mixture of azo- and hydrazo-benzene being obtained almost quantitatively as by-product.

R. BRIGHTMAN.

Production of ammonia by synthesis from its elements. F. W. DE JAHN, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,701,478, 5.2.29. Appl., 18.2.26).—The incoming gases from the purifiers are passed in cold exchange to the ammonia liquefiers, the uncondensed gases returning through the cold exchangers and passing thence in heat exchange with the hot reaction gases to the catalytic chamber. The preheated gases are finally brought to reaction temperature by passing them in free thermal contact with the catalyst, and then by reverse flow into direct contact with the catalyst, the cool uncatalysed gas serving to protect the walls of the reaction chamber against excessive temperatures.

R. BRIGHTMAN.

Concentration of brine. W. E. BURKE and H. DE ROPP, Assrs. to AMER. POTASH & CHEMICAL CORP. (U.S.P. 1,712,787, 14.5.29. Appl., 6.2.24).—Brines containing carbonates, sulphates, and chlorides of sodium and potassium which are substantially saturated with sodium chloride and contain less than the amount of sodium carbonate necessary to saturate the brine are evaporated to a concentration less than that at which glaserite forms, the temperature is raised, and the evaporation continued.

W. G. CAREY.

Manufacture of anhydrous sodium acetate. COURTAULDS, LTD., F. ADCOCK, N. G. BAGULEY, and D. L. WILSON (B.P. 313,352, 23.7.28).—Anhydrous acetic acid is treated at a raised temperature (30–60°) with sodium or with electrolytic sodium amalgam; sodium acetate combined loosely with, e.g., 1 or 2 mols. of acetic acid is precipitated by cooling the solution. The combined acetic acid may be expelled by heating the crystals after removal from the mother-liquor.

L. A. COLES.

Production of a disodium phosphate having two molecules of water of crystallisation. T.

BENCKISER, A. REIMANN, SEN., and A. REIMANN, JUN. (B.P. 308,566, 5.10.28. Ger., 23.3.28).—Pure phosphoric acid containing 600–700 g. P_2O_5 per litre is neutralised with the calculated amount of soda and the phosphate is crystallised above 50° . A more dilute solution may be used if the crystallisation is carried out at reduced pressure.

W. G. CAREY.

Manufacture of crystallised non-caking trisodium phosphate [deca]hydrate. L. R. WESTBROOK, ASSR. to GRASELLI CHEM. CO. (U.S.P. 1,711,707, 7.5.29. Appl., 27.1.28).—An aqueous saturated solution of the dodecahydrate is heated above its crystallisation temperature, seeded with decahydrate crystals, and the resulting decahydrate crystals are removed.

W. G. CAREY.

Production of sodium perborate. H. R. CARVETH, ASSR. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,716,874, 11.6.29. Appl., 27.9.27).—Sufficient sodium peroxide is added to an aqueous solution of borax to convert it into a mixture of metaborate and perborate, oxidation being completed by the addition of hydrogen peroxide.

H. ROYAL-DAWSON.

Production of dry ferric chloride, manganic chloride, or the like metallic chlorides. VEREIN. STAHLWERKE A.-G. (B.P. 290,568, 3.5.28. Ger., 14.5.27).—Volatilised chlorides obtained by the chlorination of appropriate ores are purified from hydrochloric acid and steam by passing through magnesium oxide or similarly acting oxides at a temperature near that of vaporisation of the chloride. By-product magnesium oxide from the "abraum-salts" of the potash industry may be used.

W. G. CAREY.

Production of aluminium sulphate. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 312,726, 19.3.28).—Clay, bauxite, etc. is treated in a rotary kiln in countercurrent with a stream of gases containing sulphur trioxide or sulphur dioxide and oxygen at a temperature below the decomposition temperature of ferric sulphate, and the mass subsequently heated to a higher temperature to decompose the ferric sulphate and form aluminium sulphate.

W. G. CAREY.

Production of aluminium chloride. II. BLUMENBERG, JUN. (U.S.P. 1,716,102, 4.6.29. Appl., 31.8.26).—A mixture of aluminium sulphide and an alkali chloride is dehydrated, carbonaceous material is mixed therewith, and aluminium chloride is distilled off, in a closed retort, below the volatilisation point of the alkali chloride.

F. G. CLARKE.

Preparation of Epsom salts. S. B. HEATH, ASSR. to DOW CHEM. CO. (U.S.P. 1,709,398, 16.4.29. Appl., 23.2.25).—When making magnesium sulphate from magnesium hydroxide containing chloride impurities, an excess of sulphuric acid is heated with concentrated mother-liquor from a previous run to remove hydrochloric acid as gas, and the liquid is then added to magnesium hydroxide.

W. G. CAREY.

Manufacture of barium compounds [silicates]. S. WITTOUCK (B.P. 312,975, 3.3.28).—Barium compounds other than the carbonate, e.g., barium sulphate, are calcined at about 1500° with silica and, if desired, carbon, in the presence of a portion of the product

obtained by a previous calcination in quantity sufficient to prevent fusion.

L. A. COLES.

Calcination of lime. K. M. NAHIKIAN, R. W. FOSTER, and E. T. BELDEN, ASSRS. to BREWER & CO., INC. (U.S.P. 1,709,226, 16.4.29. Appl., 27.4.28).—A mixture of an alkaline-earth oxide or hydroxide and water is introduced as a spray into the bottom of a vertical kiln so that it comes into contact with the products of combustion from the fuel before they reach the limestone to be treated and precipitates hard and non-plastic products.

W. G. CAREY.

Solidification of nitrate of lime by rotary cooling drums. APPAREILS ET EVAPORATEURS KESTNER (B.P. 292,532, 23.5.28. Fr., 21.6.27).—Adherence of the nitrate to the drum is reduced by interposing a skin of supersaturated solution free from crystals between the drum and a layer of partially crystallised nitrate solidified on it, by heating the surface of the solution primed for crystallisation contained in the basin of the drum, or by heating the surface of the drum between the scraper and the line at which the drum enters the solution.

L. A. COLES.

Treatment of acidulous by-product calcium sulphate. R. S. EDWARDS, ASSR. to RUMFORD CHEM. WORKS (U.S.P. 1,713,868, 21.5.29. Appl., 18.7.28).—The slurry of calcium sulphate from the manufacture of phosphoric acid is simultaneously ground and neutralised, and is then passed to ovens for the production of an acid-free plaster.

F. G. CLARKE.

Fire-extinguishing liquid. R. W. AUSTIN (U.S.P. 1,716,476, 11.6.29. Appl., 18.7.25).—A solution of calcium chloride and potassium carbonate is claimed for anti-freezing extinguishers.

J. A. SUGDEN.

Manufacture of calcium hypochlorite. MATHIESON ALKALI WORKS, INC., ASSCES. OF [A, C, D] A. GEORGE and R. B. MACMULLIN, [B] J. A. GUYER (U.S.P. 1,713,650, 1,713,654, and 1,713,668—9, 21.5.29. Appl., [A—D] 4.8.26).—(A) The crude hypochlorite, in the form of a slurry, is agitated with alkali hypochlorite equivalent in quantity to the calcium chloride present. When well mixed, the product is dried. (B) Caustic alkali is used in place of alkali hypochlorite. (C) An aqueous mixture of lime and caustic alkali, the latter being in excess, is chlorinated. A calcium compound equivalent in quantity to the alkali is added, and the calcium hypochlorite separated. (D) Milk of lime is chlorinated, caustic alkali added, and the mixture further chlorinated, when the calcium hypochlorite is separated.

F. G. CLARKE.

Manufacture of complex tungsten and molybdenum compounds [and pigments, colour lakes, etc. containing them]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,209, 9.3.28).—Mixtures giving rise to the complex compounds, e.g., aqueous solutions containing sodium tungstate, sodium phosphate, and sodium molybdate, are treated with reducing agents, e.g., sodium hyposulphite, sodium bisulphite, or dextrose, preferably in an acid medium; or, alternatively, the tungstates and molybdates are reduced before admixture with sodium phosphate etc. The complex compounds may be precipitated by salting

out or by treatment with hydrogen chloride, or may be recovered by concentration; or pigments etc. may be precipitated by adding to the solutions basic dyes or aromatic amines free from acid groups together, if desired, with substrata and/or dispersive agents.

L. A. COLES.

Colloidal sulphur and its manufacture. A. HARTZELL and F. H. LATHROP, Assrs. to W. C. O'KANE and P. MOORE (CROP PROTECTION INST.) (U.S.P. 1,705,862, 19.3.29. Appl., 10.6.24).—Sulphur is dissolved in naphthalene or other solid distributing agent, *e.g.*, paraffin wax, and disintegrated to a fine dust or emulsified, *e.g.*, in a solution containing fish-oil soap.

R. BRIGHTMAN.

Manufacture of pure hydrofluoric acid from polluted fluorspars. M. BUCHNER, Assr. to A. F. MEYERHOFFER (U.S.P. 1,701,225, 5.2.29. Appl., 5.1.26. Ger., 16.1.25).—See B.P. 234,852; B., 1925, 670.

Manufacture of alkali hydroxides. L. P. CURTIN (B.P. 312,732, 20.3.28).—See U.S.P. 1,678,767; B., 1928, 746.

Treatment of solutions containing tin and arsenic for precipitation of tin. H. HARRIS (U.S.P. 1,714,667, 28.5.29. Appl., 9.9.25. U.K., 18.9.24).—See B.P. 244,526; B., 1926, 156.

Phosphorescent or luminous mass or compounds. H. M. MINES, Assr. to F. CURD (U.S.P. 1,716,972, 11.6.29. Appl., 10.5.26. U.K., 18.5.25).—See B.P. 245,612; B., 1926, 248.

Production of hydrogen-nitrogen mixtures. L. CASALE (M. CASALE-SACCHI, adrix.) (U.S.P. 1,716,813, 11.6.29. Appl., 15.6.27. It., 24.6.26).—See B.P. 292,342; B., 1928, 603.

Apparatus for centrifugal separation (U.S.P. 1,700,928).—See I. **Brazing flux** (U.S.P. 1,717,250).—See X. **Piezo-electric substances** (B.P. 311,055). **Electrolysis of chlorides** (B.P. 309,408).—See XI.

VIII.—GLASS; CERAMICS.

Use of trachyte in the manufacture of glass. I. I. KITAIGORODSKI and S. V. RODIN (Trans. State Exp. Inst. Silicates, Moscow, 1928, 3—62).—Trachyte, containing ($K_2O + Na_2O$) 5—17, SiO_2 63—73, Al_2O_3 12—25, Fe_2O_3 1—4, ($CaO + MgO$) 1—5%, although high in alumina, was used in glass manufacture in proportions of 85—88%, with lime or dolomite and 0—5% of sand. Barium could be added as the carbonate, but the sulphate produced a poor glass. Trachyte glass is resistant to acids and alkalis, and has a low linear coefficient of expansion and good mechanical properties.

CHEMICAL ABSTRACTS.

Colouring of glass. K. FUHA (J. Mazda Res. Lab., 1928, 3, 21—49).—Ammonium molybdate does not colour glass, but sometimes causes opalescence on reheating. Tungstic oxide and tungstates do not impart a colour, but produce opalescence. Metallic tungsten produces a grey colour. For the preparation of fluorescent glass with uranium compounds, potassium and boric acid should be present; potash-magnesia and potash-zinc-boron-silica glasses acquire a strong fluorescence.

CHEMICAL ABSTRACTS.

Properties of simple enamel glasses. A. I. ANDREWS (J. Amer. Ceram. Soc., 1929, 12, 390—394).—The two systems $Na_2O-B_2O_3-SiO_2$ and $Na_2O-PbO-SiO_2$ were tested for acid-resistance (spot test with 70% citric acid, also treatment of powder with constant-boiling hydrochloric acid at 100°) and for general qualities. Such glasses were successfully applied to cast iron by the dry process and made into enamels by the addition of tin oxide. An increase in silica content generally (but not always) increases the acid-resistance. The effect of soda, boric acid, and lead oxide is dependent on the composition to which they are added.

J. A. SUGDEN.

Importance of the glass phase in porcelain. H. M. KRANER (J. Amer. Ceram. Soc., 1929, 12, 383—389).—The properties of a body depend as much on the nature of the glass as, or more so than, on the crystal structure. Support is given to the idea that dissolution of mullite and quartz may cause weakening of porcelain with excessive fire and may consequently lower the resistance to thermal shock in spite of a decrease in the coefficient of expansion. Small changes in chemical composition cause vital changes in the properties of the glass.

J. A. SUGDEN.

Determination of air in plastic [clay] mixes. V. CARTWRIGHT (J. Amer. Ceram. Soc., 1929, 12, 395—398).—The sample is boiled with water in a vessel sealed at the bottom with mercury, and the air evolved is measured in a burette so arranged that a definite temperature and pressure can be applied.

J. A. SUGDEN.

Clay sewer-pipe manufacture. VI. **Heat-balance determinations.** VII. **Low-temperature oxidation [of clays etc.].** R. E. ARNOLD and G. H. DUNCOMBE, JUN. VIII. **Measurement of draught distribution.** C. E. PARMELEE (J. Amer. Ceram. Soc., 1929, 12, 410—422, 423—428, 429—433).—VI. The method of calculation of kiln efficiency is described and the results of a large number of tests are given. The efficiency was found to vary from approx. 15% to 29%, although over some ranges of temperature a value as high as 40% was found. In general, the efficiency decreases with increase of temperature.

VII. Plant experiments showed that "black cores" are most readily eliminated by slow heating over the range 250—550°. Tests showed that the black core material consisted chiefly of carbon with some magnetic oxide. Laboratory tests showed that some forms of carbon burn much more readily than others, and it is suggested that oxidation in plant practice takes place with difficulty at higher temperatures because at such temperatures the carbon is converted into a slower-burning variety. The catalytic effect of certain metallic oxides on the oxidation of carbon has been studied, and the "accelerators" are found to be those usually occurring in clays.

VIII. The methods of measurement and calculation and possibilities in the correction of faulty conditions are described.

J. A. SUGDEN.

See also A., July, 762, **Colloid-chemical properties of kaolin** (SHUKOV and SOKOLOVA). 766, **Ceramics of**

refractory substances: system $\text{ZrO}_2\text{--ThO}_2$ (RUFF and others).

PATENTS.

Lehrs, annealing furnaces, etc. UNITED GLASS BOTTLE MANUFGRS., LTD., T. C. MOORSHEAD, and F. A. HURLBUT (B.P. 312,890, 2.11.28).—A continuous, wire-woven or similar conveyor belt is preheated by passing the return belt in close proximity to the upper portion. This is accomplished by supporting the return belt on rollers and allowing it in turn to support the upper belt through a series of transverse floating rollers. In addition, the belt passes around a hollow preheating drum. A. COUSEN.

Glass. CHANCE BROS. & Co., LTD., C. E. GOULD, W. M. HAMPTON, and H. S. MARTIN (B.P. 312,728, 19.3.28).—A non-solarising, ultra-violet light-transmitting glass is made from such ingredients as silica and anhydrous borax together with a powdered metal such as zinc, aluminium, or tin, which acts as a reducing agent and prevents the presence of deleterious gaseous impurities, such as carbon dioxide, in the glass. A. COUSEN.

Manufacture of compound glass. E. GEISEL (B.P. 313,269, 2.5.28).—Condensation products of formaldehyde and urea, thiourea, or other urea derivatives are applied in liquid form between glass plates and allowed to harden, with or without addition of accelerating catalysts. A. COUSEN.

Non-splintering glass sheets or plates. J. TALBOT (B.P. 313,273, 7.5.28).—A single sheet of glass is sealed between sheets of cellulose acetate, and the edges are sealed by coating with a viscous solution of the latter material. A. COUSEN.

Bubble or blister glass. R. HALEY (U.S.P. 1,715,130, 28.5.29. Appl., 7.10.27).—A core of glass gathered on the blow pipe is coated with finely-divided bubble-forming material which is covered with a further layer of glass. The mass is then blown to shape. J. A. SUGDEN.

Tunnel ovens and the like. GIBBONS BROS., LTD. From AMER. DRESSLER TUNNEL KILN, INC. (B.P. 312,786, 16.5.28).—The oven comprises preheating and firing zones, and products of combustion passing out from the latter by side-wall ports are caused to flow through banks of horizontally extending pipes placed along the sides of the preheating zone. These pipes pass through chambers formed in the walls of the kiln which serve to heat the preheating air. Circulation of the air is obtained by exhaust fans which draw it into pipes placed along the crown of the kiln and drive it down into the preheating chambers, whence it flows once again into the kiln by a series of injector nozzles suitably placed in the wall. A. COUSEN.

Production of clear, vitreous silica. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of B. F. NIEDERGESASS (B.P. 296,059, 22.8.28, U.S., 24.8.27).—Finely-powdered silica is introduced into the flame of a gas burner and is made to impinge on to an opaque silica foundation, which is maintained at 800° . The clear layer thus deposited may be ground and polished to the desired shape while still adhering to the foundation. F. SALT.

Ceramic product moulded by casting in a hot state. PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H., Assees. of R. MOHR and H. BECKER (B.P. 281,249, 3.11.27. Ger., 26.11.26).—A readily fusible siliceous rock, such as phonolith or its decomposition products, is melted with at least 20% of fluorspar or other mineral containing fluorine, alumina being added, if necessary, in the form of clay or kaolin in the proportion of 5–20%. The melting operation is interrupted before a homogeneous glass is formed. F. SALT.

Manufacture of refractory compositions. T. S. CURTIS, Assr. to PACIFIC-SOUTHWEST TRUST & SAVINGS BANK (U.S.P. 1,715,449, 4.6.29. Appl., 11.12.24).—A mixture of magnesia (not less than 35%) and iron oxide (not more than 50%) is heated to a temperature sufficient to sinter the mass. J. A. SUGDEN.

Manufacture of refractory composition and articles therefrom. L. J. TROSTEL, Assr. to GEN. REFRACTORIES Co. (U.S.P. 1,716,395, 11.6.29. Appl., 7.6.26).—Cyanite is added to a highly aluminous clay mix in order to compensate for the firing shrinkage of the clay. J. A. SUGDEN.

Refractory materials. J. A. JACK (B.P. 312,700, 6.3.28).—A material resistant to slagging by alkalis (to be used in the removal of sulphur and silicon from metals) is prepared by adding 5–25% of carbon to a highly siliceous (ganister) mix. J. A. SUGDEN.

Kiln box or support for firing bull-nose and other curved glazed tiles in the oven or kiln. W. R. BROAD, A. E. LOVATT, and G. G. PETRIE (CROMLECH TILE Co.) (B.P. 313,026, 5.3.28).

Saggers or receptacles for use in firing pottery. A. HEATH and H. COLCLOUGH (B.P. 312,794, 21.5.28).

Feeding of [molten] glass. W. J. MILLER (B.P. 312,887, 18.10.28).

Drawing of sheet glass. P. HADDAN. From VIRGINIA PLATE GLASS CORP. (B.P. 313,821, 24.9.28).

Manufacture of compound sheets of glass and cellulose derivatives. G. E. HEYL (B.P. 313,695, 19.4.28).

Refractory brick (U.S.P. 1,714,506).—See IX.

IX.—BUILDING MATERIALS.

Use of plaster of Paris and allied substances for dental models. R. N. JOHNSON and C. S. GIBSON (Brit. Dental J., 1929, 50, 681–696; cf. Sodeau and Gibson, B., 1927, 878).—The setting of ordinary “fine” plasters and artificial stone compositions has been studied. The compositions included both the anhydrous calcium sulphate (AS7) and the cement plaster (AS8) types. Measurements were made of setting time, expansion, hardness (Brinell), and crushing strength. A decrease in the amount of mixing water (compatible with good mixing) causes an increase in hardness, strength, and expansion, and an acceleration of setting. “Over-stirring” greatly increases the expansion. When anti-expansion solution is used the amount of liquid or time of mixing has little effect on the expansion. Air-drying at room temperature increases the hardness, strength, and expansion. Maximum hardness of plaster

and ASS (mixed with water or anti-expansion solution) is reached in two or three days, but the AS7 type continues to harden for seven days or more. Plaster and artificial stone, mixed with water, show the maximum expansion, hardness, and strength at about the same time, but when plaster is mixed with anti-expansion solution the maximum expansion is reached in several hours. Increase in hardness continues to take place after the material has reached complete dryness. Drying in an oven causes the hardness and strength to increase to a maximum and then decrease again. The higher is the temperature the sooner the reversion occurs. A new anti-expansion solution (4% of Rochelle salt and 0.2% of borax) makes a harder material which shows smaller dimensional changes in the vulcaniser. The hardness and strength of all materials are greatly reduced in the vulcaniser, and the dimensional changes are not uniform in all directions. Freshly cast material shows greater change in dimensions than that which has been allowed to air-dry. The formation of a coarse granular product in the vulcaniser is governed by the temperature. The granular product has a high combined water content, but the normal product is almost anhydrous. A tentative explanation of this change is suggested.

J. A. SUGDEN.

PATENTS.

Burning of lime. C. J. WRIGHT, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,716,572, 11.6.29. Appl., 15.8.23).—As the limestone passes down a shaft it is decomposed by an upward current of steam and carbon dioxide, which is introduced circumferentially into the mid-portion of the shaft; the resulting lime is cooled to a point just above the dissociation temperature of the hydrate, as it passes to the exit at the base, by a current of saturated steam introduced at the bottom.

F. G. CLARKE.

Manufacture of hydraulic cement. R. DE LUCE (U.S.P. 1,713,172, 14.5.29. Appl., 11.1.27).—A mixture of lime, silica, alumina, and water in proportions suitable for the production of Portland cement is violently agitated at 100° under pressure until an insoluble calcium aluminium silicate is formed, which is subsequently heated to incipient fusion.

W. G. CAREY.

[Refractory] brick and cement for furnace use. R. P. HEUER, Assr. to GEN. REFRACTORIES CO. (U.S.P. 1,714,506, 23.5.29. Appl., 29.12.24).—Small amounts of clay, magnesia, and a solution of a magnesium salt are added to chrome ore body.

J. A. SUGDEN.

Utilisation of molten slag and production of cast slag in a form suitable for use in road-making. C. A. MIDGLEY and J. T. GOODWIN (B.P. 312,838, 29.6.28).—A mould for casting slag consists of a shallow frame with detachable sides and perforated cross-partitions. The base of the mould may be of broken chippings of granite or other material suitable as a facing material for the blocks.

C. A. KING.

Binding agent for materials used for road construction. F. C. HOPKIRK, and C. TENNANT & Co., LTD. (B.P. 313,824, 29.9.28).—Tar (61%) and light coal-tar oil (5%) are mixed and hot bitumen (34%) is added at not above 150°, the mixture being agitated by revolving beaters.

W. G. CAREY.

[Bituminous] emulsion and its manufacture. L. KIRSCHBRAUN (U.S.P. 1,707,809, 2.4.29. Appl., 17.1.21).—Bituminous material, *e.g.*, asphalt, is melted and mixed at 150–190° with an aqueous suspension of colloidal clay, containing talc, or other protecting agent. The clay etc. may also be added to the mixture after emulsification.

R. BRIGHTMAN.

Impregnation [staining] of wood. H. P. VANNAH and J. G. GOSSELINK, Assrs. to BROWN CO. (U.S.P. 1,714,701, 28.5.29. Appl., 20.5.27).—An acid solution of a dye intermediate, which precipitates a pigment on oxidation in an alkaline medium, *e.g.*, 2:4-diaminophenol hydrochloride or 2-naphthylamine-1-sulphonic acid, and then an aqueous alkali are successively sucked through the wood, preferably after air has been removed therefrom.

F. G. CLARKE.

Mixing of quick-setting cementitious materials. G. M. THOMSON (B.P. 313,018, 31.1.28).—See U.S.P. 1,660,242; B., 1928, 334.

Manufacture of aluminous cements. E. MARTIN (U.S.P. 1,716,527, 11.6.29. Appl., 27.4.26. Fr., 30.4.25).—See F.P. 597,978; B., 1926, 409.

Rotary mixing machines [for concrete etc.]. FILS DE JULES WEITZ (B.P. 306,504, 11.9.28. Fr., 22.2.28).

Manufacture of artificial stone specially applicable to concrete tiles. R. ABRAHAM, LTD., and R. A. ABRAHAM (B.P. 313,295, 26.5.28 and 26.3.29).

Manufacture of [hollow] tiles. NAT. FIRE PROOFING CO., Assees. of R. A. SHIPLEY (B.P. 296,369, 31.7.28. U.S., 30.8.27).

Preservation of wood [in situ and under reduced pressure]. W. B. MAKINS, and BROWN & MAKINS, LTD. (B.P. 312,719, 13.3.28).

Tar products (B.P. 312,372).—See II. **Sulphuric acid and cement** (B.P. 309,661). **By-product calcium sulphate** (U.S.P. 1,713,868).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Metallisation of the oxide of iron in ilmenite. R. J. TRAILL and W. R. MCCLELLAND (Amer. Electrochem. Soc., May, 1929. Advance copy. 10 pp.).—A process for recovering iron and a titanium dioxide concentrate from ilmenite or titaniferous magnetite has been investigated on a semi-large scale. The crushed ore mixed with crushed coal and coke is heated at about 1000° in a rotary kiln with exclusion of excess air. By magnetic separation the reduced product yields a magnetic concentrate consisting of metallic iron and titanium dioxide. The iron can be removed by leaching with sulphuric acid or, if it is to be recovered as electrolytic iron, by leaching with ferric chloride solution. The residue is a high-grade concentrate of titanium dioxide low in iron.

H. J. T. ELLINGHAM.

Sponge iron, a raw material for electric steel. N. K. G. THOLAND (Amer. Electrochem. Soc., May, 1929. Advance copy. 22 pp.).—Sponge iron is now produced on a commercial scale at Höganäs, Sweden, from the purest Swedish magnetite. The product contains over

95% Fe (usually 96—97.5%), with an average of 0.014% P and 0.025% S. Sponge iron is advantageous as a base for fine steels and alloy steels owing to its comparative purity. Comparative figures for the properties of steels manufactured from sponge iron, high-grade scrap, and pig iron are quoted, and the advantages and future prospects of sponge iron are discussed.

H. J. T. ELLINGHAM.

Mechanical and metallurgical properties of spring steels as revealed by laboratory tests. G. A. HANKINS and (Miss) G. W. FORD (Iron and Steel Inst., May, 1929. Advance copy. 28 pp.; cf. B., 1926, 827).—Endurance fatigue limits were determined on low-chromium, high-chromium, nickel-chromium, and two water-quenched carbon steels in various conditions of heat treatment, the quenching temperatures being arranged to produce the desired hardening conditions. The fatigue limits so obtained on polished specimens are higher than those given by spring plates and wires under normal conditions; this is probably due to the presence of a thin layer of soft decarburised material at the surface of the latter, and also to lines and scratches which reduce the effective resistance. The endurance fatigue limit of any particular steel under any particular heat treatment is closely related to the tensile strength and Brinell number, and is little affected by the composition. Generally, the proportional limits increase with increasing tensile strength and Brinell number, but variations are much more marked than for fatigue limits. For each steel the Izod numbers decrease with increasing tensile strength and Brinell numbers. Other things being equal, the highest Izod number is the best one to select. A good combination of properties is exhibited by chromium-vanadium and water-quenched silicon-manganese steels; the water-quenched 0.46% carbon steel is definitely inferior to the alloy steels and to the 0.6% carbon steel oil-quenched from 950°. It is suggested that the mean values of 30.1×10^6 lb./in.² for the elastic modulus and 11.6×10^6 lb./in.² for the modulus of rigidity may be used with safety for all spring steels, irrespective of composition or heat treatment.

M. E. NOTTAGE.

Recovery of hyper-tempered steels. P. CHEVENARD and A. PORTEVIN (Compt. rend., 1929, 188, 1670—1672).—Priority is claimed for the two types of dilatometric recovery-cycle of austenitic steels in the non-equilibrium state (hyper-tempered) described by Michel and Benazet (B., 1929, 358). Examples are: (1) a hyper-tempered manganese-carbon steel (2.26% Mn, 1.5% C) in which austenite is converted by heat into ($\text{Fe}_3\text{C} + \text{Fe}_\alpha$) or sorbite, on cooling of which there is no further reaction; and (2) a hyper-tempered chromium-carbon steel (2.26% Cr, 1.5% C) in which austenite is converted by heat into a less carburised form and a complex iron chromium carbide, whilst cooling produces martensite (secondary tempering). In both cases there is an increase in hardness of about 200 Brinell.

J. GRANT.

Normalising of sheet steel. W. PARKER (Engineering, 1929, 127, 799).—Lack of uniformity of annealing of steel sheets by the older methods in boxes has led to the development of single-sheet annealing now

almost entirely used in the United States. Steel sheets, singly or at most in packs of two or three, are conveyed through an open furnace on power-driven discs forming a conveyor. The furnace is gas-heated and the various conditions of heating, cooling, and atmosphere can be controlled to give the most uniform crystal structure throughout the material. The time required for the complete cycle is about 8 min. as compared with about 32 hrs. by the box method.

C. A. KING.

Influence of various elements on the nitrogenation of steel. T. NAKAYAMA (J. Study Met., 1928, 5, 413—427).—When iron is heated in a current of ammonia, the increase in weight corresponds with the nitrogen content. The velocity of nitrogenation is small at 300°, and increases rapidly with rise of temperature above 400°. The nitrogen absorption in 1 hr. is maximal (10%) at 450°, decreasing nearly to zero at 750°. For large particles the velocity is small. Elements which form stable nitrides (aluminium, silicon, titanium, chromium), when alloyed with the iron accelerate nitrogenation (max. after 1 hr. at 500°). With copper, nickel, or cobalt, as also with manganese, the maximum is at 600°. When aluminium, silicon, titanium, chromium, or manganese is present, much nitrogen is retained at a high temperature.

CHEMICAL ABSTRACTS.

Rapid determination of vanadium in ferro-vanadium. J. SILBERSTEIN (Chemist-Analyst, 1929, 18, No. 2, 11).—The material is fused with sodium hydroxide in a nickel crucible, and an aliquot part of the aqueous extract is treated with potassium iodide in presence of phosphoric acid. After 5 min. the quadrivalent vanadium is titrated with 0.05N-sodium thiosulphate.

CHEMICAL ABSTRACTS.

Thermal and electrothermal reduction of zinc oxide. O. DONY-HENAUET (Amer. Electrochem. Soc., May, 1929. Advance copy. 4 pp.).—Electrical heating by means of a resistor consisting of a single piece of soft iron proved satisfactory up to 1200°. Furnaces of this type have been used in a study of the velocity of reduction of zinc oxide by carbon monoxide at various temperatures and pressures.

H. J. T. ELLINGHAM.

Rapid determination of tin in lead-base alloys containing antimony. T. B. DIANA (Chemist-Analyst, 1929, 18, No. 2, 8).—The alloy, mixed with sufficient tin to increase the tin content to at least three times that of the antimony, is decomposed with 7.5N-nitric acid; the excess of acid is boiled off, and 40 c.c. of water are added. The precipitate is collected and washed ten times with hot water and 1.5N-nitric acid, alternately. The weight of antimony tetroxide, separately determined, is subtracted from that of the residue, giving the weight of stannic oxide. Some lead is usually present.

CHEMICAL ABSTRACTS.

Preparation and mechanical properties of duralumin. M. GOTO, S. FUKATU, S. HORIGUCHI, and T. NAGAI (Rep. Aeronaut. Res. Inst. Tokyo, 1928, 3, 273—403).—The cast metal can be rolled at $450 \pm 20^\circ$. Iron improves the pouring and rolling qualities, but is otherwise undesirable; 1% of silicon improves the mechanical properties, but large quantities are undesirable. Zinc up to 3% is advantageous. Alkalis are

undesirable. Silver improves the malleability of the poured metal, which normally contains Cu 3—4, Mg 0.5—0.75, Mn 0—0.5, Al 96.5—94.75%. A new hypothesis of the ennoblement of aluminium alloys is developed.

CHEMICAL ABSTRACTS.

Tungsten carbide alloys. J. L. GREGG and C. W. KÜTTNER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 184, 3—12).—Of five commercial alloys, four consisted of W_2C and WC, the fifth being almost entirely WC.

CHEMICAL ABSTRACTS.

Electrolytic zinc refining. II. Influence of foreign elements on the hydrogen overvoltage. H. SETO (Hokko, 1928, 24, 1—22).—The relation between the overvoltage of platinum, copper, iron, lead, zinc, cadmium, nickel, and cobalt in 2*N*-sulphuric acid and their surface condition was examined. There is no great difference in the overvoltage when the plated surface is smooth or crystalline, whilst that obtained with spongy platinum, spongy nickel, or platinised platinum is much smaller. Addition of gelatin or cinchonine increases the hydrogen overvoltage. Dextrose, tannin, and particularly gelatin, when added to a zinc sulphate solution, decrease the overvoltage at the surface of the deposited zinc, probably owing to small adsorption of gas.

CHEMICAL ABSTRACTS.

Measurements of hydrogen-ion concentration in plating baths. R. E. BREWER and G. H. MONTILLON (Amer. Electrochem. Soc., May, 1929. Advance copy. 26 pp.).—The suitability of electrometric and colorimetric methods of measuring the p_H value of typical baths for plating zinc, iron, cobalt, nickel, and chromium has been investigated. Electrometric measurements were made with the hydrogen, oxygen, quinhydrone, antimony-antimony trioxide, and lead dioxide electrodes, employed either in combination with the corresponding electrode in a standard buffer solution or with a standard calomel electrode. The oxygen electrode functioned satisfactorily in all cases except in acid iron baths, where oxidation of the bath occurred, and in chromium-plating baths. The antimony-antimony trioxide electrode is unsuitable for baths of high acidity and was found to be unsatisfactory in the cobalt bath examined, but this electrode and the lead dioxide electrode are the only two which are suitable for the highly oxidising chromium bath. Otherwise the quinhydrone electrode is recommended for general usefulness and ease of measurement. The accuracy of Thompson's colorimetric drop-ratio method (B., 1922, 469) is checked by electrometric titrations using oxygen or quinhydrone electrodes. The variation of the potentials of oxygen and antimony-antimony trioxide electrodes with change of p_H has been re-examined and empirical relations are given.

H. J. T. ELLINGHAM.

Electrolysis of chromium sesquioxide. EWING and others.—See XI.

See also A., July, 766, **System cadmium-antimony** (MURAKAMI and SHINAGAWA). 774, **Electrodeposition of chromium** (ROUDNICK). 775, **Electrolytic copper** (MARIE and JACQUET).

PATENTS.

Furnace for fusing finely-divided materials, and agglomeration of dust from blast-furnaces. A.

DAWANS (B.P. 294,639, 15.6.28. Belg., 29.7.27).—In an L-shaped furnace chamber finely-divided material is melted on the hearth by means of a blast burner, and is discharged down the inclined hearth. The upright limb of the furnace forms a chimney inside of which is a separate duct to preheat the fine material and to feed it on to the hearth.

C. A. KING.

Making of castings. ANNENER GUSSTAHLWERK A.-G. (B.P. 311,393, 22.10.28. Ger., 12.5.28).—For castings which in certain parts are subject to heavy wear, e.g., tram and railway rails, crossings, etc., a hard wear-resisting alloy is first cast in a mould to the required thickness, and as soon as it has cooled to a pasty consistency the mould is filled with a softer supporting metal.

C. A. KING.

Case-hardening of iron. C. HUMMEL (B.P. 312,759, 16.4.28).—A liquid cementation medium is atomised and injected into a retort by means of a gas under pressure, which itself acts as an accelerator of the process.

C. A. KING.

Fire-resistant [iron] alloy with high electric resistance. H. G. A. VON KANTZOW (U.S.P. 1,717,284, 11.6.29. Appl., 19.12.27. Swed., 15.12.26).—An alloy containing iron alloyed with 0.5—14% Al, up to 30% Cr, and 0.5—6% Co is claimed.

H. ROYAL-DAWSON.

[Corrosion-resistant steel] alloys. (SIR) R. A. HADFIELD (B.P. 313,471, 10.3.28).—The alloy contains, in addition to iron, 0.05—1% C, 2—20% Ni, 5—30% Cr, 1—10% W, and 0.20—5% Cu, with or without 0.05—4% Si and 0.10—4% Mn; the combined nickel and chromium content should be 18—32%.

M. E. NOTTAGE.

Refining of copper. H. II. ALEXANDER (B.P. 313,270, 3.5.28).—Copper is refined in a furnace by excluding any air above the molten metal by the introduction of superheated steam, which may also be caused to inject a reducing agent on to the metal.

C. A. KING.

Mechanically-worked zinc product. W. M. PEIRCE and E. A. ANDERSON, ASSTS. to NEW JERSEY ZINC CO. (U.S.P. 1,716,599, 11.6.29. Appl., 9.2.28).—A zinc-base alloy, free from aluminium, is used containing at least 92% Zn, 0.005—0.5% Mg, and 0.05—5% Cu.

F. G. CROSSE.

Metal foil. E. C. R. MARKS. From AMER. MACHINE & FOUNDRY CO. (B.P. 313,300, 29.5.28).—To replace tin foil an alloy of lead containing 0.25—0.5% Mg is rolled down to less than 0.0005 in., preferably 0.00045 in. in thickness.

C. A. KING.

Metallic alloy. C. A. BOULTON (B.P. 312,845, 11.7.28).—An alloy resistant to acids and alkalis contains 8—15% (11.3%) Sn, 64—81% (73.2%) Cu, 10—20% (15.4%) Pb, 0.06% P, and 0.04% Zn, by wt.

F. G. CROSSE.

Chilled-roll alloy. F. D. TAGGART (U.S.P. 1,707,332, 2.4.29. Appl., 16.7.27).—The alloy contains approx. 3.5% C, 1.75% Cr, 0.2% V, 1% Si, 0.5% Mn, and traces of sulphur and phosphorus.

H. ROYAL-DAWSON.

Non-ferrous welding rod. A. R. LYTLE, ASST. to UNION CARBIDE & CARBON RES. LABS., INC. (U.S.P. 1,716,590, 11.6.29. Appl., 6.12.27).—The rod contains

55–65% Cu, 0.1–4% Si, and zinc in amount such that the sum of the copper and zinc contents is 82.5%.

F. G. CROSSE.

Flux for welding or soldering of aluminium and other metals. W. REUSS (B.P. 310,226, 16.6.28. Addn. to B.P. 296,923; B., 1928, 821).—Common salt (15 pts.) and sulphuric acid (1 pt.) are added to 30 pts. of the flux described in the prior patent.

H. ROYAL-DAWSON.

Brazing flux. J. J. PHELAN, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,717,250, 11.6.29. Appl., 19.6.28).—The flux consists of not less than 25% of potassium hydrogen fluoride and at least 40% of potassium tetraborate.

H. ROYAL-DAWSON.

Metal cleaning. J. H. GRAVELL, Assr. to AMER. CHEM. PAINT Co. (U.S.P. 1,713,653, 21.5.29. Appl., 22.6.26).—A mixture for cleaning metal, *e.g.*, motor bodies, comprising an acid and an alcohol, or other solvent for grease, contains arsenic, so that the latter is deposited upon the cleaned metal, and rusting prevented.

F. G. CLARKE.

Metallurgical furnace. B. TALBOT (U.S.P. 1,716,391, 11.6.29. Appl., 31.1.25. U.K., 24.4.24).—See B.P. 235,346; B., 1925, 638.

Manufacture of malleable-iron castings. NAT. MALLEABLE & STEEL CASTINGS Co., Assees. of H. A. SCHWARTZ (B.P. 282,671, 16.12.27. U.S., 23.12.26).—See U.S.P. 1,688,438; B., 1928, 931.

Rendering iron pipes immune from attack by water and apparatus therefor. C. BÜCHER (U.S.P. 1,716,205, 4.6.29. Appl., 3.2.27. Ger., 12.2.26).—See B.P. 265,932; B., 1928, 590.

De-rusting of ferrous metals. W. H. COLE (U.S.P. 1,715,695, 4.6.29. Appl., 31.12.27. U.K., 22.8.27).—See B.P. 298,328; B., 1928, 898.

Reduction of metallic oxides. A. J. F. J. COUSIN, Assr. to SOC. ANON. J. COCKERILL (U.S.P. 1,714,767, 28.5.29. Appl., 14.3.23. Belg., 18.5.22).—See B.P. 197,903; B., 1924, 474.

[Flanged] annealing pot. G. MORGAN (B.P. 312,886, 13.10.28).

Refractory materials (B.P. 312,700).—See VIII. **Slag for road-making** (B.P. 312,838).—See IX.

XI.—ELECTROTECHNICS.

Behaviour of solutions of chromium sesquioxide on electrolysis. D. T. EWING, J. O. HARDESTY, and T. H. KAO (Mich. Eng. Exp. Sta. Bull., 1928, No. 19, 3–13).—In experiments with solutions of chromic oxide (250 g. per litre), the percentage of current expended in the deposition of chromium increased, whilst that used in generating hydrogen decreased, with rise of temperature and increase of current density.

CHEMICAL ABSTRACTS.

Frequency and arcing in the alternating-current electrolysis of water. J. W. SHIPLEY (Amer. Electrochem. Soc., May, 1929. Advance copy. 21 pp.).—See A., 1928, 245, 1337.

H. J. T. ELLINGHAM.

Transformer and switch oils. LOCHER.—See II. **Yellow phosphorus.** SHIKHUTSKI.—See VII. **Sponge iron for electric steel.** THOLAND. **Reduction of**

zinc oxide. DONY-HENAULT. **Zinc refining.** SETO. **Measurement of hydrogen-ion concentration in plating baths.** BREWER and MONTILLON.—See X.

See also A., July, 774, **Electrolytic formation of sodium arsenate** (ESSIN). **Electrodeposition of chromium** (ROUDNICK). 775, **Electrolytic copper** (MARIE and JACQUET). **Electrosynthesis of hydrocarbons** (BERMEJO and BLAS).

PATENTS.

Electrostatic condenser. S. RUBEN (U.S.P. 1,714,319, 21.5.29. Appl., 22.8.27).—The dielectric is formed by a suspension of a metal powder in a saturated solution of potassium fluoride, or other electrolyte giving rise to a current-blocking film. The suspension is preferably applied to a porous spacing element.

F. G. CLARKE.

Gaseous conduction apparatus. C. G. SMITH, Assr. to RAYTHEON, INC. (U.S.P. 1,714,407, 21.5.29. Appl., 30.7.23).—By using cathodes, *e.g.*, of tin or potassium-sodium alloy, which are liquid at the operating temperature, the life of the apparatus is prolonged, since the gas pressure, *e.g.*, of helium, remains constant.

F. G. CLARKE.

Piezo-electric substances. P. FREEDMAN and W. J. RICKETS (B.P. 311,055, 29.3.28).—Durable piezo-electric masses of any desired shape or size, consisting of large numbers of oriented hemihedral crystals either in juxtaposition or embedded in a suitable medium, are made from such substances as beeswax, Japan wax, carnauba wax, Chinese insect wax, hydrogenated fats and oils, natural resins, zinc oxide, nickel sulphide, cinchonine sulphate, cupreine sulphate, dicamphahexanedione, dicamphahexadiene peroxide, etc. The crystals may be oriented by crystallisation in an electrical field from the melted solid; this method is applicable to the waxes, which are to some extent conductors when melted and insulators when solid. Zinc oxide or nickel sulphide may be oriented by electrolytic deposition. Alternatively, the substance may be deposited from solution on a medium of m.p. 45–85°. Repeated rolling or other mechanical working orients optically active substances in solid solution in celluloid. The piezo-electric masses produced by these methods may be used as diaphragms in telephonic receivers, or for television purposes.

C. HOLLINS.

Metallic-vapour lamps for producing ultra-violet radiation. P. FREEDMAN (B.P. 313,726, 22.5.28).—A metallic-vapour arc or spark device having two or more electrodes, of which at least one is composed of vaporisable material, *e.g.*, mercury, and is enclosed within a glass envelope transparent to ultra-violet radiation, *e.g.*, of Uviol or Vitaglass, is arranged in an atmosphere containing, in addition to metallic vapours, one or more gases transparent to ultra-violet radiation, *e.g.*, argon, helium, hydrogen, at a pressure of a few mm. to 60 cm. of mercury.

J. S. G. THOMAS.

Metallic-vapour lamps and gaseous-discharge devices. P. FREEDMAN (B.P. 313,292, 23.5.28).—The passage of current from mains at ordinary domestic voltages through the lamp causes a glow discharge which is replaced by an arc discharge owing to liberation of gas or metallic vapour from a suitable source, *e.g.*,

charcoal or palladium-black, arranged within the lamp and heated by the glow discharge. The gas or vapour evolved is re-absorbed on interrupting the current.

J. S. G. THOMAS.

[Gas-filled] electric discharge tubes [containing mercury]. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPFABR. (B.P. 313,184, 30.1.28).—The quantity of mercury in the filling is so small that its condensation on the wall of the tube does not substantially affect the transmission of ultra-violet radiation. [Stat. ref.]

J. S. G. THOMAS.

Carrying-out the oxidation or reduction of electrolytes. SIEMENS & HALSKE A.-G. (B.P. 298,461, 13.3.28. Ger., 8.10.27).—A hollow container having perforated walls and lined with close-mesh wire netting is filled with the oxidising or reducing agent and immersed in the electrolyte.

J. S. G. THOMAS.

Partitions for separating the electrolytic products in the fusion electrolysis of chlorides, particularly of magnesium. I. G. FARBENIND. A.-G. (B.P. 309,408, 27.4.28. Ger., 23.12.27).—Wedge-shaped plates of a dense non-conducting ceramic material, e.g., porcelain, having tongue-and-groove joints are built in a straight arch.

J. S. G. THOMAS.

[Cutting of] piezo-electric crystals. ASSOC. ELECTRICAL INDUSTRIES, LTD., Assees. of D. G. LITTLE (B.P. 289,471, 25.4.28. U.S., 28.4.27).

[Frosted] electric incandescence lamps [for automobiles]. F. J. ALDERSON (B.P. 298,077, 25.9.28. Can., 1.10.27).

Electric discharge tubes. J. ALLEN (B.P. 312,971, 3.3.28.)

[Automatic] device for testing the sensitiveness of human skin, substances, and fabrics to irradiation. J. SAIDMAN (B.P. 313,801, 7.8.28).

Purification of gases etc. (B.P. 292,479).—See I. Lubricating oil (B.P. 294,099). Olefines and diolefines (B.P. 313,756).—See II. Cellulose hydrolysis products (B.P. 313,258).—See XVII.

XII.—FATS; OILS; WAXES.

Determination of saponification value [of fats and oils]—a more stable alcoholic potash reagent. D. T. ENGLIS and V. C. MILLS (J. Assoc. Off. Agric. Chem., 1929, 12, 248—250).—The addition of 5 g. of sodium hyposulphite per litre of the alcoholic potash otherwise prepared according to the official method (cf. *ibid.*, 1925, 288) is effective in preventing coloration of the solution. The mixture is shaken, the solid allowed to settle, and the clear supernatant liquid is used. The values obtained with this reagent agree with those obtained with the official reagent. An attempt to find a general inhibitor of colour development during the saponification was unsuccessful. W. J. BOYD.

See also A., July, 785, Apparatus for extraction with filtration (KULMAN). 789 and 790, Oils of elasmobranch fish (HEILBRON and others). 793, Unsaturated acids of sardine oil (TOYAMA and TSUCHIYA). 809, Isomeric ergosterols (HEILBRON and others). Zymosterol from yeast (PENAU and TANRET;

FABRE and SIMONNET). 818, Congo copal oil (WESTENBERG). 851, Antimony trichloride reaction of fish oils (VON EULER and others). 855, Seeds of *Euphorbia Paralias* (GILLOT). Composition of spinach fat (SPEER and others). Seed fats of the *Umbelliferae* (CHRISTIAN and HILDITCH).

PATENTS.

Saponification of fats and oils. C. BERGELL (B.P. 313,019, 2.2.28).—Complete and rapid saponification is effected by boiling with lye (1—2% of electrolyte to 56% of fatty acids), and in the second stage adding sufficient water to reduce the fatty acid content to 50% and boiling for a short time. E. LEWKOWITSCH.

Purification of [fatty] oils. H. ODEEN (U.S.P. 1,705,824—5, 19.3.29. Appl., 14.7.26).—Liquid (A) and solid (B) fatty oils containing less than 4% of water are refined by treatment below 60°, e.g., at 30—35° for liquid or at 40—55° for solid oils, with 2—10% of a dry reagent consisting of sodium hydroxide intimately incorporated with a finely-divided absorbent material, sufficient water being added separately to promote rapid reaction or "break" but leave the oil substantially anhydrous.

R. BRIGHTMAN.

Manufacture of vulcanised products from [fatty] oils. IMPERIAL CHEM. INDUSTRIES, LTD., and W. J. S. NAUNTON (B.P. 313,252, 5.4.28).—Largely unsaturated fatty oils are emulsified with water or other non-solvent (e.g., glycerin) with the aid of a suitable emulsifier (e.g., ammonium oleate) and a protective colloid, and vulcanised at above 100° (160—170°) by (colloidal) sulphur or other sulphurising agent; suitable organic accelerators (e.g., reaction product of aniline and heptaldehyde) and activators (e.g., zinc oxide) may be added.

E. LEWKOWITSCH.

Manufacture of soap [flakes]. E. FLAMMER and C. KLEBER (U.S.P. 1,715,999 and 1,716,000, 4.6.29. Appl., [A, B] 16.8.26).—See B.P. 265,714; B., 1927, 304.

Production of vitamin preparations. A. W. OWE (U.S.P. 1,715,945, 4.6.29. Appl., 11.3.26. Norw., 11.10.24).—See B.P. 266,905; B., 1927, 339.

Distilling liquids (B.P. 313,254).—See I. Glycerol (B.P. 300,254). Condensation products from fatty acids etc. (B.P. 310,562).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Determination of active oxygen in oxidising Russian turpentine. K. N. KOROTKOV (Mem. Beloruss. State Acad. Agric., 1927, 5, 16—24).—Oxidation is more rapid in sunlight than in artificial light; Russian turpentine has a higher oxidising value than French. The products (not hydrogen peroxide) are thermostable.

CHEMICAL ABSTRACTS.

Composition of oleoresin and colophony from *Pinus sylvestris*. V. SHKATELOV (Mem. Beloruss. State Acad. Agric., 1927, 4, 113—127).—Fresh oleoresin gave a dextrorotatory product, $[\alpha]_D +5.26^\circ$, which afforded four isomerides. CHEMICAL ABSTRACTS.

Peru balsam. ROTHENHEIM.—See XX.

See also A., July, 746, Determination of particle size by X-rays (BRILL).

PATENTS.

Manufacture of fireproofing material for paints. F. S. VIVAS, ASSR. to INTERNAT. FIREPROOF PRODUCTS CORP. (U.S.P. 1,707,587, 2.4.29. Appl., 11.3.27).—Boric acid, zinc sulphate, borax, and sodium tungstate are successively dissolved in water at 55°, and an aqueous solution of gelatin is added; followed by asbestine pulp, titanium oxide, and silica; the paste is mixed with fireproof drying oil, *e.g.*, linseed oil, obtained by dissolving borax, ammonium chloride, boric acid, and sodium tungstate in an equal weight of water and mixing the solution with drying oil at 120°.

R. BRIGHTMAN.

Fireproofing and weather-resisting paint. N. M. HOPKINS, ASSR. to BURNOT FIREPROOFING PRODUCTS, INC. (U.S.P. 1,706,733, 26.3.29. Appl., 1.7.22. Renewed 29.5.25).—Upwards of 50% by wt. of borax is incorporated with pigments in a suitable vehicle; *e.g.*, a mixture of borax, white lead, zinc oxide, asbestine, barytes, lead and zinc borates in a medium of boiled linseed oil, treated tung oil, Japan drier, and varnish is claimed.

R. BRIGHTMAN.

Preparation of dammar resin for use in cellulose ester lacquers. H. E. POTTS. From J. SCHEIBER, and L. BLUMER CHEM. FABR. (B.P. 309,701, 15.3.28).—Certain binary or ternary mixtures of solvents of constant b.p. have been found to dissolve the dammarolic acid and α -resene constituents of dammar resin in a condition suitable for immediate use in cellulose ester varnishes, leaving the β -resene in a substantially unswollen state. Suitable mixtures are: benzene-alcohol (67:64:32:36), b.p. 68.25°; toluene-alcohol (38:62), b.p. 76.5°; benzene-alcohol-water (74:1:18.5:7.4), b.p. 64.85°; and toluene-alcohol-water (49:46:5), b.p. 75.1°.

E. HOLMES.

Artificial shellac and its manufacture. E. C. R. MARKS. From ALOX CHEM. CORP. (B.P. 309,384, 6.10.27).—When a petroleum hydrocarbon mixture in the liquid state, preferably a fuel-oil distillate (*d* 1.38—1.33), is treated with air or oxygen-enriched air at 130—160° and at 150—350 lb./in.², in the presence of about 0.1% of an oxidising catalyst such as a compound of manganese, copper, or iron, petroleum-soluble carboxylic acids followed by petroleum-insoluble hydroxycarboxylic acids are formed. The reaction is continued until the reaction mixture consisting of three layers contains 20—40% of these latter compounds. The top and bottom layers, containing unchanged hydrocarbons and numerous petroleum-soluble materials, and an aqueous solution of acids, respectively, are removed, and the residue is washed, dried, and then distilled at a pressure of 10—20 mm. of mercury and at 140—275°. The higher temperatures give harder and more brittle products, but the less brittle, even "tacky" products have high dielectric properties.

E. HOLMES.

Phenol-acetylene resin. H. W. MATHESON and J. A. NIEUWLAND, ASSRS. to CANADIAN ELECTRO PRODUCTS CO., LTD. (U.S.P. 1,707,940, 2.4.29. Appl., 13.5.22).—Phenol is condensed with acetylene, preferably in presence of a mercury salt or oxide as catalyst, at 50—150°. *E.g.*, dry acetylene is passed into phenol (or cresol) containing 2.4—4% of mercurous sulphate

and 1—2% of sulphuric acid at 90—115°, until approx. 14% of acetylene has been absorbed. Glacial acetic acid may be used as solvent and the temperature raised to 120—150° at the finish. The product may be combined with aldehydic hardening agent.

R. BRIGHTMAN.

Manufacture of coumarone-indene resins. R. L. EMERY, ASSR. to NEVILLE CHEM. CO. (U.S.P. 1,705,857, 19.3.29. Appl., 3.3.28).—Crude solvent naphtha No. 2, or other coal distillate containing at least 80% of indene and coumarone, is polymerised with sulphuric acid, and the product is diluted with light petroleum. The resin solution is decanted, washed from acid, and distilled under high vacuum, using live steam to remove heavy oil if required.

R. BRIGHTMAN.

Lead [paint] coating of articles. O. BRANDENBERGER, ASSR. to ZAHNER & SCHIESS & Co. (U.S.P. 1,717,140, 11.6.29. Appl., 17.8.27. Ger., 30.8.26).—See B.P. 276,643; B., 1928, 936.

Printing ink for producing designs [in colour] on rubber or other like materials. H. N. MORRIS (U.S.P. 1,715,209, 28.5.29. Appl., 8.8.28. U.K., 3.5.27).—See B.P. 296,461; B., 1928, 827.

Treatment of lamp blacks etc. (B.P. 300,610). **Tar products** (B.P. 312,372).—See II. **Esters of organic acids** (U.S.P. 1,706,639).—See III. **Pigments** (B.P. 310,354).—See IV. **Tungsten and molybdenum pigments** (B.P. 313,209).—See VII. **Piezo-electric substances** (B.P. 311,055).—See XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation phenomena in *Hevea* [rubber] latex. VIII. **Influence of some heavy-metal salts on coagulation and coalescence.** O. DE VRIES and N. BEUMÉE-NIEUWLAND (Med. Proefstat. Rubber, No. 40; Arch. Rubbercultuur, 1929, 13, 125—218).—Manganese sulphate and nickel sulphate exhibit normal coagulation phenomena, having no influence on the spontaneous coagulation of latex by bacterial action, distinct coagulating power of their own, and accelerative influence on coagulation by acetic acid; except in the case of the nickel salt at higher concentrations they also fail to inactivate coalse. On the other hand, copper sulphate and mercuric chloride are abnormal in inhibiting spontaneous bacterial coagulation, inactivating coalse and retarding coalescence in acetic acid coagulation. Of all these salts mercuric chloride has the most marked coalescing power in itself.

D. F. TWISS.

Thermal changes accompanying the spontaneous contraction of raw rubber. H. SACHSE (Gummi-Ztg., 1929, 43, 2216—2218).—Making use of a modified "ice calorimeter" employing diphenyl ether instead of water, measurement is made of the heat of swelling (with toluene) of samples of rubber of different age or pretreatment; from a comparison of the results with these in their natural state and after moderate heating it is possible to assess the heat change associated with the change in density which occurs spontaneously in storage. The greatest value, 6.4 g.-cal./g., was obtained with a sample of unworked crêpe rubber 10 years old.

D. F. TWISS.

PATENTS.

Production of synthetic rubber. E. KLEIBER and P. GILARDI (B.P. 312,741, 26.3.28).—Petroleum is treated with an oxidising agent, *e.g.*, nitric acid, and, after neutralisation, is distilled repeatedly over rosin. After treatment of the distillate with alkali and removal of the aqueous lowermost layer the liquid can be converted by acetic acid or formaldehyde, together with rubber or the like as catalyst, into a syrupy liquid from which a vulcanisable elastic mass is obtainable by precipitation with acetone or alcohol. D. F. TWISS.

Manufacture of synthetic rubber. I. G. FARBENIND. A.-G. (B.P. 286,272 and Addn. B.P. 300,167, [A, B] 1.3.28. Ger., [A] 2.3.27, [B] 7.11.27).—Isoprene, butadiene, dimethylbutadiene, or analogues or mixtures of these are polymerised in the presence of an aqueous suspension or a colloidal solution or an aqueous dispersion medium, containing, *e.g.*, soaps, albumins, or carbohydrates, in the presence of suitable electrolytes (excluding alkalis) and, if desired, in the additional presence of oxygen, ozone, or a substance yielding oxygen, or of other accelerators of polymerisation, or of an inert gas. D. F. TWISS.

Manufacture of artificial rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,949, 2.1.28).—Diolefines are polymerised in the form of creamy or gelatinous mixtures with aqueous solutions, these mixtures containing at least 75% by vol. of the hydrocarbons. Additional substances may also be present, *e.g.*, polymerising agents such as oxidising agents, buffer mixtures, and materials capable of modifying the surface tension of emulsions. D. F. TWISS.

Manufacture of polymerisation products of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,188, 9.2. and 6.7.28).—The polymerisation of diolefines in the presence of latex or similar vegetable saps is effected in the presence of oxidising or emulsifying agents. D. F. TWISS.

Manufacture of an elastic caoutchouc-like body. J. BAER (B.P. 298,889, 1.6.28. Switz., 15.10.27).—An elastic mass capable of being milled, compounded, moulded, and vulcanised like rubber is obtained by submitting an aldehyde, preferably formaldehyde, or a mixture with halogen-substitution products of paraffin hydrocarbons, to treatment with solutions of polysulphides of the alkali or alkaline-earth metals. D. F. TWISS.

Manufacture, from latex, of an artificial gutta-percha and a non-hygroscopic rubber. E. S. ALI-COHEN (B.P. 313,373, 5.9.28).—By mixing an emulsion of resins or waxes (> 100 pts.) in a soap solution with latex (rubber 100 pts.) and coagulating with alum so as to get more than 15 pts. of aluminium soap in the product, a material resembling gutta-percha is obtained. With other proportions the product is less satisfactory as a substitute for gutta-percha, but is still water-repellent and possesses outstanding technical and electrical properties. D. F. TWISS.

Incorporation of fillers and other compounding ingredients, and of gas black, into rubber latex or the like. DUNLOP RUBBER CO., LTD., W. H. CHAPMAN,

and P. D. PATTERSON (B.P. 313,027—8, 5.3.28).—(A) Compounding ingredients such as softeners, vulcanising agents and accelerators, reinforcing agents, etc. are incorporated in a water-insoluble plastic carrier such as rubber, reclaimed rubber, bitumen, or clay, or a mixture of these, the resulting mass then being dispersed in an aqueous medium; this dispersion is then mixed with latex. (B) Gas black is introduced into latex as in (A). D. F. TWISS.

Heat-plastic derivative of rubber. W. C. GEER and H. A. WINKLEMAN, Assrs. to B. F. GOODRICH CO. (U.S.P. 1,705,757, 19.3.29. Appl., 28.4.25).—Crude rubber is mixed with 3—5% of a softener, *e.g.*, pine tar, turpentine, coal-tar distillates, phenol, naphthalene, petroleum distillates, vegetable oils and waxes, etc., the isomerising agent, *e.g.*, sulphuric acid, is added, and the mix is heated and finally masticated. Alternatively, organic sulphonic acids or sulphonyl chlorides may be used in place of the sulphuric acid and part of the softener. R. BRIGHTMAN.

Thickening and treatment of latex. D. S. SUTTON, and VEEDIP, LTD. (B.P. 312,947, 2.1.28).—By adding adjusted proportions of zinc oxide to latex, *e.g.*, 1% to latex containing 0.75—1% of ammonium hydroxide, the viscosity of latex can be increased. It is convenient first to form a paste of the zinc oxide with ammonia solution and to mix this intimately with a portion of the latex before introducing it into the bulk. The thickening is assisted by heating to 95° and stirring. If the proportion of ammonia in the latex is undesirably high it can be previously reduced by heating or neutralisation with dilute acids. D. F. TWISS.

Vulcanisation of rubber. M. L. WEISS, Assr. to DOVAN CHEM. CORP. (U.S.P. 1,705,948, 19.3.29. Appl., 25.7.25).—*s*-Diarylguanidines, in which at least one of the aryl groups is disubstituted, are used as accelerators. The compounds claimed include the *s*-di-*m*-xylyl-, *s*-di- α -naphthyl-, *s*-phenyl-*m*-xylyl-, *s*-phenyleumyl-, *s*-dicumyl-, and *s*-phenyl- α -naphthyl-guanidines. R. BRIGHTMAN.

Treatment [vulcanisation] of rubber. E. G. BRIDGWATER and A. S. SLACK, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,705,712, 19.3.29. Appl., 3.7.26).—The products obtained by condensing 2 mols. of a primary aromatic amine with 1 mol. of an aliphatic aldehyde and treating the resultant methylenedi-imine with a higher fatty acid are incorporated in rubber mixes for vulcanisation. *E.g.*, methylenediphenylimine, $\text{CH}_2(\text{NHPh})_2$, from aniline and formalin at 70—80°, is mixed at 70—80° with molten stearic acid, and, after cooling, the mixture is incorporated with rubber stock. R. BRIGHTMAN.

Manufacture of vulcanised rubber. GOODYEAR TIRE & RUBBER CO., Asses. of J. TEPPEMA (B.P. 298,622, 5.6.28. U.S., 13.10.27).—Vulcanisation is accelerated by a condensation product of an amine, *e.g.*, aniline, and a hydroxyaromatic aldehyde such as β -hydroxy-naphthaldehyde. D. F. TWISS.

[Rubber] tube lining. J. SCHWAB, JUN. (U.S.P. 1,715,213, 28.5.29. Appl., 4.1.25. Renewed 6.10.28).—A vulcanising rubber solution (30 pts.) is mixed with melted vulcanised rubber (30) and then with honey

(40), the mixture then being heated to approx. 127° to produce a self-healing composition for inflatable rubber tubes.

D. F. TWISS.

Manufacture of rubber articles. U. PESTALOZZA, Assr. to SOC. ITAL. PIRELLI (U.S.P. 1,717,248, 11.6.29. Appl., 8.7.27. It., 31.1.27).—See B.P. 284,608; B., 1929, 612.

Manufacture of sponge rubber. R. J. NOAR (U.S.P. 1,717,168, 11.6.29. Appl., 1.6.28. U.K., 19.7.27).—See B.P. 284,938; B., 1928, 277.

Treatment of lampblacks etc. (B.P. 300,610).—See II. **Vulcanised products from oils** (B.P. 313,252). See XII.

XV.—LEATHER; GLUE.

Acidity control of chrome[-tanned] leather. J. S. MUDD and P. L. PEBODY (J. Soc. Leather Trades' Chem., 1929, 13, 205—214).—For the determination of acid sulphates in the aqueous extract, 15 g. of the fat-free leather are soaked overnight in 150 c.c. of distilled water, boiled under a reflux for 45 min., cooled to 18°, filtered, and after determining its p_H value, an aliquot portion of the filtrate is titrated with 0.1N-sodium hydroxide, using phenolphthalein as indicator. The content of acid sulphates is calculated from the value obtained by titration. In determining the ionogenic sulphate groups (cf. B., 1927, 261), 3 g. of the original leather are extracted with 50 c.c. of a 4% solution of pyridine for 1 hr. in a shaking machine, the used solution is filtered, and the extraction repeated. After filtering, the leather is washed with water, the combined extracts and washings are concentrated, acidified with hydrochloric acid, and the sulphate is determined gravimetrically. Tests on different experimental chrome-tanned leathers have shown that the p_H value of the aqueous extract varies inversely with the ionogenic sulphate groups and increases with increase in the basicity of the chrome tanning liquors used. It is also shown that as the amount of neutralising agent was increased, the ionogenic sulphate groups were diminished in inverse proportion, but the "complex-bound" sulphate groups decreased only slightly. The quantity of fat entering the leather during fat-liquoring diminished rapidly at first (up to 2% of borax) and then increased slightly. Sodium hydroxide, sodium carbonate, and borax had similar neutralising powers, but sodium phosphate had very little. The sodium hydroxide did not attack the complex-bound sulphate radicals at the concentration used.

D. WOODROFFE.

Differentiation of skin- and bone-glue with the aid of sulphosalicylic acid precipitation, and the applicability of the sulphosalicylic acid test in the testing of gluten preparations. O. GERNGROSS and H. MAIER-BODE (Kolloid-Z., 1929, 48, 184—191).—Experiments on 17 different kinds of glue show that precipitation with sulphosalicylic acid provides a simple and safe method of distinguishing between skin- and bone-glue, for whilst the former gives a viscous liquid precipitate of relatively small volume, bone-glue gives a flocculent precipitate of large volume. The test can further be used to assess the value of a mixed glue, according to the volume of precipitate obtained. The

different effects obtained with skin- and bone-glue are probably not due to differences in the chemical substance of the original material, but are caused by the different kinds and quantities of degradation products of gluten produced under the different conditions of manufacture. Other precipitation reagents, such as thiocyanate and sulphuric acid, acid sulphite-cellulose liquor, and neutral sodium sulphate, also distinguish between these glues, but their use is not so advantageous as that of sulphosalicylic acid.

E. S. HEDGES.

See also A., July, 764, **Theory of vegetable tanning** (DE JONG).

PATENTS.

[Autoclave] treatment of hides in production of leather. C. W. NANCE (B.P. 310,823, 1.12.27).—Hides are suspended in an autoclave provided with a ram to compress any liquid and with internal tubes extending from below a false bottom to a spray pipe in the upper part of the autoclave for the circulation of the liquors with the aid of heat. After pretreatment, which may or may not be carried out in the autoclave, the hides are subjected to a high vacuum, and while still in the autoclave are treated with a tan liquor from which all the air has been removed, and then, while still in contact with the liquor, are subjected to high pressure. A subsidiary autoclave may be provided above the main one to contain a supply of tan liquor under vacuum for the latter. A chamber having a baffle plate is fitted between the main autoclave and the vacuum apparatus to act as a foam trap.

D. WOODROFFE.

Manufacture of glutinous products [adhesives]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,101, 5.1.28).—Yeast or the product of yeast autolysis is heated for less than 1 hr. with water, under pressure if desired, or for less than $\frac{1}{2}$ hr. with aqueous solutions of weak alkalis, e.g., ammonia, alkaline-earth hydroxides, of weak acids, e.g., carboxylic acids, phosphorus acids, hydrogen sulphide, or of salts of these acids and bases. The solutions obtained are filtered and concentrated, the products being used alone or in admixture with other adhesives.

L. A. COLES.

Production of leather, [gas-tanned] leather, and chrome-tanned leather for dyeing. R. H. PICKARD, D. J. LLOYD, and A. E. CAUNCE (U.S.P. 1,715,621—3, 4.6.29. Appl., [A—C] 22.8.25. U.K., [A—C] 27.8.24).—See B.P. 243,089—243,091; B., 1926, 101.

Production of washable printings on leather. C. S. FRIEDMAN, Assr. to ANC. ETABL. A. COMBE & FILS & CIE. (U.S.P. 1,716,120, 4.6.29. Appl., 13.4.23. Fr., 30.11.22).—See B.P. 207,778; B., 1924, 268.

Improving animal materials (B.P. 294,890).—See VI.

XVI.—AGRICULTURE.

Composition of some Montenegrin soils. E. BLANCK and H. KEESE (Chem. Erde, 1929, 4, 157—166).—Chemical and mechanical analyses of a number of surface soils from Montenegro show that these with only a few exceptions belong to the usual Mediterranean red-earth type, characterised by low silica and relatively high alumina and ferric oxide.

H. F. HARWOOD.

Do soil samples become acid on drying? B. AARNIO (Z. Pflanz. Düng., 1929, 8B, 37—39).—A reply to Brenner (cf. B., 1927, 309; 1928, 618, and following abstract). The reaction of certain Finland soils alters considerably on drying, particularly those containing sulphides from the region examined by Brenner. The necessity for using fresh soil samples for examination is emphasised. A. G. POLLARD.

Alleged acidification of soil samples on drying. W. BRENNER (Z. Pflanz. Düng., 1929, 8B, 39—42).—The notable changes in the reaction of soils during drying recorded by Aarnio (B., 1929, 569) are ascribed to the use of an enclosed drying chamber, in which, and in the laboratory containing it, substantial amounts of sulphur dioxide were present. Experimental data are given and the relatively insignificant changes of reaction resulting from drying are confirmed. A. G. POLLARD.

Chemical analysis of soils and the molecular ratios. K. URESCHER (Z. Pflanz. Düng., 1929, 13A, 265—274).—Methods of examination previously described (B., 1928, 582) are extended to some Swedish and Dutch soils. In these soils the methods of the Prussian Geological Institute yield similar results to those of the International Society, in which the sample is treated with ten times its weight of 20% hydrochloric acid. The former process is simpler, but may be affected by the presence of acid-soluble minerals which are but slightly weathered. Hot concentrated hydrochloric acid decomposes the weathered silicate complexes, but weaker acids cannot be utilised successfully for this purpose. A. G. POLLARD.

Use of dialysis and electro-ultrafiltration for the determination of the nutrient requirement of soils. P. KÖRTGEN and R. DIEHL (Z. Pflanz. Düng., 1929, 8B, 65—105).—The principles of electrodialysis are applied to a study of the rate of dissolution of soil constituents concerned in plant nutrition. The apparatus of Gutbier and Schieber, of Golodetz, and, more particularly, a modification of the Bechhold electro-ultrafiltration apparatus are utilised for the purpose. Buffer curves are prepared from measurements of the rate of dialysis of hydrogen ions from the soil complex, and similar measurements for phosphates are indicative of the soil's capacity to maintain a supply of soluble phosphate for plant growth. The immediate need of soils for the other, more soluble plant nutrients may rapidly be determined by this means. Fertiliser requirements so calculated were more closely in accord with the results of field trials than values obtained by the customary chemical methods or by Neubauer's seedling method. The methods and apparatus described are suitable for the examination of both mineral and forest soils, and in the latter may be utilised to examine the state of humification of the organic matter. A. G. POLLARD.

Methods for the determination of the nutrient requirement of soils for phosphate and potash. E. BECKER (Z. Pflanz. Düng., 1929, 13A, 274—294).—There is a general agreement between the results obtained by the methods of Sigmond, of Lemmermann, and of Neubauer, but certain discrepancies are observed and

examined. The percentage solubility and the "relative solubility" by Sigmond's and Lemmermann's methods agree well in acid soils, but diverge as the chalk content of the soil increases. Citric acid dissolves much less soil phosphate than nitric acid even when the quantities used are calculated on a basicity basis. Sigmond's values give the better representation of the phosphate actually utilisable by plants. Neubauer's method indicates that acid soils contain more soluble phosphate than alkaline ones, whereas in Sigmond's process the reverse is indicated. The Sigmond values are considered the more satisfactory. The secondary effect of the soil reaction on the ability of the plant to utilise nutrients, even when present in a normally assimilable form, must be considered in these cases. The presence of much carbonate in soil reduces the plant's power of utilising phosphate, although such conditions are coincident with apparently high solubility. The exchangeable potash in soil (Hissink) is so closely related to the assimilable potash as to afford a satisfactory means of calculating this latter value. The methods of Sigmond and of Neubauer yield generally parallel results for assimilable potash. No relationship exists between the amounts of assimilable potash and the reaction or the "lime condition" of soils. A. G. POLLARD.

Utilisation of increasing applications of nitrogen by various species of winter wheat. J. WEIGERT and F. FÜRST (Z. Pflanz. Düng., 1929, 8B, 265—303).—Numerous field trials extending over 7 years with a number of varieties of winter wheat are recorded. The effect of nitrogen manuring is most apparent in the period from the middle to the end of April. The effects on the characteristic growth of the different varieties is recorded. The variations in crop yields following fertiliser treatment were generally similar to those obtained with rye (B., 1929, 447), but the relative yields were increased to a much greater extent in the case of wheat. The weight per 1000 grains of wheat increased with the smaller dressings of nitrogen (25—40 kg./hectare), but with larger applications there was a steady decline. A. G. POLLARD.

Effect of one element of plant food on the absorption by plants of another element. J. F. BREAZEALE (Ariz. Agric. Exp. Sta. Tech. Bull., 1928, 19, 465—480).—With wheat, nitrate stimulates the absorption of potassium on account of the demand for the building-up process. Absorption of nitrogen is not affected by the presence of potassium, phosphorus, or calcium sulphate, nor that of calcium by nitrogen, potassium, or phosphorus. The absorption of phosphorus is slightly increased by the presence of nitrogen, and diminished by an excess of calcium sulphate.

CHEMICAL ABSTRACTS.

See also A., July, 854, **Influence of complete fertiliser on cultivated plants** (BLANCHARD and CHAUSSIN). 855, **Effect of boron deficiency on growth of tobacco plants** (McMURTREY).

PATENTS.

Colouring foliage (U.S.P. 1,714,838).—See VI. **Colloidal sulphur** (U.S.P. 1,705,862).—See VII. **Seed grain immunising media** (B.P. 287,093).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Pan-boiling control [of cane syrups]. S. J. SAINT (Tropical Agric., 1928, 5, 279—283).—Pan-boiling control according to Claassen's methods was instituted in a West Indian factory, the concentration of the mother-liquors being determined by the Zeiss pan refractometer. The optimum coefficient of supersaturation was found to be 1.15 for first strikes, and 1.2 for seconds and thirds. Tables were constructed for the three strikes, from which, given the level of the massecuite in the pan and the vacuum, the Brix could be read, which figure was compared by the panman with that found from the refractometer. If the concentration as shown by the refractometer was too high, then the inlet valve was opened more, whilst if the concentration was too low the valve was closed until the correct concentration was registered.

J. P. OGILVIE.

Prevention of sugar losses during milling using chlorine. J. N. SARKAR (Rev. Agric., Mauritius, 1928, 258—263).—Disinfection by chlorine has been found of great practical benefit in lessening losses during extraction and manufacture (cf. Haldane, B., 1927, 920). A wash-down of the crusher, mills, gutters, channels, mill-beds, and strainers with a solution of 1 pt. of chlorine in 500 pts. of water before starting operations and every time after stopping work is recommended. Similarly, measuring tanks, juice-heaters, and defecators should be washed with the same solution at least once a day.

J. P. OGILVIE.

Clarifying diffusion juice with magnesium sulphate. V. I. KUZNETZOV (Nauch. Zapiski, 1928, 7, 158—163).—Magnesium sulphate may replace carbon dioxide; magnesium hydroxide and calcium sulphate are precipitated, the latter being eliminated at the b.p. The amount of lime required is small.

CHEMICAL ABSTRACTS.

New method of defecating cane juices. G. H. DE VRIES (Archief, 1928, 36, 263—267; Internat. Sugar J., 1929, 31, 320).—Instead of adding milk of lime to the juice, the procedure in which this order is reversed is considered advantageous, laboratory tests having shown that the clarification was improved, there being a greater volume of precipitate, pointing to a more thorough elimination of non-sugars. Moreover, the juice finally obtained was both lighter in colour and clearer.

J. P. OGILVIE.

Determination of the end-point in carbonatation [of beet juice]. O. SPENGLER and C. BRENDL (Z. Ver. deut. Zuckerind., 1929, 79, 39—43).—In order to determine the point at which in the carbonatation of beet juice all the alkalis are converted into carbonates (the optimum alkalinity for the precipitation of calcium salts), Winkler's method of titrating the alkalinity before and after the addition of barium chloride cannot be used, owing to the reaction being incomplete, but the following process is recommended:—To the still hot liquid are added 5 drops each of phenolphthalein solution (1:100) and of a 15% solution of calcium chloride, and, after shaking, the colour of the solution is observed after 15—30 sec. If this is a dull pink, the carbonatation has reached the right stage; if colourless, less carbon

dioxide is required; whilst if a distinct pink or red colour is observed, further carbonatation is necessary.

J. P. OGILVIE.

Sugar content of carbonatation scums. O. SPENGLER and C. BRENDL (Z. Ver. deut. Zuckerind., 1929, 79, 61—76).—Filter-press cake was triturated with hot water for 1 hr., filtered and washed, and the sugar determined in the total liquid obtained, when the amount was found to be considerably more (about 150%) than in the original press-cake when examined by the usual way, e.g., using the ammonium nitrate method (cf. Kunz, B., 1925, 255; Orth, B., 1927, 711), the discrepancy being now explained by the presence in the cake of colloidal matter which had the effect of retaining a large proportion of sugar unless digested with an excess of hot water.

J. P. OGILVIE.

Occurrence of zinc in products of sugar manufacture. O. SPENGLER and K. ZABLINSKY (Z. Ver. deut. Zuckerind., 1929, 79, 251—262).—Sugars of various kinds from German refineries contained 0.6—3.3 mg. of zinc per kg. Molasses from raw beet-sugar factories, white-sugar factories, and refineries contained 49, 15, and 18 mg./kg., respectively. The zinc is derived mainly or entirely from the original beets, which contain about 2—3 mg./kg. Prohibition of the use of zinc-coated vessels in the manufacture of foodstuffs, as has been suggested [in Germany], would therefore not result in zinc-free sugar, and it would greatly embarrass the refineries. Experiments described show that a zinc coating forms a practically perfect protection for iron against attack by sugar solutions, whilst tinned iron corrodes very readily and colours the sugar solutions. Zinc in traces is a normal constituent of foodstuffs, and the proportions found in refined sugar are much less than those found in grain products and eggs, and no greater than those naturally present in milk.

J. H. LANE.

Determination of hydrogen-ion concentration; application in sugar manufacture. I. A. KUKHARENKO and B. G. SAVINOV (Monograph, 1928, 11—147).—A review.

CHEMICAL ABSTRACTS.

Limit of sugar extraction from cosettes in the diffusion process. S. S. KUTZEV (Bull. Sakharotresta, 1928, No. 9, 65—68).

Quality and quantity of [sugar liquor] wash-water from bone-black filters. M. I. NAKHMANOVICH and J. F. ZELIKMAN (Nauch. Zapiski, 1928, 7, 201—222).

Waste waters from beet-sugar factories. BACH.—See XXIII.

See also A., July, 772, Concentration of sugar and rate of hydrolysis in an acid medium (COLIN and CHAUDUN). 796, Action of hydrogen sulphite solutions on sugars (MARUSAWA and others). 797, Compounds of alkaline-earth hydroxides with sugars. (MACKENZIE and QUIN).

PATENTS.

Treatment of sugar solutions to increase the sucrose content. W. H. DICKERSON, Assr. to INDUSTR. WASTE PRODUCTS CORP. (U.S.P. 1,713,118, 14.5.29.

Appl., 29.1.23. Renewed 2.10.28).—The sugar solution is sprayed into a drying gas heated to a temperature above the b.p. of the solution, and the desiccated product, in which the ratio of sucrose to invert sugar is greater than that of the original sugar solution, is immediately cooled to prevent decomposition.

F. R. ENNOS.

Clarification of cellulose hydrolysis products.

INTERNAT. SUGAR & ALCOHOL Co., LTD. From Soc. INDUSTR. DE LA CELLULOSE (B.P. 313,258, 14.4.28).—After conversion of cellulose into sugar by means of hydrochloric acid, the whole is clarified by electrolysis at a low potential of 2–3 volts in a diaphragm cell. The sugar solution flows continuously into the anode chamber, whilst the cathode space contains hydrochloric acid, or sugar and hydrochloric acid, at a concentration corresponding to that of the product under treatment.

F. R. ENNOS.

Manufacture of extra-fine soft granulated sugar.

B. H. VARNAU and T. B. WAYNE (U.S.P. 1,715,049, 28.5.29. Appl., 4.9.26).—See B.P. 294,800; B., 1928, 832.

Adsorbing agents (U.S.P. 1,715,439).—See VII.

XVIII.—FERMENTATION INDUSTRIES.

Technological method for the study of yeast.

E. E. WERNER and W. SIEDHOFF (Cereal Chem., 1929, 6, 196–201).—The quality of the yeast is judged from a modified baking test, carried out on the Werner system, in which two loaves are baked, one with excess of sugar, and one without sugar. The interpretation of the results is discussed.

E. B. HUGHES.

Determination of the reliability and fermentative powers of wine yeasts. E. ARAUNER (Pharm. Ztg., 1929, 74, 713).—Two 150 c.c. samples of sweetened must are sterilised in boiling water and cooled. One is then treated with the ferment under examination and the other with a standard culture, and the specimens are then placed side by side in a warm place at 20–25°. Satisfactory yeasts ferment in 5–6 days and the fermentation is finished in 8–10 days. If fermentation is not evident in 8 days, moulds may appear on the surface and acetic acid may be detected by its smell. The fermented liquors must possess a pleasant odour.

S. COFFEY.

Brewing barley. Y. NAKAMURA (J. Fac. Agric. Hokkaido, 1928, 23, [2], 29–86).—Determinations of protein, nitrogen, ash, and mol. wt. of Chevalier, Hokudai No. 1, and Golden Melon barleys are recorded, and the fats and starches were examined.

CHEMICAL ABSTRACTS.

Direct production of absolute alcohol from the fermented mash. E. SZILAGYI (Z. Spiritusind., 1929, 52, 199).—The apparatus designed by Guinot has been improved, and it is now possible to obtain pure absolute alcohol directly from a mash containing 6–7% of alcohol with a consumption of 350 kg. of steam per hectolitre of alcohol. The improvement consists in the method of connecting the distillation column to the dehydrating column so that the latter may be completely heated by the highly concentrated alcoholic vapour from the former. In the ordinary form of plant the temperature of the alcoholic vapour is 78°,

and is sufficiently high to ensure the partial heating of the dehydrating column, which varies in temperature in the azeotropic method from 60° to 81° according to the nature of the liquid added for the withdrawal of the water. By moderately increasing the pressure of the vapour by means of a column of water 5 m. high, the temperature of the alcoholic vapour is raised to 89°, which is sufficiently high to allow complete heating of the dehydrating column.

C. RANKEN.

Determination of the volatile acidity of wines.

D. COSTA [with C. CANNELLA and F. TROST] (Annali Chim. Appl., 1929, 19, 189–213).—The various methods and forms of apparatus suggested for this determination are discussed, and experiments are described which show that, from 50 c.c. of wine containing up to 0.5% of volatile acidity, all the acetic acidity passes over in 300 c.c. of distillate. The recommendation that, even with wines containing 0.2% of volatile acid, 25 c.c. diluted to 50 c.c. should be distilled, is hence unnecessary. To obtain rapid elimination of the acetic acid it is advisable to distil 50 c.c. directly to about 25 c.c. before the current of steam is started. The whole operation should be completed in 30–40 min., and during the distillation the intensity of the heating should be maintained as nearly constant as possible and steam pressure should be the minimum necessary.

T. H. POPE.

See also A., July, 347, **Fermentative formation of allantoic acid from allantoin** (FOSSE and BRUNEL). **Influence of formaldehyde on dilute malt extracts** (MÜLLNER). **Activation of oxidase preparations** (PUGH). **Influence of p_H on glycerophosphatase** (INOUE). **Cataphoresis of glycerophosphatase** (KOBAYASHI). 849, **Mechanism of alcoholic fermentation** (KLUYVER). **Yeast stable towards boiling** (RENNING). **Co-enzyme** (VON EULER and MYRBÄCK). **Growth and fermentative power of wine yeasts** (KROEMER and KRUMBHOLZ; KRUMBHOLZ and SOÓS). **Accelerating action of lanthanum salts on fermentation** (RICHTER and BRAUMANN).

PATENTS.

Preparation of yeast compositions. B. M. DAWSON, ASSR. to FLEISCHMANN Co. (U.S.P. 1,706,564–6, 26.3.29. Appl., 30.6.22).—Mixtures, preferably freshly prepared, of yeast with (A) milk and possibly malt extract, (B) fruit juice and possibly milk, and (C) malt extract and possibly fruit juice, are atomised into a hot, but not lethal, gas, whereby the yeast is dried without being killed. The compositions are stable since the dry particles of live yeast are coated with the other dried ingredients.

F. G. CLARKE.

Glycerol (B.P. 300,254).—See III.

XIX.—FOODS.

Manganese in cereals and cereal mill products.

J. DAVIDSON (Cereal Chem., 1929, 6, 128–133).—No correlation was found between manganese and ash contents or between manganese content and diastatic properties of the cereals studied. Hard winter and red spring wheats were higher in protein and lower in manganese than soft wheats. Large variations in

manganese content of oats and maize were probably due to effect of locality. The removal of the germ and outer parts of the grain in milling results in low content of ash and manganese. Both uncoated rice and maize, which are associated with beri-beri and pellagra, respectively, are extremely poor in manganese. W. J. BOYD.

Phosphorus of grains. J. E. GREAVES and C. T. HIRST (Cereal Chem., 1929, 6, 115—120).—The total phosphorus of wheat, oats, and barley increases as the irrigation water applied during the growing stage increases, the increase being mainly organic phosphorus. It also varies with the soil on which the grain is grown. In maize the increase on increased irrigation is slight and mainly due to inorganic phosphorus. In wheat about 6.3% of the total phosphorus is inorganic, in oats 9.8%, in barley 9.6%, and in maize 12.2—16%. The addition of barnyard manure to the soil increases the proportion of inorganic phosphorus in the maize kernel.

W. J. BOYD.

Carotinoid pigments of flour. C. G. FERRARI and C. H. BAILEY (Cereal Chem., 1929, 6, 218—240).—It is considered that for the colorimetric examination of flour such methods as those using the Lovibond tintometer or comparing the colour of petroleum spirit extracts of the flour with that of standard colour preparations are not sufficiently accurate. Careful measurements have been made by means of a spectrophotometer, the instrument used being the Keuffel and Esser colour analyser (cf. A., 1925, ii, 1193). By measuring the transmittancy of petroleum spirit solutions of carotin for a wave-length $\lambda = 435.8 \text{ m}\mu$, the concentration of carotin can be calculated from the expression $bck = -\log_{10} T$, where b is the thickness in c.c. of the layer of solution, c is the concentration of substance (carotin) in mg. per litre, k the specific transmissive index, and T is the transmission (%). A graph is given for this expression calculated from the data obtained by Schertz (cf. B., 1924, 288), and from which the carotin content of a solution in petroleum spirit can be obtained in mg. per litre, and thence calculated back to the flour used. The absorption spectra of carotin in petroleum spirit and petroleum spirit extracts of flour have been compared, and results confirm the work of Monier-Williams (B., 1912, 1144) that these are practically identical. It is also shown that the presence of a small quantity of another pigment like xanthophyll would not cause any appreciable error in the method described for the colour measurement of flour. Details of procedure for the determination are to be given in a later paper. E. B. HUGHES.

Wheat and flour studies. XV. Use of the viscosimetric method for measuring the proteo-clastic activity of flours. A. H. JOHNSON, B. L. HERRINGTON, and S. G. SCOTT (Cereal Chem., 1929, 6, 182—196).—After auto-digestion of the flour (12 or 18 g. in 100 c.c. of water with toluene) it is made up to 1 litre. It is thrice washed, once at 40° and twice at 25°, with 1 litre of water rendered free from carbon dioxide, decanting between each washing. The residue is made up to 100 c.c., acidulated with 0.5 c.c. of 20% lactic acid, and its viscosity determined by the MacMichael viscosimeter. The changes in viscosity produced by auto-digestion are appreciable, the decrease

being rapid in the early stages. The change in viscosity is much greater than the degree of change in the amino-nitrogen. Low-grade flours give the greatest changes in viscosity.

E. B. HUGHES.

Modifications of Rumsey's method for determination of diastatic activity in flour. J. G. MALLOCH (Cereal Chem., 1929, 6, 175—181).—The following modifications of Rumsey's method (Amer. Inst. Baking, Bull. No. 8, 1922) are proposed: (a) Control of acidity, using a buffer solution composed of 2 pts. of 0.1N-hydrochloric acid, 8 pts. of a solution of 21.008 g. of crystallised citric acid and 200 c.c. of N-caustic soda in 1 litre, in order to produce the optimum p_H of 4.7. The buffer reagents have no other effect on the diastatic activity. (b) Correction for volume of flour. The correction for 10 g. of flour is taken as 7 c.c. (c) The procedure for determination of the blank is altered by using acid in this as in the determination itself, allowing the reaction to proceed for 60 min. at 27° and then determining reducing sugars immediately. A method is also given for obtaining diastase-free flour.

E. B. HUGHES.

Hydrogen-ion determination in flour and bakery products. E. O. WHITTIER and E. GREWE (Cereal Chem., 1929, 6, 153—162).—Watson's method (B., 1928, 33) for the determination of the p_H of cheese has been applied to flour and bakery products. The flour or crumb is mixed with sufficient quinhydrone to give the mix a grey-black colour, water being added to form a stiff dough if the crumb itself will not cohere. Portions of this mixture are pressed on to the gold electrode and then moulded into "cocoon" about 2.5 cm. long and 1 cm. diam., so that the electrode extends about 2 cm. into the cocoon, and after 1 hr. the p_H is measured. The water added to aid coherence does not affect results, identical values having been obtained for dry and moistened crumb. This "ball" method gave results with various bakery products differing from those given by aqueous extracts of the material, both by the Bailey hydrogen electrode and the capillary quinhydrone electrode, though relative values for the different materials were of the same order whichever electrode was used. It is concluded that results obtained by the "ball" quinhydrone method are more truly representative of actual hydrogen-ion concentration. E. B. HUGHES.

Determination of degree of acidity of flours by colorimetric spot-test. R. HERTWIG and J. S. HICKS (Cereal Chem., 1929, 6, 162—163).—The progress of the "maturing" of flours with "Beta Chlora" is followed by means of the coloration produced by drops of dye reagents of various p_H ranges when placed on the surface of samples of standard and test flours placed in depressions of porcelain test plates. F. G. CROSSE.

Hygroscopicity in flour ash and a discussion of direct ash weighing. L. U. LIDDEL (Cereal Chem., 1929, 6, 134—139).—The hygroscopicity of flour ash affects the results of ash determinations very little, and direct weighing of the ash, dumped on the balance pan, gives more uniform results than weighing the ash in the crucible.

W. J. BOYD.

[Laboratory] gluten-washing machine. T. R. JAMES (Cereal Chem., 1929, 6, 244—246).—A machine

for analytical washing of flour gluters takes a 10-g. sample of flour, which is made up into a dough and allowed to remain under water as usual. It is then placed in the machine, which imitates the action of squeezing a dough ball by hand in a flow of water. E. B. HUGHES.

Single-figure estimates of baking scores. R. K. LARMOUR (*Cereal Chem.*, 1929, **6**, 164—174).—The numerical score values to be given to the various characteristics of a loaf (such as loaf volume, texture, colour, etc.) in order to arrive at a total from which the relative value of the flour for bread baking purposes can be judged are discussed. E. B. HUGHES.

Serodensimetric constant (C.S.D.) for detecting the watering of milk. F. OLIVARI (*Annali Chim. Appl.*, 1929, **19**, 214—233).—Consequent on the lactose-chloride equilibrium established by Porcher (*Rev. gén. du Lait*, 1906), the density Q (excess over 1000) and the parts per 1000 of chlorides (as sodium chloride) C in milk serum are connected by a relationship expressible by the formula: $Q + 3.85C = K$. This constant sum, known as the serodensimetric constant, is more useful than the mere density in the detection of watering as it exhibits for smaller variation and a better statistical distribution about the typical value for milks of any origin. For acetic serum the mean value of K is 35.1. This constant is more simply and more rapidly determined than the simplified molecular constant of Mathieu and Ferré (*B.*, 1914, 214; 1916, 613), and serves to detect small degrees of watering in a larger number of cases. Dilution of milk with isotonic solutions of dextrose, glycerol, sodium bicarbonate, etc. alters the value of the serodensimetric constant, whereas the cryoscopic index remains unchanged. T. H. POPE.

Microscopical identification of malted milk and its flavoured products. F. ILLIG and B. G. HARTMANN (*J. Assoc. Off. Agric. Chem.*, 1929, **12**, 238—240).—The microscopical appearance of various preparations is described and illustrated by photomicrographs. In genuine malted milk the malt extract solids and milk solids are incorporated into homogeneous irregular fragments having a stippled surface. These are readily recognisable even in admixture with cocoa, sugar, etc. Mechanical mixtures of spray-dried whole milk and spray-dried malt extract are easily distinguishable from the genuine product. W. J. BOYD.

Margarine containing egg yolk. J. ANGERHAUSEN and G. SCHULZE (*Pharm. Zentr.*, 1929, **70**, 389—392).—Heating of egg yolk at 90° for 3 hrs. or at 65° for 6 hrs. renders it insoluble in 2% salt solution (cf. *B.*, 1929, 338). Dry egg yolk added to margarine without previous swelling or dissolution in milk or salt solution is extracted only with difficulty by means of 2% salt solution. If the yolk is added to the mixes as a suspension in milk, salt solution, or molten fat, there may be sedimentation resulting in uneven distribution of the proteins in the margarine. Under these circumstances the Fendler test is not reliable. The compositions and properties of dried egg yolk, "Heliozithin" (a lecithin product of animal origin), and soya-bean phosphatide are compared. "Heliozithin" reacts negatively to Fendler's tests, whereas soya-bean phosphatide gives a

positive reaction on boiling the salt solution extract with hydrochloric acid and a negative result for the dialysis test. The presence of egg yolk in margarine may in some cases be detected by determining the nitrogen content of the product. This may be increased from 1% due to milk proteins to 1.35% according to the amount of yolk added. In many cases it may be detected by determining the lecithinphosphoric acid.

W. J. BOYD.

Comparative results with Arland's potato starch tester and Reimann's potato balance. A. ARLAND (*Z. Spiritusind.*, 1929, **52**, 206—207). B. LAMPE (*Ibid.*, 207).—Opposing Ekhard's statement (*ibid.*, 1928, **51**, 317—318) that potatoes tested in his apparatus need not be air-dried, the author gives the results obtained by testing several series of samples both dry and after treatment with water for various periods and wiping. The results, both with his own and Reimann's apparatus, show variations after moistening up to 0.75% of starch, and these not always higher, as stated by Ekhard. Reimann's scale is said soon to become untrustworthy with use.

LAMPE adversely criticises Arland's apparatus, since it does not admit of a dirt determination, his conclusions, as expecting too great accuracy of the methods, and his statement regarding Reimann's scale. F. E. DAY.

Bull's rapid method of determining fat in fish, meat, and other animal or vegetable products. J. SEBELIEN (*Chem.-Ztg.*, 1929, **53**, 489—490).—The material is thoroughly minced and mixed, 5 g. are weighed out (on a "per cent. balance" described below), and 7.5 g. of anhydrous sodium sulphate (for unsalted wares) or 15 g. of ignited gypsum (for salted wares) are incorporated therewith in a mortar and pestle. The mixture is placed in a wide-mouthed bottle with ground stopper and shaken with exactly 40 c.c. of benzene at intervals during 1 hr. After settling, 16 c.c. of the clear fat solution are withdrawn by means of a pipette fitted with a filter consisting of a short tube filled with cotton wool, run into the warm balance pan, evaporated to dryness, and the fat is weighed. In the case of milk more sodium sulphate should be used. For dry meals no addition of water-binding salt is necessary, but longer time of extraction is allowed. The "per cent. balance" consists of a beam suspended on the edge of a cylindrical container so that the projecting arm is thrice the length of that hanging over the container. The short arm carries a pan suspended within the container by a rod passing through a hole in the lid. In the lower part of the container is an electrical heater, whereby the pan can be warmed. The weighings are made by means of suitable riders hung on the graduated longer arm of the beam, and adjustment of the balance is by means of a screw on the end of the shorter arm. Sample and salt are weighed out on the cold pan, and the latter is then warmed in readiness for evaporation of the solvent from the fat. W. J. BOYD.

Shortening the time of [Kjeldahl] nitrogen determinations by the use of the grid burner. H. D. SPEARS and W. G. TERRELL (*J. Assoc. Off. Agric. Chem.*, 1929, **12**, 250).—Using copper sulphate as catalyst in Kjeldahl determinations of the nitrogen in

feeding stuffs it was found that the time of digestion could be shortened to $1\frac{1}{2}$ hrs. by using a Fisher grid burner (cf. Shedd, B., 1927, 954). W. J. BOYD.

Labour-saving devices for the commercial protein-testing laboratory. W. O. WHITCOMB (Cereal Chem., 1929, 6, 215—217).

Phosphorus of grains. GREAVES and HIRST.—See XVI. Zinc in sugar products. SPENGLER and ZABLINSKY.—See XVII. Quality of yeast. WERNER and SIEDHOFF.—See XVIII.

See also A., July, 852, Comparative vitamin-B values of pulses and nuts (PLIMMER and others). 853, Distribution of vitamin-B₂ in certain foods (AYKROYD and ROSCOE). 854, Chemical changes in pears on storage (EMMETT). 855, Titanium in phanerogamic plants (BERTRAND and VORONCA-SPIRT). 857, Glutelins of rye and barley (CSONKA and JONES).

PATENTS.

Manufacture of milk powders; cream powders, and the like. J. E. NYROP (B.P. 312,875, 30.8.28. Addn. to B.P. 297,256; B., 1928, 825).—To prevent the development of "powder taste" when milk powder is stored, the powder is subjected, before packing, to additional drying by evacuating the containers in the presence of drying agents; a dry inert gas such as carbon dioxide may subsequently be admitted to the container. E. B. HUGHES.

Ice-cream mixture and its preparation. J. R. HATMAKER (B.P. 309,417, 7.11.27).—A suitable proportion, e.g., 40%, of a given quantity of natural milk is dried and the solids are incorporated with the remainder of the milk. Cream, sugar, and/or other flavouring materials, with or without a stabilising agent are added, and the whole is mixed. The product should contain about 60% of water. W. J. BOYD.

Manufacture of ice cream or similar frozen food product. C. E. GRAY and G. D. TURNBOW (B.P. 312,804, 26.5.28).—Ice cream containing a larger amount of solids-not-fat than can ordinarily be used without development of "sandiness" due to the crystallisation of lactose is prepared by adding to a partly-frozen mix rather low in content of solids-not-fat the required additional milk solids, such as skim-milk powder, in which the lactose is in an undissolved and finely-divided condition. E. B. HUGHES.

Emulsified [food] products and their manufacture. A. K. EPSTEIN and M. C. REYNOLDS (B.P. 313,237, 26.3.28).—A modified egg beater is described in which emulsions such as mayonnaise are prepared in an atmosphere of inert gas, e.g., carbon dioxide, under slight pressure. E. B. HUGHES.

Leaching-out protein-containing substances such as [salt-containing] fish waste and the like. M. STERN (CHEM. FABR. SCHLUTUP DR. M. STERN) (B.P. 282,068, 7.12.27. Ger., 7.12.26).—To remove the salt the extraction liquid is deprived of its solvent power for proteins, before or during the extraction, by the addition of metallic salts (e.g., of iron, aluminium, zinc, or chromium) or colloids capable of precipitating protein. Protein and

fat removed by extraction with untreated liquid may be precipitated by the addition of such precipitants to the liquid after the extraction, and recovered.

W. J. BOYD.

Preservation of citrus fruit juices. V. R. KOKAT-NUR (U.S.P. 1,715,932, 4.6.29. Appl., 14.1.27).—To the juice of the fruit is added an aqueous extract of the skin, and the whole is then heated at 45—55° for at least $2\frac{1}{2}$ hrs. No preservative is added.

E. B. HUGHES.

Production of food preservatives, food adjuncts, condiments, etc. R. HADDAN. From SMOKED PRODUCTS Co. (B.P. 309,519, 9.1.28).—Salt is agitated in presence of smoke produced by burning carbonaceous material, preferably hickory wood. The smoke may be submitted to the action of an electrical field in order to precipitate certain constituents on the wall of the rotating chamber through which it is passing and in which the salt is agitated. The precipitate is removed from the wall by a scraper or by the abrasive action of the salt, and mixed with the latter, which is not subjected to the electrical action. W. J. BOYD.

Ovens for baking, roasting, and drying purposes. J. B. Cox (B.P. 312,016, 3.10.28).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Collargol. A. F. GERASIMOV (Trans. Sci. Chem. Pharm. Inst. Moscow, 1926, No. 16, 69—108).—The preparation of collargol from egg-white and silver oxide is described. In low concentrations, ammonium chloride and thiocyanate, potassium iodide and bromide, calcium chloride, and ferric chloride hinder coagulation by acetic acid, whilst these salts in high concentrations, or potassium chlorate, potassium nitrate, sodium sulphate, ammonium sulphate, or magnesium sulphate in any concentration, induce coagulation. The results are discussed. CHEMICAL ABSTRACTS.

Salicylsulphonic acid. J. RAE (Pharm. J., 1929, 122, 618).—Examination of various specimens of sulphosalicylic acid (purity 86.3—98.7%, water content 0.7—14.4%, free salicylic acid content 0—1.1%) indicates that it exists in two forms, m.p. (anhyd.) about 110° and 120—124°, respectively. H. BERTON.

Determination of camphor in camphor liniment. I. U.S.P. X. method. C. F. POE, G. LIPSEY, and C. L. VAUGHN (J. Amer. Pharm. Assoc., 1929, 18, 580—586).—The method given in the U.S.P. X. gives consistently low results due to the oxidation of the triolein present in the cotton-seed oil used for making the liniment. Dishes made from different materials were used, but none of them was satisfactory. A more satisfactory method has been developed, using a vacuum oven, and will be published later. C. C. N. VASS.

Bioassays. Tincture of *Strophanthus*. C. W. EDMUNDS, H. W. LOVELL, and S. BRADEN (J. Amer. Pharm. Assoc., 1929, 18, 568—573).—The assay of various tinctures of *Strophanthus* by the 1-hr. frog (U.S.P.), 4-hr. frog, the intravenous, and cot methods gives results varying from 2.45 to 8.00 mg. of ouabain (limits). The results for any one preparation do not

agree, and are usually lowest by the cot method and highest with the 4-hr. frog method. It is considered that the U.S.P. standard requires revision.

C. C. N. VASS.

Examination of Peru balsam by the extended capillary diagram and the analytical quartz lamp. C. A. ROTHENHEIM (Pharm. Ztg., 1929, 74, 712—713; cf. Rapp, B., 1929, 110).—A number of balsams, natural resins, and esters were tested by this means in the presence of ether, ethyl acetate, ligroin, alcohol, acetone, petrol, benzene, chloroform, methyl alcohol, and water, but only aqueous extracts of Peru balsam gave a characteristic electric-blue fluorescence in ultra-violet light. This may be used as a test for Peru balsam.

S. COFFEY.

Examination of bogs for medicinal purposes. K. STOCKFISCH and W. BENADE (Z. angew. Chem., 1929, 42, 663—668).—Some properties of earths used for medicinal baths and treatments have been examined.

S. I. LEVY.

Essential oils of *Heracleum villosum*, Fisch. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 119—126).—As the fruit ripens, the aldehydes disappear and alcohols appear. Saponification of the oil affords octyl alcohol and acetic and butyric acids. CHEMICAL ABSTRACTS.

Borneol values of the Siberian pine oil of Altai. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 127—130).—A sample had d_{20}^{20} 0.9581, $[\alpha]_D$ -39.59°, n_D^{20} 1.4748, acid value 9.67, ester value 139.21 (after acetylation 190.84); α -pinene and camphene were present. Borneol (40%) was obtained by saponification (18%), distillation with steam (2%), and treatment with phthalic anhydride (20%). CHEMICAL ABSTRACTS.

Essential oils from Caucasian *Citrus* species. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 69—85).—Oil from leaves of *Citrus bigardia*, Risso, contained camphene, limonene, dipentene, α -terpineol, geraniol, and nerol. Oil from leaves of a Japanese orange (Unshiu) was also examined. CHEMICAL ABSTRACTS.

Essential oils of *Hyssopus officinalis*. L. P. GUSEVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 159—167).— α -Pinene, *l*-pinocamphone, an aldehyde (semicarbazone, m.p. 202—203°), alcohols, and a sesquiterpene are present. CHEMICAL ABSTRACTS.

Essential oils of peppermint from Poltava Government. V. A. KUZMIN (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 168—182).—Yields are tabulated, and constants of the oils are recorded.

CHEMICAL ABSTRACTS.

Essential oils of leaves and stems of camphor from the Sukhum district. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 25—38).—The oil contained *d*- and *dl*- α -pinene, cineole, limonene, dipentene, *d*-camphor, borneol, *d*-camphene, and unidentified phenols.

CHEMICAL ABSTRACTS.

Essential oils of Crimean junipers. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 142—150).—Oil from twigs and needles of *Juniperus excelsa*, M.B., d_{20}^{20} 0.8812, $[\alpha]_D$ +31.5°, n_D^{20} 1.4769, acid value 2.52, ester value 5.52 (after acetylation 28.49), contained α -pinene, an aldehyde, a hydrocarbon having d_{20}^{20} 0.9286, $[\alpha]_D$ -6.75°, n_D^{20} 1.500, cedrene, and a cedrol, m.p. 82—84°, $[\alpha]_D$ +9.8°. CHEMICAL ABSTRACTS.

Essential oils of Crimean salvias. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 109—118).—Oil from *Salvia grandiflora*, Etzl., contained α - and β -pinene, camphene, *d*-camphor, and caryophyllene.

CHEMICAL ABSTRACTS.

Essential oils of *Abies nordmanniana*, Spach, and *Abies cephalonica*, Link. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 131—141).—The oil of *Abies nordmanniana*, Spach, d_{20}^{20} 0.8816, $[\alpha]_D$ -22.40°, n_D^{20} 1.4775, acid value 1.08, ester value 28.24 (content 9.89%), ester value after acetylation 48.53 (total alcohols 13.84%), contained α - and β -pinene and camphene. The oil of *Abies cephalonica*, Link, d_{20}^{20} 0.8995, $[\alpha]_D$ -20.00°, n_D^{20} 1.4773, acid value 3.29, ester value 48.14 (after acetylation 73.3), contained α -pinene, *dl*- α -pinene, and camphene. Borneol was obtained by saponification. CHEMICAL ABSTRACTS.

Essential oil from *Artemisia annua*, L. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 15—24).—The oil contains α -pinene, cineole, two ketones (d_{20}^{20} 0.8755, $[\alpha]_D$ -2.04°, n_D^{20} 1.4702, semicarbazone, m.p. 94—96°; and semicarbazone, m.p. 68—71°), a camphor (m.p. 175—177°, $[\alpha]_D$ -44.52°), borneol, cuminaldehyde, a phenol, and acetic and butyric acids.

CHEMICAL ABSTRACTS.

See also A., July, 806, **Germicidal action of hydroxydiaryl sulphides** (HILBERT and JOHNSON). 819, **New dihydroterpene** (BLANCO). 829, **Microchemical reactions of piperine** (WAGENAAR). **New reaction of hydrastine and papaverine** (ROJAHN and STRUFFMANN). 830, **Belladonnine, bellatropine, and chlorotropan** (M. and M. POLONOVSKI). **Colour reactions of sinomenine and sinomenol** (GOTO). **Strychnine and brucine** (PERKIN and ROBINSON). 832, **Determination of ergot preparations** (VAN URK). **Use of vanillin as alkaloidal reagent** (PORTNOV). 833, **Chemotherapy of bromine derivatives of phenylarsinic acids and arsenobenzenes** (HAYTHORNTHWAIT). **Reagent for identifying arsenic acids** (BARBER). 836, **Micro-determination of chloroform** (YODOMIGAWA). **Determination of citric and tartaric acids** (PIRRONE). 846, **Reagent for detection and determination of quinine** (STERKIN and HELFGAT). **Micro-determination of morphine** (FLEISCHMANN). **Buffer antiseptics** (GJALDBAEEK). 850, **Diphtheria toxin** (LEULIER and others). **Purification of vaccine virus** (YAIO and KASAI). 851, **Crystalline insulin** (JENSEN and others.). **Insulin; physiological assay** (CULHANE and others). 852, **Purification of antineur-**

itic vitamin-**B** (DE CUGNAC). Separation of vitamin-**B**₂ from -**B**₁ in yeast (CHICK and ROSCOE). 853, Vitamin-**B**₂ in certain foods (AYKROYD and ROSCOE). 855, Water in tobacco (GAVRILOV and EVSLINA). 857, Alkaloidal constituent of *Artabotrys suaveolens*, Blume (MARAÑON; SANTOS).

PATENTS.

Medicinal preparation. E. W. LIPSCHUTZ, Assr. to BROOKLYN SCIENTIFIC PRODUCTS Co., INC. (U.S.P. 1,714,430, 21.5.29. Appl., 24.5.27).—A mixture comprising oleic acid and bile salts is used for the treatment of diseases of the biliary tract. F. G. CLARKE.

Manufacture of tuberculin preparations. I. G. FARBENIND. A.-G. (B.P. 285,087, 9.2.28. Ger., 11.2.27).—Albumins are precipitated from solutions of tuberculin preparations, *e.g.*, those obtained from Koch's broth by precipitation with alcohol, by the addition of heavy-metal compounds other than complex metal compounds, *e.g.*, dialysed ferric chloride or lead acetate, and the active constituents are precipitated from the solutions after filtration by the addition of, *e.g.*, picric acid or acetone. L. A. COLES.

Separation of active ovarian hormones. N. V. ORGANON TOT BEREIDING VAN ORGAANPREPARATEN OP WETENSCHAPPELIJKE GRONDSLAG (B.P. 291,081, 3.4.28. Holl., 28.5.27).—The urine of pregnant women or female animals is extracted with a fat solvent and the residue left after evaporation of the solvent is extracted with water, insoluble constituents being removed.

L. A. COLES.

Manufacture of 8-amino-6-alkoxyquinolines. I. G. FARBENIND. A.-G., W. SCHULEMANN, F. SCHÖNHÖFER, and A. WINGLER (B.P. 310,559, 27.1.28. Addn. to B.P. 267,457; B., 1927, 379).—6-Alkoxyquinoline-8-carboxylic esters are subjected to the Curtius reaction. *Methyl 6-methoxyquinoline-8-carboxylate*, m.p. 77–79°, b.p. 163–165°/1 mm., is converted by way of the *hydrazide*, m.p. 178–179°, *azide*, and *urethane*, m.p. 76–77°, into 8-amino-6-methoxyquinoline.

C. HOLLINS.

Manufacture of organic arsenic preparations and the application thereof as seed grain immunising media. I. G. FARBENIND. A.-G. (B.P. 287,093, 13.3.28. Ger., 14.3.27).—The products comprise compounds, insoluble or sparingly soluble in water, containing arsenic attached to an aromatic nucleus and produced in a colloidal form as described for the manufacture of colloidal organic mercury compounds in B.P. 243,361 (B., 1926, 691), the compounds being used alone or in admixture with other fungicides or bactericides.

L. A. COLES.

Manufacture of organic mercury compounds. "TASCH" LAB., LTD. (LABORATORIUM "TASCH" A.-G.) (B.P. 301,023, 8.11.28. Ger., 23.11.27).—The sulphamic acid of antipyrène (4-amino-1-phenyl-2:3-dimethylpyrazolen-5-one) is treated with mercuric oxide and sulphuric acid to give a soluble compound having spirillo-cidal properties. C. HOLLINS.

Extraction of essential oils or odorous substances. VIANOVA GES.M.B.H. F. CHEM. IND. (B.P.

292,982, 26.6.28. Ger., 28.6.27).—Natural material containing the oils etc. is extracted with liquid carbon dioxide. L. A. COLES.

Immunising product and its production. N. S. FERRY, Assr. to PARKE, DAVIS & Co. (U.S.P. 1,717,198, 11.6.29. Appl., 2.6.24. Renewed 8.12.28).—See B.P. 293,401; B., 1928, 692.

Manufacture of salts of inositolhexaphosphoric acid. F. GOEDECKE (U.S.P. 1,715,031, 28.5.29. Appl., 14.11.27. Ger., 11.8.25).—See B.P. 308,403; B., 1929, 453.

Manufacture of esters and other valuable organic products. F. C. ZEISBERG, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,708,460, 9.4.29. Appl., 26.3.27).—See B.P. 287,846; B., 1929, 549.

Alkoxyacridines. L. BENDA and W. SCHMIDT, Assrs. to WINTHROP CHEM. Co., INC. (U.S.P. 1,715,332, 28.5.29. Appl., 26.4.23. Ger., 24.5.22).—See B.P. 217,715; B., 1924, 768.

Products of the benzodiazine series (B.P. 310,076).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Production of direct positives. R. SESMA (Bull. Soc. franç. Phot., 1929, 16, 108–110).—The exposed film is preferably treated with a desensitiser, developed, transferred to an acid bath, washed, and then immersed in a bath which at the same time removes the reduced silver and hardens the surrounding gelatin. The bath contains copper sulphate (10 g.), potassium bromide (8 g.), chromic acid (1 g.), and water (1 litre). When reduction is complete, the film is washed, dried, and then redeveloped to a positive by a rapid developer. Incomplete drying leads to general fog. J. W. GLASSETT.

See also A., July, 741, **Photographic sensitivity** (MASAKI). 771, **Photographing explosion waves in carbon monoxide-oxygen mixtures** (BONE and FRASER). 777, **Photochemical reactions with ferric thiocyanate and with neocyanine** (BHATTACHARYA and DHAR).

PATENT.

Photographic developers [in tablet form] and developing processes. J. HALDEN & Co., LTD., and J. HOLDEN (B.P. 311,546, 4.5.28).

XXII.—EXPLOSIVES; MATCHES.

Explosibility of atmospheres behind [mine] stoppings. H. F. COWARD (Trans. Inst. Min. Eng., 1929, 77, [2], 94–115).—A graph is given, based on previously published data (Coward and Harwell, B., 1926, 426), from which it can be predicted whether a given mixture of methane, oxygen, and nitrogen is explosive or not. The error introduced by reckoning the carbon dioxide usually present as nitrogen is very small and on the side of safety. The limits of inflammability of the above gas mixture, as deduced from the graph, are tabulated. Similar graphs are also given for mixtures of oxygen and nitrogen with hydrogen and also with

carbon monoxide, based on unpublished results of Jones, and a method of calculating by means of these curves the explosive ranges of complex mixtures of these gases with methane is described. Curves showing the lower limits of inflammability of all mixtures of methane, hydrogen, and carbon monoxide in air are appended to the paper.

S. K. TWEEDY.

See also A., July, 771, **Ignition temperatures of carbon monoxide-air mixtures** (PRETTRE and LAFFITTE). **Photographic study of flame movement in carbon monoxide-oxygen explosions** (BONE and FRASER). **Explosion of combustible gases mixed with air** (HABER).

PATENTS.

Explosive. C. O. BLACK and W. A. MOORE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,713,816, 21.5.29. Appl., 18.7.27).—A moss, *e.g.*, sphagnum, is used as the absorbent in a dynamite or other composition containing a liquid explosive such as nitroglycerin.

F. G. CLARKE.

Explosive for signalling. W. O. SNELLING, Assr. to TROJAN POWDER Co. (U.S.P. 1,709,636, 16.4.29. Appl., 11.12.24).—A composition of nitrated starch and a non-explosive signalling material is detonated.

W. G. CAREY.

Propellant powder charge. J. B. FIDLAR (U.S.P. 1,716,434, 11.6.29. Appl., 5.12.21).—The propellant powder includes 35 pts. of nitrocellulose, 25 pts. of nitroglycerin, 5 pts. of dinitrotoluene, 20 pts. of trinitrotoluene, and 10 pts. of flame-reducing materials.

F. G. CROSSE.

Preparation of pentaerythritol tetranitrate. II. A. AARONSON (U.S.P. 1,705,699, 19.3.29. Appl., 8.11.23).—Pentaerythritol is stirred into about 6 pts. of nitric acid (at least 90%) at about 15°. After 15 min. the spent acid is withdrawn and the pentaerythritol tetranitrate washed with water and soda and crystallised.

R. BRIGHTMAN.

XXIII.—SANITATION; WATER PURIFICATION.

Proposal to render innocuous the waste waters from beet-sugar factories. H. BACH (Z. Ver. deut. Zucker-Ind., 1929, 79, 241—250).—In cases where sufficient suitable land is not available for the disposal of the factory effluent by irrigation, it is proposed that after a preliminary purification from suspended matters in settling tanks the effluent should be discharged without further treatment, and that the river or stream into which it flows should be aerated for a sufficient distance from the point of discharge to maintain the normal amount of dissolved oxygen in the water. It is claimed that this would prevent injury to fish and also odours due to putrefaction, both being the result of abstraction of dissolved oxygen from the water by the organic matters in the effluent. The plant suggested is a pipe supplied with air from a compressor and extending along the bank of the stream in the direction of flow for a distance of a mile or more, if necessary, with branch pipes at intervals leading to air-discharge boxes of finely-perforated or porous material immersed at a sufficient depth in the stream.

J. H. LANE.

Determination of hardness in water by means of soap solution. G. BRUHNS (Chem.-Ztg., 1929, 53, 469—470).—The calcium hardness and the magnesium hardness may both be determined in the same sample by a slight modification of the Boutron-Boudet method. The sample is first treated with 0.1*N*-sodium hydroxide in presence of phenolphthalein to neutralise free carbonic acid, and then titrated with the solution very slowly. A faint rose colour and a permanent lather mask the calcium hardness point; on further addition the colour deepens, but the lather breaks, until the end-point for magnesia hardness is reached with a red colour and a renewed permanent lather.

S. I. LEVY.

PATENTS.

Manufacture of materials suitable for addition to toilet baths. C. DRANGSHOLT (B.P. 285,356, 13.2.28. Norw., 12.2.27).—When the mixture of humic acids obtained in known manner from peat etc., by treatment with alkalis, is extracted by alcohol or other suitable solvent, humatomeanic acid is obtained, the use of which, in the form of its sodium or lithium salt, in baths is claimed.

C. JEPSON.

Purification of polluted liquids. H. D. ELKINGTON. From TRAVERS-LEWIS PROCESS CORP. (B.P. 312,754, 10.4.28).—If the colloidal matter present in a polluted liquor be given a negative charge by maintaining a p_H value of 8—11 it becomes capable of combining with or adsorbing positively-charged ions from any electrolyte producing material which is able to function without precipitation in an alkaline solution, *e.g.*, calcium sulphate. The suspensoids thus produced may be coagulated, *e.g.*, by means of an iron salt, with the removal of the bulk of the organic matter present and consequent purification of the polluted liquid. Waste material containing calcium carbonate and/or sulphate, and an iron salt, *e.g.*, cement dust, sludge from the purification of iron pickle, or from alkali plants, may be utilised for this purpose, the composition being adjusted, if necessary, to approximate to 50% of electrolyte and 15% of coagulant. The amount of such material to be used depends on the quantity of colloidal matter present, which it is suggested should be determined by dialysis in the presence of an inert gas (cf. B., 1928, 732).

C. JEPSON.

Treatment of acidified mine water. J. T. TRAVERS, Assr. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,714,828, 28.5.29. Appl., 9.7.28).—The acid water falls upon, and percolates through, successive masses of travertine.

F. G. CLARKE.

Purification of polluted liquids and industrial waste. J. T. TRAVERS, Assr. to TRAVERS PROCESS CORP. (U.S.P. 1,715,877, 4.6.29. Appl., 17.10.27).—See B.P. 312,754; preceding.

Respiratory gas masks or face pieces. F. C. JONES (B.P. 312,594, 28.2.28).

Refuse destructors. HEENAN & FROUDE, LTD., and A. E. W. JAMES (B.P. 313,757—8, 25.6.28).

Base-exchange materials (B.P. 313,206).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 30, 1929.

L—GENERAL; PLANT; MACHINERY.

Flow of mercury vapour through nozzles.

W. J. KEARTON (Inst. Mech. Eng., June, 1929. Advance copy. 33 pp.).—The mass discharge of mercury vapour through a convergent-parallel nozzle is shown to be in excess of the theoretical value for stable expansion in thermal equilibrium when the initial superheat is below 10° , but compatible with expansion in the supersaturated state down to the throat pressure, with nozzle efficiencies of 85–95%. The apparently excessive mass discharge is not due to the presence of liquid particles. Data from search-tube experiments are consistent with expansion of vapour in the supersaturated state, but inconsistent with expansion in the saturated state. Whatever degree of supersaturation is reached in the first stage of the flow process, the vapour never reverts to the saturated state. Qualitative tests indicate the possibility of supersaturation of the order of 2000 being reached before the establishment of thermal equilibrium begins.

C. J. SMITHELLS.

Condensation of steam. D. F. OTHMER (Ind. Eng. Chem., 1929, 21, 576–583).—The effect of temperature, concentration of small amounts of air, and of temperature drop on the rate of condensation of steam on an isothermal condensing surface has been studied. Within the limits of experimental error (using steam at 100 – 115° and percentages of air up to 11.4%), various empirical relationships were obtained which are expressed mathematically. The results obtained show that a rise in temperature of the steam causes the value of the coefficient of heat transfer from the steam to the condensing surface to rise. This is due to the fact that the viscosity of the condensed water film decreases with rise in temperature, thus increasing the rate of drainage of the water from the condensing surface. In addition, the thermal conductivity of the water film increases with the temperature.

H. INGLESON.

Extended steam tables. H. L. CALLENDAR (Inst. Mech. Eng., June, 1929. Advance copy. 21 pp.; cf. A., 1928, 1179).—Extended steam tables for saturated steam at 400 – 717° F. and 400 – 3200 lb./in.² and for superheated steam at 400 – 1000° F. and 400 – 4000 lb./in.² are given, together with other tables arranged both on a temperature and on a pressure basis, in both Fahrenheit and Centigrade units. C. J. SMITHELLS.

Gravitational flow of fertilisers and other comminuted solids. W. E. DEMING and A. L. MEHRING (Ind. Eng. Chem., 1929, 21, 661–665).—The rate of flow of comminuted solids, such as potassium nitrate pellets, crystallised monoammonium phosphate and urea, glass beads, lead shot, marbles, crushed phosphate rock, and several types of seeds, from bins or hoppers

depends on the average particle size, the kinetic coefficient of friction, and apparent density of the material, the diameter of the aperture, and the vertical angle of the hopper or, if all the material does not flow out, on the angle between opposite slopes of the remaining material. A mathematical expression is derived for the rate of such gravity flow and is found to agree satisfactorily with practice.

D. F. TWISS.

Two pieces of apparatus for measuring the sp. gr. of liquids by means of a hydrometer. P. FUCHS (Chem.-Ztg., 1929, 53, 526–527).—To measure the sp. gr. of a hot liquid in a shallow basin by means of a hydrometer, the instrument is placed inside a narrow cylinder open at the lower end and terminating in a narrow tube at the upper end; the lower end of the cylinder is placed below the surface of the solution, and by applying suction to the tube the liquid is drawn into the cylinder until the hydrometer floats. In using a hydrometer in a narrow cylinder the instrument may be prevented from touching the walls by placing over its narrow upper tube a small copper wire ring with three arms at angles of 120° to one another, the arms being bent so that when the wire rests on the cylinder the ring is directly over its longitudinal axis.

A. R. POWELL.

PATENTS.

Furnace. I. HARTER, Assr. to FULLER-LEHIGH Co. (U.S.P. 1,712,919, 14.5.29. Appl., 16.12.25).—The walls of the furnace are double with an air space between, through which cooling air circulates; cooling of the inner walls is accelerated by backing the refractory tiles of which it is built up with thin metallic sheets having numerous projections extending into the cooling space.

A. R. POWELL.

Regenerative furnaces. WOODALL-DUCKHAM (1920), LTD., and M. H. MCEWAN (B.P. 314,171, 28.4.28).—In regenerative furnaces which are adapted to burn either lean gas (which is preheated in a manner similar to the air) or strong gas (which is not preheated, thus releasing additional regenerator settings for preheating air), the inlet valves which determine whether lean gas or air passes to the regenerators are separate from the inlet valves which are used in turn with exhaust valves to effect reversal.

B. M. VENABLES.

Heat-exchange apparatus. R. VUILLEUMIER, Assr. to SAFETY CAR HEATING & LIGHTING Co. (U.S.P. 1,716,333, 4.6.29. Appl., 14.10.16. Renewed 6.12.20).—The inner fluid passes through a number of tube coils and the outer fluid between the coils and an elongated container. Each fluid space is filled with beads of conducting material, e.g., copper, to improve the heat transmission. To prevent undesired transmission in

the longitudinal direction, strata of non-conducting material is crushed in a vertical annular space between a hollow rotating hub and a fixed central boss, and ground in a horizontal annular space between the above two parts extended outwards, this space being provided with balls which are located in "concentric rings" but are not constricted in grooves. B. M. VENABLES.

Repairing heated structures such as furnaces, retorts, etc. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 313,728, 24.5.28).—A charge of jointing material is inserted into a container with an explosive cartridge behind it. The container is placed on a carrier which also includes a nozzle adapted to guide the material into the crack and is provided with a long handle made up of screwed rods; heavier models may also be provided with wheels. After the apparatus has been placed opposite the crack to be sealed, the cartridge is exploded by the heat of the furnace permeating to it (or electrically if more convenient) and the material is forced rapidly and deeply into the crack.

B. M. VENABLES.

Heated drum apparatus for desiccating liquids and semi-liquids. R. HADDAN. From HEYL-BERINGER FARBENFABR. A.-G. (B.P. 313,817, 5.9.28).—A heated drum is supplied with a layer of the material by two (or more) feed rollers in series, the object of the intermediate roller(s) being to prevent the first roller (which dips into the feed tank) from becoming too hot by conduction from the heated drum.

B. M. VENABLES.

Freezing of colloidal liquids. B. P. TSITOVITSCH (B.P. 288,310, 4.4.28. Esthonia, 8.4.27).—The colloidal liquid such as milk, fruit juices, medicinal sera, beverages, is instantaneously frozen by being delivered in a finely-divided state into an air chamber in such a manner that no movement of the air is induced, the chamber being maintained below the f.p.; the settled finely-divided solid or snow is stored at a temperature below its m.p. until such time as it is desired to resume its original condition, which it will do, with its flavour etc. unchanged, by simple thawing.

B. M. VENABLES.

Crusher. H. H. RUMPEL, Assr. to SMITH ENG WORKS (U.S.P. 1,717,894, 18.6.29. Appl., 22.8.27).—The crushing motion is similar to that of a gyratory crusher, but the vertical shaft rotates without gyration at its lower part in bearings in the lower fixed part of the crusher; the upper part of the shaft is obliquely cranked and carries the crushing head on bearings permitting the shaft to rotate independently of the head.

B. M. VENABLES.

Crushing mills. E. C. LOESCHE (B.P. [A] 313,690, [B, C] 313,844—5, [A, B] 14.4.28, [C] 7.1.29).—(A) A concave dish is rotated about a vertical axis, and a roller engages with the interior at the circumference where the surface is rising outwardly. The roller runs on an axis that is fixed except that it is pressed downwards by spring pressure. The ratio of the diameters roll : dish must not exceed 1 : 2.5, and preferably should not exceed 1 : 2. An upstanding rim may be provided at the edge of the dish. (B) The roller is not allowed quite to touch the dish, a definite stop being provided. (C) Outside the dish and fixed to it are a number of inclined vanes which lift material that has dropped into the containing casing and return it to the grinding zone.

B. M. VENABLES.

Crushing, grinding, or milling apparatus. R. E. TROTIER (B.P. 296,723, 3.8.28. Fr., 6.9.27).—The

Grinding, crushing, pulverising, or disintegrating mills. W. A. CLOUD (B.P. 313,656, 16.3.28).—A rotating mill is divided into a number of short cylindrical sections increasing in diameter as the grinding progresses. The linings may be fluted and each section may contain one or more correspondingly fluted grinding rollers. If several rollers are used they are held apart by links and pins, and may be provided with springs to increase the pressure. Radial partitions are provided between each pair of zones, preferably double-walled, with lifting scoops in the spaces between. The holes for transfer of material preferably become larger in the direction of travel. The outlet is axial and an indirect air draught may be used, also a screen to prevent exit of large particles.

B. M. VENABLES.

Mixing solids with liquids. W. EVANS (B.P. 314,261, 10.8.28).—A continuous measured stream of finely-divided solids (which may be air- or gas-borne) is projected at high velocity against the surface of a measured stream of the liquid. The solid and the whole of the liquid may be admitted together into a downtake compartment partitioned off from a large tank, or the solid may be projected on to the surface of a pool formed in the stream of part only of the liquid and the carrier gas, partly deprived of its solid content, scrubbed in a tower or other device by another partial stream of liquid, the above two streams then being combined and added to the remainder of the liquid.

B. M. VENABLES.

Apparatus for continuously treating liquids. A. E. FLOWERS, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,701,068, 5.2.29. Appl., 22.6.26).—The fluid and other material are mixed and circulated continuously through partitions devised to divide the mixture into thin layers inclined at an angle substantially less than the angle of repose of either material, so that the lighter material or fluid is removed continuously at one end, and the heavier discharged at the other.

R. BRIGHTMAN.

Filtering material. H. BLUMENBERG, JUN. (U.S.P. 1,702,104, 12.2.29. Appl., 10.8.27. Renewed 22.12.28).—Calcium hypochlorite is incorporated with 5–10% of sodium carbonate and about 70% of diatomaceous earth for use in the filtration, e.g., of mineral oil distillates, industrial waste waters, sewage, etc.

R. BRIGHTMAN.

Continuous-filter thickening apparatus. A. L. GENTER, Assr. to GENTER THICKENER CO. (U.S.P. 1,716,040, 4.6.29. Appl., 16.10.25).—A Genter thickener is provided with a common filtrate receiver situated centrally among a number of banks of filter elements which may or may not be in separate containing vessels. Connexions with timed valves are so arranged that the filter elements are discharged by pressure in small groups at a time, the vacuum remaining on the other groups.

B. M. VENABLES.

Evaporator. B. S. and S. HUGHES (U.S.P. 1,717,927,

18.6.29. Appl., 8.3.26).—A shell is divided into an upper and a lower chamber with a tubular heating unit between the two. The charge of material in the lower chamber is pumped (or otherwise lifted) to the top of the heating unit, and evaporation takes place on the downward flow of the liquid. The rising pipe from the pump is centrally placed within the heating unit and is surrounded by an unobstructed large tube for ascension of the vapour.

B. M. VENABLES.

Device for preventing the boiling over of liquids which produce foam when boiling. D. HEYNIS (U.S.P. 1,709,529, 16.4.29. Appl., 29.4.27. Holl., 30.3.26).—The liquid is heated in a removable receptacle of such shape as to cause a lateral displacement of the centre of gravity of its contents when foam is produced. The receptacle is mounted on a platform which is tilted through a small angle by the raising of the foam, and the heating is controlled in response to the tilting movement of the support.

R. BRIGHTMAN.

Arrangement for cooling liquids or fluids. H. MAGER (U.S.P. 1,717,237, 11.6.29. Appl., 8.7.25. Austr., 7.2.25).—To reach a discharge chamber from a chamber placed immediately above, the fluid passes through a number of vertical cylinders, the end walls of which have holes arranged in a circle, registering with holes in the upper and lower chambers. The cylinders are arranged in pairs, the members of each pair rotating in opposite directions.

F. G. CLARKE.

Gas and liquid contact apparatus. F. W. SPERR, JUN., ASSR. to KOPPERS Co. (U.S.P. 1,715,252—3, 28.5.29. Appl., [A] 8.12.21, [B] 9.4.25).—A tower is charged with alternate strata having distributive and contact functions, respectively. In (A) the distributors are regularly arranged short vertical tubes with spiral vanes within, more than one layer being provided for each distributive stratum, and the contact strata are formed from irregularly arranged fragmentary material. In (B) both sorts of strata may be formed from rectangular bars of wood or other material; the bars of successive layers are placed at right angles forming a gridwork, and there are several layers to each stratum. The distributive bars are arranged with their diagonals vertical (and horizontal); the contact bars are preferably oblong in section and have their long axes vertical.

B. M. VENABLES.

Apparatus for bringing gaseous and liquid materials into contact. W. H. CAMPBELL (B.P. 313,983, 16.3.28).—Ribs are formed on the undersides of a number of flat plates which are piled together, the ribs are of varying depths to cause a slope on the plates when piled, and the channels between the ribs become deeper and narrower in the direction of flow of the liquid, which is zig-zag in both horizontal and vertical views, and countercurrent to the flow of gas. The apparatus is suitable for the cooling and crystallising of a liquid with or without evaporation. The ribs of a plate above may be on a sheet of rubber or other soft packing placed on the flat surface of a plate below, the rubber preventing both leakage and formation of a solid cake of crystals.

B. M. VENABLES.

Apparatus for condensing mercury or other vapour and vaporising water or other liquid.

BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of A. R. SMITH (B.P. 303,013, 21.12.28. U.S., 24.12.27).—The mercury is condensed on the outer walls of a bundle of tubes with headers at each end. As this steam-boiler unit will be subjected to very great expansion and contraction relative to the containing shell, yieldable sealing plates are provided between the boiler headers and the shell; the form shown is S-shaped in section forming short bellows. One shell may be provided for each boiler unit, or several boilers may be placed in one non-circular shell; in the latter case shields partly surround the tube bundles, openings being left facing the mercury inlet to effect uniform distribution of the mercury vapour.

B. M. VENABLES.

Catalytic apparatus. SELDEN Co., ASSEES. of A. O. JAEGER (B.P. 301,491 and 301,799, [A] 6. and [B] 20.11.28. U.S., [A] 2. and [B] 5.12.27).—Two types of plant involving the principle of the double countercurrent heat-exchange described in B.P. 306,884 (B., 1929, 672) are illustrated.

C. HOLLINS.

Vertical drying and dehydrating apparatus. M. BAECHLER, ASSR. to BAECHLER, KISER & CIE. (U.S.P. 1,718,104, 18.6.29. Appl., 26.11.27. Fr., 4.12.26).—See B.P. 281,664; B., 1928, 696.

Communiting solid substances. W. OSTERMANN, ASSR. to INDUSTRIAL SPRAY DRYING CORP. (U.S.P. 1,718,184, 18.6.29. Appl., 13.7.28. Ger., 18.5.23).—See B.P. 216,110; B., 1925, 1.

Apparatus for separating materials of different density. J. W. HARTLEY (U.S.P. 1,717,707, 18.6.29. Appl., 2.4.28. U.K., 4.4.27).—See B.P. 293,137; B., 1928, 658.

Air and gas washers and humidifiers. C. S. HANSARD and A. E. NETZEL (B.P. 314,283, 22.9.28).—See U.S.P. 1,691,971; B., 1929, 80.

Automatic-stoker retort furnaces. G. W. JOHN-SON. From AMER. ENGINEERING Co. (B.P. 314,624, 23.4.28).

Furnace walls. AMER. ENGINEERING Co. (B.P. 305,010, 7.5.28. U.S., 28.1.28. Addn. to B.P. 297,094).

Refrigerating machines. BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of C. STEENSTRUP (B.P. 293,394, 4.7.28. U.S., 5.7.27).

Refrigerating systems and apparatus. FRIGIDAIRE CORP., ASSEES. of H. B. HULL (B.P. 299,028, 23.1.28. U.S., 20.10.27).

Absorption refrigerating machines. SULZER FRÈRES Soc. ANON. (B.P. 295,009, 17.5.28. Switz., 6.8.27).

Compression refrigerating apparatus. J. A. GRIER and J. A. WARREN (B.P. 288,138, 19.3.28. U.S., 31.3.27).

Bag filters for cleaning air. MASCHINENFABR. BETH A.-G. (B.P. 314,297, 31.10.28. Ger., 1.10.28. Addn. to B.P. 281,994).

Mixed liquids and power generation (U.S.P. 1,716,130). **Removing carbon dioxide and hydrogen sulphide from gas mixtures** (B.P. 286,622).—See II. **Walls, furnaces, etc. of refractory materials** (B.P. 287,556).—See VIII. **Chemical balance** (U.S.P. 1,717,462).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Influence of mineral constituents and especially iron oxide on the hydrogenation of coal. B. HLAVICA (*Brennstoff-Chem.*, 1929, 10, 201—203; cf. B., 1928, 629).—A low-ash "coal" prepared by heating starch at 300° in an autoclave yielded only 6.2% of oil on hydrogenation. The yield, however, was increased to 13.0% in the presence of 23.9% of zinc chloride. The ash of ordinary coals contains varying amounts of iron compounds, of which pyrites has an especially marked catalytic effect on the hydrogenation. The addition of ferric oxide to coal increases the oil yield on hydrogenation, its influence being greater with bituminous coals than with brown coals or lignites.

A. B. MANNING.

Bitumen and the water-soluble and pyridine-soluble constituents of some brown coals. H. STEINBRECHER (*Brennstoff-Chem.*, 1929, 10, 198—201; cf. B., 1927, 593).—A number of brown coals have been extracted successively with a benzene-alcohol mixture, water, and pyridine. Large variations were observed in the amounts of bitumen (6.3—36.6%) and water-soluble ash (0.8—5.5%) which were extracted. The bitumen was resolved into a benzene-insoluble wax, an acetone-insoluble wax, and an acetone-soluble resin. There appeared to be a relation between the resin content of the coal and the explosibility of its dust. Considerable variations were observed in the acid, saponification, and esterification values of the bitumens. The water-soluble ash consisted of varying mixtures of sodium, calcium, magnesium, iron, and aluminium sulphates. The pyridine extracts were soluble in dilute alkaline solutions from which they were reprecipitated by acids; in chemical and physical properties they closely resembled the humic acids derived from brown coals. Treatment of the coals with pyridine reduced the ash content, partly by dissolution and partly by mechanical separation due to the swelling and consequent loosening of the material.

A. B. MANNING.

Importance of the low-temperature assay in the investigation of coking coals. P. DAMM (*Brennstoff-Chem.*, 1929, 10, 191—195, 217—221; cf. B., 1929, 382).—The coking process may be divided into three stages, the preheating stage, the plastic stage, and the final distillation. Fischer's aluminium assay apparatus has been used to study the changes occurring in each of these stages for a number of coals. In particular, the changes in caking index and swelling pressure on heating the coal at a temperature 25° below its softening point have been determined, and the cokes produced by carbonising the original coal and the preheated coal at 500° have been compared. Coals of the same oil and solid bitumen content and the same caking index may show marked differences in behaviour during the preheating period. The greater are the changes observed during this period the greater is the influence of the rate of heating on the coking process. The length of the temperature interval corresponding with the plastic stage has an important influence on the coke structure; the more is the volatile matter evolved during the plastic stage the less remains to be evolved during the final distillation and the less fissured is the

coke. No satisfactory method, however, has yet been devised to determine accurately the temperature corresponding with the end of the plastic stage.

A. B. MANNING.

Determination of sulphur in coal. N. A. NIKOLAI and N. VOROBIEV (*Izvestia Teplotekh. Inst. [Moscow]*, 1929, No. 3, 91—92).—Weighed samples of coal are burned in a calorimetric bomb in an atmosphere of oxygen under a pressure of 20—25 atm. The bomb contains also a few c.c. of a 10% solution of sodium carbonate to remove any oxides formed and to facilitate the subsequent separation of iron. After allowing to cool for 5—10 min. the residual gas is allowed to escape, the contents are poured out into a beaker, the bomb is washed several times with warm distilled water, and the combined washings are filtered, the sulphur being determined by Eschka's method. Comparative tables are given of the results obtained by Eschka's method, by the author's "bomb-washing" method, and by the sodium peroxide method. The results obtained by the first two methods agree fairly closely. A. FREIMAN.

Hardness and structure of coke. R. A. MOTT (*Fuel*, 1929, 8, 322—333).—The production of breeze during the transport of coke from the oven to the blast furnace takes place mainly when the coke is dropped into the wagons, hoppers, or into the furnace itself. The shatter test, which gives a relative measure of the liability of different cokes to form breeze in this manner, shows that the effect of one large drop is the same as that of a number of smaller drops from the same aggregate height. In successive drops through the same height, however, the breakage decreases. The shatter index may be expressed either as the percentage remaining on a 2-in. or on a 1½-in. mesh sieve. For normal cokes there is an approximately linear relationship between the two indices. In general, the 1½-in. index is to be preferred, but for research purposes it is recommended that both, and the 1-in. index as well, be determined. South Wales cokes exhibit the highest indices (average 96.6), Derbyshire cokes the lowest (average 69.7). A close relationship exists between the shatter index and the number and definition of the fractures in the coke shown by Rose's method of studying the structure (B., 1925, 834). Large-scale coking tests show that, in general, the hardness of the coke is improved by "top-charging" the coal instead of compressing it. A rapid rate of heating may be disadvantageous when coking a high-volatile strongly caking coal. The hardness of the coke increases to an extent with decrease in the size of the coal used; there is, however, no advantage in extremely fine grinding (100% through ¼-in. mesh). Crushed slack gives a harder coke than crushed large coal, and clean slurry a still harder coke. By blending 20—30% of low-temperature breeze or anthracite duff with a good coking coal the shatter index of the coke may be appreciably raised.

A. B. MANNING.

Flame speeds and their calculation. W. PAYMAN and R. V. WHEELER (*Fuel*, 1929, 8, 4—9, 91—98, 104—114, 153—162, 204—219).—The evidence for the "law of flame speeds" (B., 1922, 359 A) is collected and discussed in detail (cf. B., 1923, 42 A, 436 A, 437 A, 757 A;

A., 1926, 689; 1927, 317, 630). The method of application of the law to complex gaseous mixtures is described.

A. B. MANNING.

Rapid method of determining the maximum adsorption of benzene by activated charcoal. H. BURSTIN and J. WINKLER (Brennstoff-Chem., 1929, 10, 121—124).—The relationship $A_{max} = KQ$, between A_{max} , the maximum amount of benzene which a sample of activated charcoal will adsorb from a current of air saturated with the vapour, and Q , the heat of wetting of the charcoal by benzene, which may be deduced from theoretical considerations, has been verified experimentally. If Q is expressed in kg.-cal./g. and A_{max} as a percentage, the value of the constant K is 1.9. If, therefore, Q be determined A_{max} may be calculated. Q may be determined conveniently in a small, vacuum-jacketed vessel through the stopper of which passes a thermometer graduated in 0.1°. Benzene (10 c.c.) is introduced into the vessel, and the temperature, which is preferably adjusted to be about 20°, is read. Then 2 g. of the dry charcoal at the same temperature are added and the vessel is shaken for $\frac{1}{2}$ min., which is usually sufficient for the temperature to attain equilibrium. Q is calculated from the observed rise in temperature.

A. B. MANNING.

Industrial process for obtaining liquid hydrocarbons by simultaneous cracking and hydrogenation ["Semo" process]. J. FOHLEN (Chim. et Ind., 1929, 21, 1141—1148).—Cracked oils and hydrogen, without condensation, are led at ordinary pressure through purifiers at 300—250° to a series of reduced nickel catalysts maintained at 250—170° without external heating. Lignites, shales, and other poor or sulphurous starting materials give good results. The purifying agents are similar in composition to the catalyst used, and serve to remove all catalyst poisons; since the removal is an exothermic reaction there need be little loss of heat. In the initial catalyst vessels the temperature is sufficient to destroy any phenols present. A Greek lignite giving by cracking alone an oil of which only 52% distilled below 320° (23.5% below 220°) yielded by this "Semo" process a much lighter oil (94% below 300°, 74% below 220°, distillation beginning at 42°).

C. HOLLINS.

Air oxidation of hydrocarbons catalysed by nitrogen oxides. C. H. BIBB and H. J. LUCAS (Ind. Eng. Chem., 1929, 21, 633—638).—Natural gas containing 85% of methane was bubbled through concentrated nitric acid at 10° and then passed with 2.5—7.0 vols. of air through an electric furnace. Conversions of up to 5% of carbon into formaldehyde were obtained. The highest concentration of formaldehyde in the condensate (25%) was found with a furnace temperature of 750°, a time of passage of 0.13 sec., and low air-hydrocarbon ratio. The catalytic oxidation of benzene vapour was studied similarly. The exit gases were scrubbed with light lubricating oil to remove benzene and phenols, and the phenol was determined as tribromophenol. Conversions up to 5% were again obtained, but a smaller yield was necessary in order to avoid the formation of carbon dioxide. With a 3.5% conversion a yield of 52% of benzene as phenol was

obtained. In this case the nitric acid is best maintained at 40°. Theoretical reasons for the superiority of oxides of nitrogen over solid catalysts are discussed.

C. IRWIN.

Acids of montan wax. D. HOLDE, W. BLEYBERG, and H. VOHRER (Brennstoff-Chem., 1929, 10, 101—108, 124—128).—By fractional distillation under reduced pressure of the ethyl esters of the acids from montan wax, followed by fractional crystallisation of the acids themselves and fractional precipitation of their lithium salts, an acid $C_{28}H_{56}O_2$ (m.p. 84.4°) has been isolated in the pure state, and the presence of the iso-acid $C_{32}H_{64}O_2$ (m.p. 89°) and probably also of an acid $C_{30}H_{60}O_2$ has been established. No evidence was found of the presence of the acid $C_{29}H_{58}O_2$ (cf. Tropsch and Kreutzer, B., 1922, 659 A).

A. B. MANNING.

Decolorisation of cresols. RAKOVSKI and MEHL.—See III. **Absolute alcohol from sulphite-spirit.** KIRMREUTHER.—See V. **Dissociation of carbon monoxide.** HUBBARD and REES.—See VII. **Silica bricks.** REES and HUGILL. **Refractories for carbonisation plant.** DALE and others.—See VIII. **Saponification values.** NORMANN.—See XII.

PATENTS.

Pulverising apparatus for solid fuels etc. G. S. LOY (B.P. 300,561, 14.11.28. Fr., 14.11.27. Addn. to B.P. 190,132; B., 1923, 917 A. Cf. also B.P. 234,366; B., 1925, 576).—The apparatus described in the main patent is modified in that the movable grinder is formed by a plate on one face of which hollow pins are so mounted that their free ends engage the stationary grinder. The face of the latter is provided with metallic wires arranged in the direction of the axis of rotation.

A. B. MANNING.

Coke oven by-product recovery system. BARRETT Co., Assees. of S. P. MILLER (B.P. 298,153, 6.9.28. U.S., 4.10.27).—The hot coke-oven gases are subjected to a regulated cooling, e.g., by sprays of ammonia liquor in the goose-necks and collector main, to such a temperature that a pitch of a desired m.p. separates from the gas, which, while being maintained at the same temperature, is scrubbed with hot pitch of similar composition to that carried in suspension in the gas. The residual oils in the gas are subsequently recovered therefrom in a suitable condensing system.

A. B. MANNING.

Burning of fuel. T. M. CHANCE (U.S.P. 1,716,815, 11.6.29. Appl., 1.2.27).—Before being burned, the coal is mixed with the unfused portion of the ash, to reduce clinkering, this ash being recovered after each combustion.

F. G. CLARKE.

Distillation of wood. R. H. TWINING, Assr. to CLEVELAND-CLIFFS IRON Co. (U.S.P. 1,716,745, 11.6.29. Appl., 12.12.24).—Wood is distilled in a retort and the issuing vapours are subjected to the countercurrent scrubbing action of hot, crude pyroligneous acid from such distillation and from which tar has been settled.

H. S. GARLICK.

Manufacture of carbon black. R. H. UHLINGER, Assr. to THERMATOMIC CARBON Co. (U.S.P. 1,707,775, 2.4.29. Appl., 12.5.26).—The mixture of gases and

carbon black from the decomposing furnace is subjected to a spray of cooling liquid, and thence passes into separating chambers where the deposited carbon black is freed from moisture by conveying it in counter-current with the mixture of gases and carbon black leaving a decomposing furnace. R. BRIGHTMAN.

Production of hydrogen and carbon by thermal decomposition of hydrocarbons. A. W. BURWELL, Assr. to ALOX. CHEM. CORP. (U.S.P. 1,717,354, 18.6.29. Appl., 22.8.27).—After a preliminary heat-treatment, the vaporised hydrocarbons enter the decomposition zone of a cyclic stream of heated inert gas. The gaseous products containing hydrogen and carbon pass through a settling chamber for the removal of the latter, and the portion of the gas stream in excess of that required for re-use is then withdrawn from the cycle. The remainder is heated to at least 1200° and introduced into the decomposition zone. F. G. CLARKE.

Production of water-gas. C. W. ANDREWS and H. A. BRASSERT (U.S.P. 1,701,253, 5.2.29. Appl., 15.7.22).—Carbonaceous material, *e.g.*, coke, is air-blasted to the desired temperature, a layer of coal is placed on the heated residue, and the whole is blasted with steam, the coal layer being agitated. The process is continued with air-blasting before each addition of coal. R. BRIGHTMAN.

Production of oil-gas. F. T. NEWITT, S. H. LA PLANT, and L. I. TURNER, Assrs. to L.T.N. MANUFACTURING & DEVELOPMENT SYSTEM (U.S.P. 1,701,892, 12.2.29. Appl., 11.3.24).—Oil, *e.g.*, gasoline, is atomised by opposition discharge into a larger steam-discharge nozzle, provided with control valve and baffle plate, the oil-gas being drawn off continuously through a pipe provided with check valve, and used in part to generate the steam. R. BRIGHTMAN.

Removing readily absorbed gases, more particularly carbon dioxide and hydrogen sulphide, from gas mixtures by absorption in water under pressure. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 286,622, 5.3.28. Ger., 5.3.27).—The gas is washed in two towers in succession. In the first the hydrogen sulphide is almost completely removed by washing under pressure with part of the water, saturated with carbon dioxide etc., withdrawn from the second tower, in which the carbon dioxide is removed from the gas by washing with regenerated fresh water. The water from the first tower is regenerated by reducing the pressure and, if necessary, subsequently removing the last traces of hydrogen sulphide by a chemical treatment, *e.g.*, with ferric oxide or sulphur dioxide; or, after permitting the pressure to fall to normal, the residual hydrogen sulphide may be removed by passing a current of a gas rich in carbon dioxide through the water. The hydrogen sulphide in the gases so recovered is converted into sulphur, *e.g.*, in a Claus furnace. To prevent corrosion of the metal of the apparatus a small proportion of an alkaline carbonate is added to the water. A. B. MANNING.

Gas-purification solution and process. W. H. HILL and D. L. JACOBSON, Assrs. to KOPPERS Co. (U.S.P. 1,700,982, 5.2.29. Appl., 13.7.25).—In a cyclic process

for purifying gas from hydrogen sulphide, humic substances are added to the washing liquid to accelerate the oxidation of the iron sulphides etc. in regenerating the iron oxide. *E.g.*, filtered solution (300 gal.) obtained by extracting ground peat with an equal volume of 9% sodium carbonate solution are added to 9% sodium carbonate solution (100 gal.), and freshly precipitated ferric compounds are added to give a suspension containing up to 1% of ferric oxide. R. BRIGHTMAN.

Production of water-gas and extraction of oil from oil shale. A. SCHILLING, R. SACHSE, D. LIAMIN, and T. CALLAERT (U.S.P. 1,716,667, 11.6.29. Appl., 26.8.26).—A confined charge of oil shale is ignited at its upper surface, and steam and combustion gases are drawn downwardly through the shale, decomposition setting in to produce water-gas and hydrocarbon vapours. The water-gas is used to generate the steam required, whilst released oil is collected and the hydrocarbon vapours are condensed. Air for combustion is admitted to the upper surface of the ignited shale in quantity automatically controlled in accordance with the temperature of the water-gas. H. S. GARLICK.

Treatment of bituminous material. W. H. HAMPTON (U.S.P. 1,707,759, 2.4.29. Appl., 17.12.19).—Bituminous shale is crushed, mixed with preheated kerosene or other light mineral oil, and ground. The fluid mixture is conveyed through a series of digesters of increasing temperature up to, *e.g.*, 340–360°, in the final digester, which is maintained at 100 lb./in.² or less. Vapours from the digesters are drawn by a steam injector into cracking coils at about 700°, the cracked vapours being condensed and fractionated. The residual digestion mixture is discharged through a heat exchanger to a centrifuge, and the solid matter is washed with light solvent, dried, and discarded or returned for re-treatment. The oil and washings from the centrifuge are circulated through the heat exchanger and fractionated by discharge through a rose into a topping tower, vapours from which pass to the fractional condensing columns, while the heavier oil from the base of the tower may be circulated to the oil supply tank for mixing with fresh bituminous material. R. BRIGHTMAN.

Apparatus for conversion of hydrocarbons. L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,716,136, 4.6.29. Appl., 23.2.24).—A still, arranged in a furnace, has a series of conduits extending along its exterior lower surface connected to similar conduits extending along its inner lower surface. Steam supplied to the former passes on through the inner conduits, which are so perforated that the steam is directed against the lower interior surface of the still, whereby direct application of extremely hot combustion gases and consequent coke deposition is avoided. H. S. GARLICK.

Apparatus for treating [cracking] hydrocarbons. L. DE FLOREZ, Assr. to TEXAS Co. (U.S.P. 1,715,643, 4.6.29. Appl., 8.7.19. Renewed 6.7.28).—The apparatus comprises a heating chamber containing a series of tubes in which the oil is cracked, the tubes in the hotter zones of the chamber being of smaller cross-section than those in the cooler so as to increase the velocity of the vapours through the hot zones and thereby prevent

deposition of carbon in the tubes. This velocity is further increased by passing back a quantity of the gaseous products of cracking into the wider tubes of the apparatus; this procedure also reduces the proportion of heavy hydrocarbon oil converted into gaseous hydrocarbons.

A. R. POWELL.

Apparatus for chemically treating natural oils.

J. L. TAYLOR and C. L. HOWSER (U.S.P. 1,717,744, 18.6.29. Appl., 26.10.26).—The treatment liquid from a supply tank is mixed with the oil as it passes through a manifold on its way to a boiler. The supply tank is provided with independent air and gas conduits; the treated mixture passes from the boiler to a settling tank.

F. G. CLARKE.

Apparatus for treating [cracking] hydrocarbon oils.

J. B. WEAVER, Assr. to GYRO PROCESS CORP. (U.S.P. 1,708,247, 9.4.29. Appl., 22.7.25).—Oil is passed from a pipe still at, *e.g.*, 370° and 25 lb./in.², into an expander, vapours from which pass on, *e.g.*, at 320°, into the header of a converter, heated to 600°. The converter tubes are filled with ferric oxide or other agent, and residues from the expander are injected with pressure steam. Vapours from the converter are cooled to 230°, the vapours are scrubbed and condensed, and the liquid is returned to the pipe still. R. BRIGHTMAN.

Cracking of hydrocarbon oils. E. W. ISOM, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,708,180, 9.4.29. Appl., 21.5.27).—Hydrocarbon oil is circulated through a heating coil to and from a main supply tank at, *e.g.*, 400–425°. Vapours from the tank pass through two reflux condensers in series; condensate from the first may be returned to the circulating oil or mixed with condensate from the second condenser and heavier feed oil, preheated, and circulated through a secondary coil at, *e.g.*, 540–665°, and a tar trap to discharge from a perforated pipe into the main supply tank. Gas oil or heavy gas oil or fuel oil may be supplied direct to the supply tank, and light gas oil or kerosene to the condensate entering the secondary cracking tube.

R. BRIGHTMAN.

Treating [cracking] hydrocarbon oils.

G. B. BOGART, Assr. to TEXAS CO. (U.S.P. 1,709,304, 16.4.29. Appl., 17.2.27).—The hydrocarbon oil is fed into the heating coil and vapours from the cracking stills are discharged to a fractionating column above which is mounted a reflux condenser. Reflux condensate is returned to the column, partly direct, partly after cooling in a coil, and the phlegms from the column are circulated with fresh oil through the heating coil. R. BRIGHTMAN.

Distillation and cracking of hydrocarbons, particularly mineral oils and tars. H. MAGNUS (B.P. 287,525 and Addn. B.P. 313,937, 14.3.28. Ger., [A] 23.3.27).—(A) The process is effected continuously at raised temperatures and pressures in two or more vessels so combined that the liquid constituents and vapours formed in each vessel are separately withdrawn and fractionated in a condensing plant, and the valuable fractions are withdrawn while the remaining fractions are fed into the next vessel in the form of vapour or liquid to be subjected therein to further treatment under higher pressure. The flow created by the excess

of pressure in the reaction vessels relatively to that in the cooling chambers is utilised for generating energy, the gases, vapours, and liquids being allowed to expand either singly or together in a turbine which directly or indirectly furnishes the power required for forcing the fractions to be further treated into the next vessel at higher pressure. (B) The preheated raw material is led to a distributing ring mounted in the distillation vessel and having nozzles arranged in a body of molten metal which partly fills the vessel and serves to transmit heat from an oil or gas burner. The nozzles are arranged in a tangential direction and serve, together with circulating channels provided in the wall of the vessel which may be covered by a cylindrical insert extending over a great part of the length of the channels or by separate covers, to keep the metal both in a rotary and in a vertical circulation, the rate of which can be increased by injecting carrier gases through some of the nozzles and providing spirally-formed circulation channels. Electrical contact devices are provided so that, if molten metal escapes the feed supply and new material, valves are automatically closed and cooling air or gases or fluids preventing combustion are driven through the furnace.

H. S. GARLICK.

Treating [cracking] fluid hydrocarbons.

F. B. FRETTER, Assr. to NAT. REFINING CO. (U.S.P. 1,707,606, 2.4.29. Appl., 18.6.25).—The hydrocarbon oil is supplied to the base of a condensing tower, provided with baffles, where it is heated by mixture with the phlegms, and the mixed oil is pumped rapidly at a higher pressure, *e.g.*, above 1000 lb./in.², through a tubular heater at, *e.g.*, 425° or above. The pressure is released through a Venturi tube or other accelerating vent, operated by a needle-valve, which discharges the heated oil into a vaporiser, substantially at atmospheric pressure, and the cracked vapours escape through a dome and wide pipe to the condensing tower.

R. BRIGHTMAN.

Obtaining light hydrocarbons from solid or liquid fuels.

P. GIRARD, F. PETIT, and A. CHARBONNEAU (B.P. 299,861, 16.5.28. Fr., 3.11.27).—The raw material is distilled at 300–650° in the presence of an oxidising agent, *e.g.*, potassium permanganate, manganese dioxide, potassium dichromate, and the vapours are subjected to a high-frequency oscillatory discharge before being condensed.

H. S. GARLICK.

Apparatus for treatment of mineral oils.

W. F. DOWNS (M. G. DOWNS, adtrix.) (U.S.P. 1,716,372, 11.6.29. Appl., 24.11.22).—A still, suitably heated, is fitted with a paddle near to the bottom for causing an upward circulation of solid matter tending to settle, and a separate beater mechanism above the paddle for breaking up any such solid matter which forms as a result of distilling a mixture of mineral oil and aluminium chloride with which the still is charged. The vapours are maintained at a temperature above the volatilising point of aluminium chloride while being led to a condenser having a filter bed therein.

H. S. GARLICK.

Preparation of products with a high benzene content from those with a low content. A. ORT (F.P. 629,481, 17.1.27. Ger., 18.1.26).—In recovering benzene from wash oils used in scrubbing coal gas, the oil is heated at 200° in the later stages with steam under

pressure in jacketed vessels. The condensed water from these vessels has a temperature of 150–160° under 6–7 atm. and its heat content may be utilised for the indirect heating of fresh quantities of oil saturated with benzene.

A. R. POWELL.

Motor fuel and its manufacture. L. KIRSCHBRAUN (U.S.P. 1,701,620, 12.2.29. Appl., 8.1.20. Renewed 5.7.28).—Pressure distillate, *d* 0.7692, obtained, *e.g.*, from Kansas gas oil, *d* 0.875, by cracking at 400° and 90 lb./in.², after fractionation from tar if necessary, is emulsified with naphthenic acid and less than 15% of water to give fuel for internal-combustion engines.

R. BRIGHTMAN.

Oil-treating process and apparatus. J. R. HALL (U.S.P. 1,718,141, 18.6.29. Appl., 25.5.27).—A centrifugal drum set with its axis horizontal is divided by a vertical partition, and comprises two spiral passages which are connected only by a port at the periphery. Crude oil, forced into one trunnion, enters one of the spiral passages, heavy impurities are ejected at the periphery, through valves which are actuated automatically at a predetermined pressure, and the clear oil passes through the second spiral passage to the other trunnion.

F. G. CLARKE.

Simultaneously separating mixed liquids of different b.p. and generating power. C. F. HIRSHFELD (U.S.P. 1,716,130, 4.6.29. Appl., 6.3.22).—Mixed vapours, *e.g.*, those from steam-distilled oil shales, are expanded in a turbine or other engine, and liquid fractions are taken off at various stages.

B. M. VENABLES.

Apparatus for drying, charring, and otherwise treating loose material. O. DOBBELSTEIN (U.S.P. 1,718,542—4, 25.6.29. Appl., [A] 12.12.24, [B] 6.12.26, [C] 24.10.28. Ger., [A—C] 18.12.23).—See B.P. 226,543; B., 1925, 655.

Purification of coke-oven gases and the like. G. CLAUDE, Assr. to L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (U.S.P. 1,717,761, 18.6.29. Appl., 4.11.26. Fr., 17.11.23).—See B.P. 224,863; B., 1925, 308.

Apparatus for manufacture of water-gas. H. NIELSEN and B. LAING, Assrs. to SENSIBLE HEAT DISTILLATION, LTD. (U.S.P. 1,718,830, 25.6.29. Appl., 20.9.28. U.K., 28.5.27).—See B.P. 299,485; B., 1929, 8.

Doors of horizontal ovens for producing gas, coke, etc. E. WOLFF (B.P. 314,281, 19.9.28).

Gas or vapour burners. I. TURNER (B.P. 314,721, 7.9.28).

Filtering material (U.S.P. 1,702,104).—See I. **Purification of sulphur** (B.P. 314,697). **Gaseous mixtures containing hydrogen** (B.P. 288,577).—See VII. **Carbon electrodes** (B.P. 294,176).—See VIII.

III.—ORGANIC INTERMEDIATES.

Decolorisation of cresols by humic acids. W. RAKOVSKI and P. MEHL (Brennstoff-Chem., 1929, 10, 221—222).—By refluxing crude cresols with 20% of humic acids and then distilling, a colourless product is obtained which does not change on exposure to light

for long periods. The humic acids may be prepared from peat, or peat itself from which the bitumens have been extracted may be used. From the tar acids of a low-temperature peat tar purified in this manner an appreciable yield of crystalline phenol has been obtained.

A. B. MANNING.

Determination of sulphuric acid in a mixture of sulphuric acid, acetic acid, and acetic anhydride. T. SOMIYA (J. Soc. Chem. Ind., Japan, 1928, 31, 306—310).—When barium acetate is dissolved in acetic acid containing a small percentage of acetic anhydride, and the resulting solution used as a standard, the sulphoacetic acid can be accurately titrated thermometrically in the presence of acetic anhydride. The standardisation of the barium acetate solution can be carried out thermometrically by titrating against sulphuric acid dissolved in acetic acid solution, which serves equally well whether it contains acetic anhydride or not.

Y. NAGAI.

Benzathrones. J. MARTINET and A. DROBATSCHEV (Chim. et Ind., 1929, 21, 227—241, 1149—1160).—A review and bibliography of benzanthrone and its derivatives.

C. HOLLINS.

Air oxidation of hydrocarbons. BIBB and LUCAS.—See II. **Absolute alcohol from sulphite-spirit.** KIRMREUTHER.—See V.

PATENTS.

Manufacture of aldehydes and alcohols [from carbon monoxide and hydrogen]. G. T. MORGAN and R. TAYLOR (B.P. 313,061, 28.2.28. Cf. Morgan, Taylor, and Hedley, B., 1928, 439).—To the mixed zinc-chromium or zinc-manganese oxide catalysts for hydrogenation of carbon monoxide, cobalt, with or without copper, in reducible form is added. This catalyst is selected as giving a high yield of ethyl alcohol.

C. HOLLINS.

Purification of alcohols obtained by the catalysed interaction of hydrogen with oxides of carbon. A. CARPMAN. From I. G. FARBENIND. A.-G. (B.P. 311,468, 16.2.28).—The crude alcohol is stirred with a small amount of an oxidant (*e.g.*, 0.15% of permanganate in water) and fractionated, with or without the addition of water and/or alkali or zinc chloride. The addition of organic bases, such as *m*-phenylenediamine, aminophenols, or phenylhydrazine, after the oxidation is advantageous.

C. HOLLINS.

Manufacture of acetaldehyde from acetylene or gaseous mixtures containing it. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,716, 10.3.28).—Formation of resinous matter when working at raised temperatures (70–90°) is avoided by using as catalyst for the conversion of acetylene into acetaldehyde an aqueous solution of an alkali hydrogen sulphate containing a mercury compound and a compound of a metal of group I or VIII; *e.g.*, aqueous potassium hydrogen sulphate (100 g. SO₄ per litre) with mercuric sulphate (50 g.) and copper sulphate (4 g.) gives 95% conversion.

C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. H. DREYFUS (B.P. 313,418, 10.1.28).—Acetic acid vapour is mixed with sulphur dioxide (5–10%) and

passed through packed tubes at 450–600°, water being removed from the resulting vapours by means of sodium hydrogen sulphate at 150–200°. C. HOLLINS.

Manufacture of aliphatic [acetic] acid anhydrides. BRIT. CELANESE, LTD., and S. J. GREEN (B.P. 312,587, 28.1.28).—The (acetic) acid vapour is passed at 550–650° over a carbon catalyst prepared by charring a solution of cane sugar with or without the addition of tungstic acid, phosphoric acid, tungstates other than alkali tungstates, or sodium phosphate. C. HOLLINS.

Manufacture of aliphatic [acetic] acid anhydrides. H. DREYFUS (B.P. 312,585–6 and 312,733, [A, B] 28.1.28, [c] 21.3.28. Addn. [c] to B.P. 280,972; B., 1928, 82).—The thermal decomposition of (acetic) acid vapours is performed in presence of (A) 1–10 pts. of carbon dioxide, (B) 1–10 pts. of carbon monoxide or mixtures of these, (C) a catalyst comprising a phosphoric acid on a carrier of carbon, charcoal, or carborundum. C. HOLLINS.

Manufacture of anhydrous acetic acid from its aqueous solutions. I. G. FARBENIND. A.-G. (B.P. 284,588, 30.1.28. Ger., 29.1.27).—Dilute (e.g., 40%) acetic acid is distilled with ethyl acetate. An azeotropic mixture of ester and water distils at 69–70° and separates in the receiver; the ester layer may be returned to the dilute acid until all water has been removed. Ester is then distilled off, leaving glacial acetic acid. C. HOLLINS.

Concentration of aqueous acetic acid. I. G. FARBENIND. A.-G. (B.P. 294,626, 16.2.28. Ger., 29.7.27. Addn. to B.P. 284,588; preceding).—In the process of the prior patent the acetic ester is used in conjunction with a hydrocarbon (benzene) or a halogenated hydrocarbon (ethylene dichloride). C. HOLLINS.

Production of [aliphatic] organic acids. W. J. HALE and W. S. HALDEMAN (B.P. 287,064, 12.3.28. U.S., 12.3.27).—An alcohol of b.p. below 350° is passed in vapour form in countercurrent with a catalyst consisting of a metal oxide reducible by the corresponding aldehyde. The alcohol meets reduced metal at about 280° and is dehydrogenated to the aldehyde, which is later oxidised by the metal oxide to acid. Cuprous oxide containing 1% of silver is a suitable catalyst, and the process can be made continuous. C. HOLLINS.

Dehydration of vapour mixtures containing acetic anhydride and water. I. G. FARBENIND. A.-G. (B.P. 285,090, 10.2.28. Ger., 11.2.27).—A solvent (benzene, benzene and dichlorobenzene or quinoline) is introduced at the top (as liquid) and at the lower portion (as vapour) of a fractionating column through which the wet acetic anhydride vapours are passed. The anhydride is collected in a receiver kept warm to allow the water-benzene vapours to pass onwards. C. HOLLINS.

Manufacture of [alkoxy-]aliphatic acids. H. DREYFUS (B.P. 313,651, 16.3.28).—Dimethyl ether is converted into methoxyacetic acid, diethyl ether into β -ethoxypropionic acid, by passing it with excess of carbon dioxide at 100–200 atm. over a catalyst (phosphoric acid on coke or graphite) at 300–400°. Other catalysts known to catalyse the formation of acetic acid

from carbon monoxide and methyl alcohol may be used. C. HOLLINS.

Manufacture of anhydrides of alkoxy-aliphatic acids. H. DREYFUS (B.P. 313,233–5, 24.3.28).—An alkali salt of an alkoxy-aliphatic acid (especially methoxyacetic acid) is caused to react with (A) sulphur chloride or sulphur and chlorine, (B) carbonyl chloride, or (C) sulphuryl chloride or a sulphur oxide and chlorine; in each case the acid anhydride may conveniently serve as diluent. C. HOLLINS.

Manufacture of metal alcoholates [alkoxides]. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 304,585, 5.11.28. Ger., 21.1.28).—Sodium hydroxide, dry or as 40% aqueous solution, is distilled under a fractionating column with an alcohol having more than 3 carbon atoms until no more water distils over. C. HOLLINS.

Production of esters from acid amides. H. G. SMITH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 313,316, 12.6.28).—An acid amide (formamide, acetamide) and an alcohol are heated with concentrated sulphuric acid. Esters boiling below the b.p. of the alcohol used may be distilled off as formed. C. HOLLINS.

Production of mono- or poly-hydric alcohols. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,783, 2.12.27. Addn. to B.P. 309,200; B., 1929, 549).—In the process of the prior patent the condensing agent is removed before hydrogenation; preferably a condensing agent is used which is insoluble or can be converted into an insoluble form, or, alternatively, the reaction product is extracted with a solvent which does not dissolve the condensing agent. C. HOLLINS.

Purification of synthetic butyl alcohol. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,512, 20.6.28).—Butyl alcohol from the hydrogenation of synthetic crotonaldehyde or butaldehyde is treated with chlorine, bromine, hypochlorous or hypobromous acid to oxidise impurities, made alkaline, and distilled. C. HOLLINS.

Manufacture of gaseous and readily volatile olefines from hydrogen and oxides of carbon. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,999, 6.2.28).—In the hydrogenation of carbon oxides to ethylene etc. the temperature of the catalyst is maintained uniformly at, e.g., 360–370°, and deposition of carbon is thus avoided, by disposing the catalyst in grooves or slots in a metal container through which a conducting fluid is circulated. The container may, for example, be a copper spiral tube carrying dry steam, at 360°; or the superheated steam may be circulated through tubes bored in copper blocks provided with gilled shelves for the catalyst. C. HOLLINS.

Manufacture of 1:3[γ]-butylene glycol. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,671, 9.2.28).—Aldol is smoothly reduced to γ -butylene glycol by passing the vapour, freed from acetaldehyde, with hydrogen at 100 atm. over a nickel catalyst containing a little alumina at 110°. C. HOLLINS.

Polymerisation of olefines. F. HOFMANN, M. OTTO, and W. STEGEMANN (B.P. 313,067, 5.3.28. Cf. B.P.

293,487; B., 1928, 664).—Olefines are polymerised by boron trifluoride in presence of water, halogen acids, or halogenated hydrocarbons capable of reacting with benzene etc. in the Friedel-Crafts reaction. Examples are: ethylene, propylene, and cyclohexene treated with boron trifluoride and water, hydrogen fluoride, hydriodic acid, ethyl fluoride, or chloride. C. HOLLINS.

Production of aldol and crotonaldehyde. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 313,466, 6.2.28).—Aqueous acetaldehyde is condensed to aldol at 30° in presence of not more than 0.00001N-alkali; oxygen is excluded, and preferably a buffer salt (sodium acetate) is added. Liquor is withdrawn from time to time (fresh aldehyde being added), acidified with dilute sulphuric acid, and distilled; acetaldehyde vapours are returned to the reaction vessel, and the crotonaldehyde distils with 26% of water. C. HOLLINS.

[Catalysts for] carrying out organic dehydration reactions. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,426, 8.3.28).—Oxides of metals of groups I, VI, VII, and VIII (e.g., copper, chromium, manganese, nickel, cobalt) are deposited on non-vitreous alumina and used with or without a carrier to catalyse dehydration reactions, such as dimethyl ether from methyl alcohol, isoprene from methylbutenol, butylene from butyl alcohol. Dehydrogenating catalysts (e.g., nickel, copper, silver, or sulphides, selenides, phosphides of these) may be used in addition to or in place of these metal oxides. C. HOLLINS.

Manufacture of maleic acid and anhydride. A. BOEHRINGER (C. II. BOEHRINGER SOHN) (B.P. 297,667, 8.2.28. Ger., 24.9.27. Addn. to B.P. 285,426; B., 1929, 636).—In the process of the prior patent a large excess (20—100 times theoretical) of air at 180° is used. C. HOLLINS.

Catalytic removal of hydrogen- or oxygen-containing groups from organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 306,884, 14.1.28. U.S., 27.2.28).—In organic catalytic reactions, such as the conversion of alcohol into acetaldehyde, isopropyl alcohol into acetone, cyclohexanol into cyclohexanone, alcohols into olefines, phthalic acid into benzoic acid, etc., the temperature control is made independent of variations in rate of flow of the gases by means of a double countercurrent heat-exchange system in conjunction, if desired, with the usual devices. The entering gases pass down the central portions of a series of double tubes, up through the outer annular spaces, and finally down through catalyst masses packed round the outside of the double tubes. C. HOLLINS.

Carrying out catalytic molecular association [condensation] of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 306,883, 14.4.28. U.S., 27.2.28).—The double countercurrent heat-exchange described in B.P. 306,884 (preceding) is applied to vapour-phase esterifications, condensations (e.g., aldolisations, crotonisations), polymerisations, production of hydrogen cyanide from carbon monoxide and ammonia, etc. C. HOLLINS.

Production of aromatic aldehydes. J. D. RIEDEL A.-G. (B.P. 290,649, 26.1.28. Ger., 20.5.27. Addn. to

B.P. 285,451; B., 1928, 873).—In the process of the prior patent nitrotoluenes or other homologous nitrobenzenes are used as oxidants. Since only *p*-propenylphenols are oxidisable by nitro-compounds, mixtures with *m*-isomerides may be used as starting materials. C. HOLLINS.

Manufacture of chlorinated compounds of the benzene series. A. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 313,207, 9.3.28).—*m*-Xylene-4-sulphonic acid is mono-, di-, or tri-chlorinated in aqueous or acid solution with chlorine in presence of a carrier (iodine or ferric chloride), or with hypochlorite. The sulphonic acid group may subsequently be removed by hydrolysis with sulphuric acid and superheated steam. The following are described: 2-chloro-*m*-xylene-4-sulphonic acid (amide, m.p. 223°), and 2-chloro-*m*-xylene, b.p. 185—187°; the 2 : 6-dichloro-sulphonic acid (amide, m.p. 330°) and 2 : 6-dichloro-*m*-xylene; 2 : 5 : 6-trichloro-*m*-xylene-4-sulphonic acid (amide, m.p. above 325°) and 2 : 5 : 6-trichloro-*m*-xylene, m.p. 95—96°, b.p. 255—260°. Chlorination of the sulphonic acids with aqua regia, or chlorate and hydrochloric acid, gives 2 : 4 : 5 : 6-tetrachloro-*m*-xylene, m.p. 218—220°. C. HOLLINS.

Production of benzoic acid derivatives [reduction of halogenated nitrobenzoylbenzoic acids]. H. W. HERREWARD, L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 311,465, 16.2.28).—Halogenated nitrobenzoylbenzoic acids, particularly 2-(*p*-chloro-*m*-nitrobenzoyl)benzoic acid, are reduced smoothly without loss of halogen by iron and water at 75°, especially in presence of a little inorganic salt (sodium chloride). C. HOLLINS.

Purification of phthalic anhydride. SELDEN Co., Assees. of A. O. JAEGER and F. A. CANON (B.P. 285,017, 8.2.28. U.S., 8.2.27).—Unsaturated impurities in crude phthalic anhydride are polymerised, especially by heating under pressure in presence of manganese dioxide on pumice fragments, and pure phthalic anhydride is then distilled or sublimed. C. HOLLINS.

Manufacture of indophenols. I. G. FARBENIND. A.-G. (B.P. 285,382, 13.2.28. Ger., 14.2.27. Addn. to B.P. 286,005; B., 1928, 740).—The process of the prior patent is extended to polynuclear heterocyclic bases hydrogenated in the heterocyclic ring, e.g., hexahydrocarbazoles, tetrahydroquinolines, octahydro- α -naphthaquinolines. C. HOLLINS.

Manufacture of [intermediate] compounds having affinity for cotton [ice and developing colours]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 313,636, 15.2.28).—Nitrobenzoyl derivatives of aromatic diaminesulphonic acids are reduced to azoxy- or azo-compounds, which on further reduction yield the corresponding amines or polyamines possessing affinity for cotton; they can be coupled on the fibre or diazotised on the fibre and coupled with developers. The azoxy- or azo-compound from *p*-nitrobenz-(4-amino-3-sulphoanilide) is diazotised on the fibre and developed with phenylmethylpyrazolone for a greenish-yellow. 1-*p*-Nitrobenzamido-4-formamidobenzene-2-sulphonic acid gives a similar product, the formyl group being removed by hydrolysis after the reduction. Other suitable starting materials are bis-*p*-nitrobenzoyl-*p*-phenylene-

diaminesulphonic acid, the *m*-nitrobenzoyl compound, 4:4'-bis-*p*-nitrobenzamido-3:3'-disulphodiphenylcarbamide, bis-*p*-nitrobenzoyl-1:4-naphthylenediamine-6-sulphonic acid, 4-*p*-nitrobenzamido-*o*-toluidine-5-sulphonic acid, and 5-*p*-nitrobenzamido- α -naphthylamine-3:6-disulphonic acid
C. HOLLINS.

Manufacture of di(halogenoacylated) diphenyl ethers and of highly active therapeutic substances therefrom. SCHERING-KAHLBAUM A.-G. (B.P. 286,688, 16.2.28. Ger., 9.3.27).—Diphenyl ether, or a hydroxy- or alkoxy-derivative thereof, is condensed with chloro- or bromo-acetyl chloride (2 mols.); the halogen may subsequently be replaced by an amino- or alkylamino-group to yield therapeutic compounds. 4:4'-*Di*(chloroacetyl)diphenyl ether, m.p. 102°, the *di*(bromoacetyl)-compound, m.p. 121°, 2-methoxy-4:4'-*di*(chloroacetyl)-diphenyl ether, m.p. 148°, the 2-hydroxy-compound, m.p. 158°, 2-hydroxy-4:4'-*di*(methylaminoacetyl)diphenyl ether, and 4-hydroxy-2:4'-*di*(chloroacetyl)diphenyl ether, m.p. 155°, are described.
C. HOLLINS.

Manufacture of new intermediates [arylamides of 2:3-hydroxynaphthoic acid] and azo dyes therefrom. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 311,194, 4.1.28).—2:3-Hydroxynaphthoic acid (2 mols.) is condensed by the method of B.P. 23,732 of 1913 (B., 1914, 70) with *p*-diamines carrying substituents in an *o*-position to each amino-group. Ice colours and pigments made from these diamides show increased fastness to light. The bis-2:3-hydroxynaphthoic acid derivatives of the following *p*-diamines are described: 4-chloro-*p*-tolylenediamine (m.p. 354–355°), 2:5-dichloro-*p*-phenylenediamine (m.p. 369–370°), 2:5-dimethoxy-*p*-phenylenediamine (m.p. 356–358°), 2:5-diamino-*p*-xylene (m.p. 364–366°), 4-chloro-2:5-diaminoanisole (m.p. 331–332°), 2:5-diamino-*p*-cresol methyl ether (m.p. 316–318°). Amongst the 66 couplings mentioned are: 2:5-dichloroaniline \rightarrow 4-chloro-2:5-diaminoanisole compound (brown); 4-chloro-*o*-anisidine \rightarrow 4-chloro-*p*-tolylenediamide (claret); 4-nitro-*o*-toluidine \rightarrow 2:5-diamino-*p*-tolyl methyl ether compound (garnet-brown); 5-chloro-*o*-toluidine \rightarrow 2:5-dichloro-*p*-phenylenediamide (full red); *m*-4-xylidine \rightarrow 4-chloro-*p*-tolylenediamide (dark red); *o*-phenetidine \rightarrow α -naphthylamine \rightarrow 2:5-diamino-*p*-tolyl methyl ether compound (black).
C. HOLLINS.

Recovery and purification of anthraquinone. IMPERIAL CHEM. INDUSTRIES, LTD., A. DAVIDSON, A. SHEPHERDSON, and J. THOMAS (B.P. 312,837, 28.6.28).—Superheated steam is passed at 180° into solutions of sulphonated anthraquinones in 80% sulphuric acid. The products from the α -sulphonation of anthraquinone, for example, are diluted to 80% H₂SO₄, filtered to remove α -sulphonic acid and anthraquinone, and the filtrate is steamed at 180°; pure anthraquinone sublimes in the steam.
C. HOLLINS.

Manufacture of 1-aminoanthraquinone-2-sulphonic acid. IMPERIAL CHEM. INDUSTRIES, LTD. A. DAVIDSON, W. W. TATUM, and G. E. WATTS (B.P. 311,977, 2.7.28).—In the bake process of B.P. 299,279 (B., 1929 47) improved yields are obtained when oxalic acid is added to the sulphate.
C. HOLLINS.

Production of sulphuric acid esters of amino-anthrahydroquinones [leuco-aminoanthraquinones]. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 312,243, 18.11.27).—An acylaminoanthraquinone, especially an anthraquinone-urethane, is treated with pyridine-sulphuric anhydride in presence of a metal (copper) and a tertiary base (pyridine), the resulting sulphuric ester being then hydrolysed with alkali to remove the acyl group. Suitable starting materials are 1- and 2-acetamido-anthraquinones, 2-anthraquinonylurethane, 3-chloro-2-acetamidoanthraquinone, and 4-bromo-1-acetmethylamidoanthraquinone.
C. HOLLINS.

Manufacture of halogenoaminoanthraquinones [1-chloro-5-aminoanthraquinone]. E. G. BECKETT, W. G. WOODCOCK, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 312,289, 21.11.27).—1:5-Dichloroanthraquinone is heated with aqueous ammonia in an iron autoclave at 170° for 6 hrs. to give a mixture of 1-chloro-5-amino-, 1:5-diamino-, and 1:5-dichloro-anthraquinones, which is dissolved in hot sulphuric acid. The solution is diluted to 80% concentration, filtered at 80° to remove dichloroanthraquinone, and on cooling deposits the pure sulphate of 1-chloro-5-aminoanthraquinone. Other dichloroanthraquinones may similarly be treated.
C. HOLLINS.

Manufacture of o-halogenoanthraquinonecarboxylic acids. I. G. FARBENIND. A.-G. (B.P. 297,001, 10.9.28. Ger., 9.9.27).—1-Aminoanthraquinone-2-sulphonic acid is diazotised and converted into the 1-cyano-compound, which is hydrolysed to the acid, and the sulphonic group exchanged for halogen in the usual manner or by treatment with free chlorine or bromine. The process is applied also to 3-aminoanthraquinone-2-sulphonic acid, 4-bromo-1-aminoanthraquinone-2-sulphonic acid, 1-bromo-2-aminoanthraquinone-3-sulphonic acid, and 2:6-diaminoanthraquinone-3:7-disulphonic acid, the last-named giving 3:7-dichloroanthraquinone-2:6-dicarboxylic acid, m.p. above 300°.
C. HOLLINS.

Organic phosphate and its manufacture. T. B. WAGNER (U.S.P. 1,716,286, 4.6.29. Appl., 10.11.27).—Steep-water obtained as a by-product in the manufacture of glucose from maize is boiled with kieselguhr to coagulate the protein, filtered, and exactly neutralised with milk of lime, whereby a mixture of calcium salts of complex organic phosphoric acids is precipitated.
A. R. POWELL.

Catalytic oxidation of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 281,307, 24.11.27. U.S., 24.11.26).—See U.S.P. 1,694,122; B., 1929, 550.

Cyclic ketonic compound and its manufacture. H. GREUNE, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,702,002, 12.2.29. Appl., 16.6.27. Ger., 24.6.26).—See B.P. 273,321; B., 1928, 887.

Manufacture of condensation products of the anthraquinone series. G. REDDELEIN and W. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,717,809, 18.6.29. Appl., 8.8.27. Ger., 4.8.26).—See B.P. 275,636; B., 1929, 164.

Base-exchange bodies (B.P. 286,212).—See VII. Carrying out photochemical reactions (B.P. 314,267).—See XI.

IV.—DYESTUFFS.

"Saftbraun" dyeing. SCHLATTER.—See V.

PATENTS.

Manufacture of [thioindigoid] vat dyes. I. G. FARBENIND. A.-G. (B.P. 284,288, 25.1.28. Ger., 27.1.27).—Arylthioglycolic acids are condensed with isatins etc. in a solvent (chlorobenzene) in presence of phosphorus pentoxide, preferably diluted with finely-divided silica. Examples are: 1-chloro- β -naphthylthioglycolic acid with isatin (claret-red); β -naphthylthioglycolic acid with 5:7-dibromoisatin (brown), 5-chloro-*o*-tolylthioglycolic acid with acenaphthaquinone (red).

C. HOLLINS.

Manufacture of new dyes [of the anthraquinone series for acetate silk; pigment dyes]. SOC. CHEM. IND. IN BASLE (B.P. 285,096, 11.2.28. Switz., 12.2.27).—A 1-hydroxy- or 1-alkoxy-4-aminoanthraquinone or a 1:4-diaminoanthraquinone is condensed with an aromatic diamine to give 4-amino-1-amino-anilinoanthraquinones, which are dyes for acetate silk or may be sulphonated with oleum, chlorosulphonic acid, or formaldehyde-bisulphite to yield wool dyes. 4-Amino-1-*p*-aminoanilinoanthraquinone, m.p. 247–248°, from 4-amino-1-methoxy(or -hydroxy)anthraquinone, gives greenish-blue shades on acetate silk, and on sulphonation with 7% oleum yields a greenish-blue wool dye. Sulphonated 4-amino-1-*p*-dimethyl-aminoanthraquinone gives greener shades.

C. HOLLINS.

Manufacture of [vat] dyes [of the dibenzanthrone series] and their application. SOC. CHEM. IND. IN BASLE (B.P. 284,656, 2.2.28. Switz., 2.2.27. Addn. to B.P. 262,774; B., 1928, 517).—Halogenated dibenzanthrones, other than those made in presence of chlorosulphonic acid or by means of ferric chloride or antimony pentachloride, are treated with chlorine in acetic acid at 130°. The products are tetra- or pentachlorodibenzanthrones, dyeing cotton a pure blue particularly fast to water.

C. HOLLINS.

Dyes and dyeing [sulphuric esters of leuco(?) 4:4'-dibenzanthronyls]. R. S. BARNES, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 312,093, 19.11.27).—A 4:4'-dibenzanthronyl, having the 2:2'-positions free, is treated with pyridine-sulphuric anhydride, copper, and pyridine, or is reduced to leuco(?) compound and esterified with chlorosulphonic acid and pyridine, to give a soluble leuco-ester which dyes textiles a bright yellow becoming reddish-blue by oxidation with nitrous acid etc.

C. HOLLINS.

Manufacture of vat dyes of the anthanthrone series. I. G. FARBENIND. A.-G. (B.P. 286,669, 1.3.28. Ger., 8.3.27).—Halogenated anthanthrones are condensed with aminoanthraquinones to give vat dyes. Examples are: 6:12-dibromoanthanthrone (from 4:4'-dibromo-1:1'-dinaphthyl-8:8'-dicarboxylic acid) with α -aminoanthraquinone (grey-blue) or β -aminoanthraquinone (brown), or 1-amino-4-benzamidoanthraquinone (green-

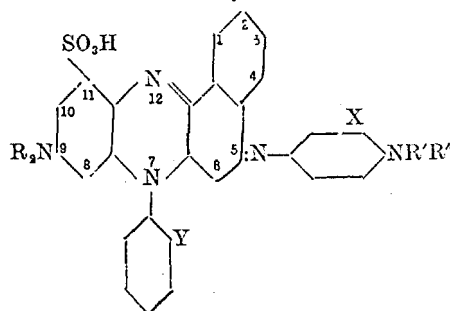
blue); commercial dibrominated anthanthrone with 1-amino-5-benzamidoanthraquinone (grey).

C. HOLLINS.

Manufacture of [vat] dyes of the anthanthrone series. L. CASSELLA & Co., G.M.B.H. (B.P. 304,613, 21.1.29. Ger., 21.1.28. Addn. to B.P. 260,998; B., 1928, 225).—The sulphonic groups in anthanthrone-sulphonic acids are replaced by halogen by the action of hydrochloric acid and chlorate or bromate, giving orange vat dyes. The sulphonic acids are prepared either by direct sulphonation of anthanthrone with oleum, or by synthesis from sulphonated dinaphthyl-dicarboxylic acids.

C. HOLLINS.

Manufacture of dyes of the phenonaphthasafranin series. J. R. GEIGY A.-G. (B.P. 285,486, 14.2.28. Ger., 19.2.27. Addn. to B.P. 265,986; B., 1928, 441).—*iso*Rosinduline dyes of the formula:



in which R and R' = alkyl, and R'' = hydrogen or alkyl, are improved in fastness to alkali and clearness of shade by sulphonation. The new sulphonic group enters the *p*-phenylenediamine nucleus *ortho* to the nitrogen occupying the 5-position, especially if this nucleus carries a methyl, alkoxy, or chlorine substituent in *meta*-position (X). In the examples, dyes of the above formula in which (a) R = Et, R' = Et, R'' = H, X = Me, with sulphonic groups at 11 and Y, and (b) R = Et, R' = Me, R'' = H, X = Me, with a sulphonic group at 10, are sulphonated with 20% oleum at 50–70° and 80–90°, respectively.

C. HOLLINS.

Prevention of settling out of paste dyes. HARDING CHEM. CO., LTD., and W. H. CLUTTERBUCK (B.P. 310,830, 1.2.28).—Vat dye pastes are mixed with less than 2% of a natural colloidal clay such as bentonite.

C. HOLLINS.

Manufacture of azo dyes [for wool and lakes]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 313,110, 7.3.28).—Azo dyes containing an aryloxyalkoyl-amino-group in either component are prepared; they show good fastness to light and fulling and are suitable for discharge printing on wool and for lakes. Examples are: aniline \rightarrow *N*-phenoxyacetyl-H-acid (pink) or *N*-*p*-chlorophenoxyacetyl-1:8:4:6-aminonaphthol-disulphonic acid (yellowish-red); *p*-nitroaniline-*o*-sulphonic acid \rightarrow *N*- β -aminoethyl- β -naphthylamine-7-sulphonic acid, acylated with *o*-chlorophenoxyacetyl chloride (red-violet); 4:6-dinitroaniline-2-sulphonic acid \rightarrow *N*- ω -*p*-toloxyacetamidooethyl-Cleve acid (reddish-blue); aniline-2:5-disulphonic acid \rightarrow *p*-xylydine \rightarrow *N*-*p*-chlorophenoxyacetyl-H-acid (clear violet); phenoxyacetyl-*p*-phenylenediamine \rightarrow 1-*p*-sulphophenyl-3-

methyl-5-pyrazolone (golden-yellow); phenoxyacet-*p*-aminoethylanilide \rightarrow 2-methylamino-8-naphthol (brown); *p*-nitroaniline \rightarrow 1-*p*-sulphophenyl-3-methyl-5-pyrazolone, reduced and acylated with phenoxyacetyl chloride (golden-yellow); phenoxyacetyl-*p*-phenylenediamine \rightarrow α -naphthol-3:6-disulphonic acid (yellowish-red). C. HOLLINS.

Manufacture of yellow monoazo dyes [for wool]. I. G. FARBERIND. A.-G. (B.P. 290,230, 10.5.28. Ger., 10.5.27).—Sulphanilic, metanilic, or orthanilic acid is diazotised and coupled with 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone to yield greenish-yellow dyes which give on wool level shades fast to light and water. C. HOLLINS.

Manufacture of azo dyes [ice colours]. I. G. FARBERIND. A.-G. (B.P. 295,289, 8.8.28. Ger., 8.8.27).—2:3-Hydroxynaphthoic arylamides are coupled in substance or on the fibre with diazotised 6-chloro-4-*m*-xylydine. Examples are the *p*-anisidine (bluish-red), *o*-anisidine (scarlet-red), 4-chloro-*o*-anisidine (bluish-red), 2:5-dimethoxyanilide (bluish-red). C. HOLLINS.

Manufacture of azo dyes [violet ice and pigment colours]. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 312,297, 21.2.28).—2:3-Hydroxynaphthoic arylamides give violet dyes when coupled in substance or on the fibre with diazotised 4-amino-6-arylamino-resorcinol dialkyl or diaralkyl ethers, obtainable, e.g., by partial reduction of 4:6-dinitroresorcinol ethers followed by arylation and reduction. 4-Amino-6-benzamidoresorcinol dimethyl or diethyl ether is coupled with 2:3-hydroxynaphthoic 4-chloro-*o*-toluidide, *o*- or *p*-toluidide, 2:5-dimethoxyanilide, *m*-nitroanilide, *o*- or *p*-anisidine, α - or β -naphthylamide, anilide, dianisidine (corinth shade), *o*-phenoxyanilide, 5-chloro-*o*-anisidine, *o*-, *m*-, or *p*-chloroanilide; 4-amino-6-anisamido- or -6-*p*-chlorobenzamido-resorcinol dimethyl ether with the *m*-nitroanilide; 4-amino-6-benzamidoresorcinol dibenzyl ether with the *m*-nitroanilide, 4-chloro-*o*-toluidide, or *o*-toluidide. C. HOLLINS.

Manufacture of [blue to violet] azo dyes insoluble in water. I. G. FARBERIND. A.-G. (B.P. 286,274, 2.3.28. Ger., 2.3.27).—Blue to violet azo dyes are obtained in substance or on the fibre by coupling a *p*-aminodiphenylamine, carrying alkyl and/or alkoxyl groups and/or halogen as substituents in one or both nuclei, with a 2:3-hydroxynaphthoic arylamide. Examples are: 4-amino-3:4'-dimethoxydiphenylamine \rightarrow 5-chloro-*o*-toluidide (greenish-blue); 4-amino-4'-methoxy-3-methylidiphenylamine \rightarrow α -naphthylamide (indigo-blue); tribromo-4-aminodiphenylamine \rightarrow α -naphthylamide (reddish-violet). C. HOLLINS.

Manufacture of substantive azo dyes. I. G. FARBERIND. A.-G. (B.P. 286,717, 1.3.28. Ger., 10.3.27).—Benzidine-3:3'-dicarboxylic acid is tetrazotised and coupled with components which confer solubility, one at least being an aryl-naphthylaminesulphonic acid; e.g., with 2 mols. of phenyl-S-acid (blue on cotton, greener after coppering), or with phenyl-J-acid and 1:5-naphtholsulphonic acid (reddish-violet, bluer on coppering), or with phenyl-S-acid and acetoacetic sulpho-*o*-anisidine (greyish-blue, becoming clear green on coppering). C. HOLLINS.

Azo dyes (B.P. 311,194).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Behaviour of cotton towards glycol and glycol-hydrochloric acid. B. RASSOW and F. WEBER (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 88—89).—The α -cellulose from cotton is not dissolved by heating it with glycol for 37 hrs. at 190° or by 4 hrs. heating in a bomb-tube at 210°; the fibres only swell and become more readily attackable by chemical reagents. The secondary ingredients, such as hemicelluloses and pentosans, which remain in the cotton after its usual purification treatment, are, however, dissolved under these conditions. If small amounts of hydrochloric acid are added to the glycol the removal of these impurities is accelerated, but the α -cellulose is soon attacked with the production of hydrocellulose. Microscopical examination of the fibres shows that they are split in directions perpendicular to the axis; this result is in agreement with recent theories of the structure and of the action of acids on cotton cellulose. Hydrocellulose produced by the glycol-hydrochloric acid mixture has a low copper number, which is explained by the fact that decomposition products formed during the hydrolysis are dissolved. The properties of the hydrocellulose products and the results of viscosity measurements indicate that the cellulose molecules are depolymerised by the action of glycol-hydrochloric acid mixtures on cotton. B. P. RIDGE.

Decomposition of straw with nitric acid. H. SUIDA, H. SADLER, and F. NOSS (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 71—79).—The effect of temperature, concentration of acid, and conditions of washing with sodium hydroxide etc. on the yield of cellulose has been investigated. Mild conditions of acid treatment must be chosen in order to give satisfactory results, and pretreatment of the straw with steam or hot water has no advantage over direct mixing of the material with acid at the correct temperature. Acid of 1—10% by wt. was used for times from about $\frac{1}{2}$ hr. to 3 hrs. at temperatures of 60° to 100°, and the material was washed with sodium hydroxide solution (about 2%) at 90° to remove residues of incrusting material. The yield of cellulose decreases linearly with increasing temperature of decomposition, independent of varying acid concentration. It also decreases with increasing acid concentration quickly up to 5% acid, and thereafter slowly up to 10% acid, whilst a further decrease takes place with increasing concentration of the sodium hydroxide used for washing. To a definite consumption of nitric acid there corresponds a definite concentration of acid, decomposition temperature, and yield of cellulose. B. P. RIDGE.

Strength of cellulose [pulp]. A. KÜNG and E. SEGER (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 96—102).—Strength tests on an unbleached Mitscherlich cellulose and on a bleached cellulose were carried out after different periods of milling (a) by the usual scientific methods, and (b) by simplified methods used in industrial works. The results obtained show that better results are obtained by grinding to a definite degree than by grinding to time. B. P. RIDGE.

Properties, analysis, and practical testing of cellulose acetates. M. DESCHIENS (Chim. et Ind., 1929, 21, 1131—1140).—Methods for the determination

of solubility, moisture, ash, viscosity, ester content, free and combined acid, copper number, stability, and plasticity of cellulose acetates are reviewed. The elongations for given stresses up to breaking stress are determined for strips of cellulose acetate film deposited from solutions in various mixed solvents, the plasticising action of benzyl alcohol being shown by an earlier yield point and later breaking point. At 20° with suitable ventilation and R.H. 50%, a cellulose acetate film deposited from 45 g. of varnish on a surface 40 cm. by 40 cm. dried to 6 g. in 4 hrs. on glass or 5 g. in 1.5 hrs. on linen; the film detached from glass reached constant weight (5 g.) in 8 days.

C. HOLLINS.

New method for the determination of copper number [of cellulose materials]. K. G. JONAS and A. DRÖSSEL (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 109—112).—A modification of the Schwalbe-Braidy method is described which reduces the limits of error and gives results comparable with those obtained by the Schwalbe-Hägglund method. Larger quantities of water are used in transferring the broken-up cellulose material to the flask in which it is treated with copper solution. The effect on the copper number of using different amounts of water in this operation is shown, and results obtained by the Schwalbe-Braidy and Schwalbe-Hägglund methods are compared for a variety of different cellulose materials. According to the procedure recommended, 2.5 g. of cellulose are steeped in 50 c.c. of water, shaken with glass beads, washed into an Erlenmeyer flask with 50 c.c. of water, the contents of the flask are heated to 100°, and a boiling Braidy solution (10 c.c. of copper sulphate and 190 c.c. of carbonate-bicarbonate solution) is added. The copper number determination is then carried out in the usual manner.

B. P. RIDGE.

New methods of analytical control in the manufacture of sulphite-cellulose. E. GRAAF (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 115—119).—Disadvantages of the usual methods and advantages of electrometric methods in the analysis of sulphite-liquors are discussed. The preparation of the necessary apparatus and solutions, the use of the quinhydrone and calomel electrodes, and methods for the determination of calcium oxide, sugars, etc. in sulphite-cellulose waste-liquors are described.

B. P. RIDGE.

Production of absolute alcohol from sulphite-spirit. H. KIRMREUTHER (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 102—106).—Difficulties hitherto experienced in the use of sulphite-spirit as a motor fuel are attributed to its water content, and they are overcome if the 94—95% technical spirit is converted into absolute alcohol. This has the further advantage that the heat of combustion of the fuel is raised by about 6.5%. On the average 40—45 litres of spirit are obtained from 1 ton of sulphite-cellulose, but it is impossible to obtain spirit more concentrated than 94.5—95% by the usual distillation process because the b.p. of pure alcohol is slightly higher than that of the 94—95% mixture. Two methods are available for the production of absolute alcohol; in one the dehydration is performed by means of lime, but this has the

disadvantage that the process is not continuous, whilst in the second the aqueous alcohol is distilled in the presence of benzene. The composition of this ternary mixture changes with continued distillation until finally absolute alcohol remains. Apparatus for conducting these distillations as a continuous process is described.

B. P. RIDGE.

Influence of "Saftbraun" dyeing on the strength of paper. A. SCHLATTER (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 94—95).—"Saftbraun" is a dye obtained by treating a mixture of brown coal and Cassel earth (ochre) with soda. When it is used for dyeing packing paper, the strength of the latter, as shown by tearing length and bursting pressure, increases with increasing dye concentration and then falls off. The tearing length shows a maximum corresponding with 6.5%, and the bursting pressure a maximum at 5% dye content. For larger amounts of dye the bursting pressure curve falls more slowly than the tearing length curve. The increase of strength thus caused by the dye is attributed to its impregnating and binding properties.

B. P. RIDGE.

Sodium hydroxide from waste liquors. FAUST.—See VII.

PATENTS.

Manufacture of artificial textile fibres. W. SEVER, JUN., and J. B. SPEAKMAN (B.P. 313,816, 3.4.28. Cf. B.P. 300,221; B., 1929, 49).—By suitably regulating the temperature and concentration of the cellulose solution and of the coagulating baths, two of which are employed, the first being so diluted that it does not bring about complete coagulation, the outer and inner portions of each filament receive different treatment and shrink unequally, causing it to curl or crimp like natural wool.

F. R. ENNOS.

Manufacture of fibrous material and moulded articles. S. F. McCORD and DORCAM MACHÉ Co., LTD. (B.P. 313,112, 7.3.28).—Fibrous material, e.g., waste paper (80—120 pts.), is dry-ground and thoroughly mixed with maize or other starch (100 pts.) and a small quantity of water. The mixture is then moulded under heat and pressure for about 10 min. If desired, fillers, e.g., whiting, may be incorporated.

D. J. NORMAN.

Degreasing of fibrous materials. IMPERIAL CHEM. INDUSTRIES, and J. SAVAGE (B.P. 311,825, 23.11.27).—Apparatus is described for degreasing materials by means of volatile grease solvents under the reflux principle and for the recovery and regeneration of the used solvent.

D. J. NORMAN.

Extracting pure cellulose from the bagasse of sugar cane. E. C. H. VALET (B.P. 287,516, 10.2.28. Ger., 23.3.27).—The process of B.P. 277,163 (cf. U.S.P. 1,630,147; B., 1927, 552) is modified as follows. The bagasse, preferably after drying and removing dirt, parenchyma, etc. mechanically, is softened by treatment at not above 90° with 10 times its weight of a solution containing 1.25% of lime and 0.125% of sodium sulphite. This liquor is then removed and replaced by a solution containing 2% of caustic soda, 0.5% of caustic potash, and 1% of sodium sulphite, in which the material

is digested at 2—5 atm. for 4—6 hrs. to dissolve fats and resins and emulsify waxes. After reducing the pressure, a solution containing 2.5% of a fatty soap is added and the cooking continued for another $\frac{1}{2}$ hr. to ensure complete emulsification of all products removed by the alkaline digestion. The liquor is then removed and the cellulose thoroughly washed with hot water. The product may be bleached and used in the paper or artificial silk industry. D. J. NORMAN.

Conditioning of cellulose fibre for conversion into cellulose derivatives. G. A. RICHTER, M. O. SCHUR, and R. H. RASCH, Assrs. to BROWN Co. (U.S.P. 1,701,543, 12.2.29. Appl., 9.10.26).—The fibre is sheeted or shredded, coated with 1—10% of a cellulose derivative, e.g., nitrocellulose, and nitrated. Alternatively, the cellulose may be regenerated from the derivative used and the product nitrated. R. BRIGHTMAN.

Manufacture of stable, highly acetylated cellulose acetate. J. G. JURLING (B.P. 313,198, 7.3.28).—The sulphuric acid which combines with the cellulose during the acetylation process using sulphuric acid as catalyst can be replaced by its equivalent of acetyl, giving a product corresponding in acetyl content with cellulose triacetate, and characterised by increased stability to heat, if, on completion of the acetylation process, the calculated quantity or a slight excess of water (or an alcohol) is added to combine with the excess of acetic anhydride and the whole is then kept until the substitution reaction is complete. Samples taken before and after stabilisation show, respectively, acetyl (as acetic acid) 57.87% and 61.75%, sulphuric acid 3.71% and 0.05%, and have charring point 130° and 210°. D. J. NORMAN.

Manufacture of artificial products from cellulose derivatives. I. G. FARBERIND. A.-G. (B.P. 285,355, 13.2.28. Ger., 12.2.27).—Artificial materials of any desired shape which are insoluble in water and organic media are made by mixing a solution in an organic solvent of a cellulose ester, which is insoluble and does not swell in water, with a cellulose ether dissolved in or swelled by water, and desiccating the emulsion so obtained. F. R. ENNOS.

Manufacture of artificial silk from cuprammonium solutions of cellulose. O. FREIHERR VON KOHORN (O. KOHORN & Co.) and A. PERL (B.P. 310,864, 31.1.28).—The precipitant water is treated with a small quantity of alkali, heated to remove ammonia, and the copper recovered as a sludge of oxide; it is then cooled and re-used after mixing if necessary with sufficient fresh water to lower the soluble salt content below that which would disturb the regular course of the spinning process. F. R. ENNOS.

Artificial silk production. BORVISK SYND., LTD., Assees. of B. BORZYKOWSKI (B.P. 287,073, 12.3.28. Ger., 12.3.27).—The artificial silk is spun on to a perforated bobbin and, immediately after washing, is treated with an oil, oil emulsion, soap solution, or other suitable softening agent to impart flexibility. F. R. ENNOS.

Treatment of artificial fibrous material. L. LILIENTFELD (B.P. 312,197, 21.11.27).—The extensibility and elasticity of artificial fibres, particularly viscose

fibres, obtained by the use of spinning baths of high acid concentration, are increased by treating the silk with carbon disulphide and alkali successively in either order or simultaneously. The material may be treated in a stretched or unstretched condition, but in the former case the stretching should be reduced or discontinued before the silk leaves the alkaline bath. *E.g.*, viscose silk in the form of finished but, if desired, unbleached skeins is impregnated with carbon disulphide, freed from excess thereof, and immersed for 1—5 min. in 18—20% caustic soda solution at 15—18°. After removing excess of soda the material is introduced into, *e.g.*, 5—10% sulphuric acid, and is subsequently washed and dried. The extensibility of the silk is thus increased from 4—5% to 13—18%, and the elasticity from 3.5—4% to 5—6%. This process may be applied to freshly-spun, washed silk. D. J. NORMAN.

Embellishment and rendering waterproof of flat pieces of material and production of articles therefrom. L. RADO (B.P. 313,550, 14.3.28).—Cellulose hydrate foil, coated on one or both sides with a transparent layer of lacquer or varnish which is insensitive to water, *e.g.*, celluloid solution, nitrocellulose solution, is applied to the material such as paper, fabric, or aluminium foil, by means of a suitable adhesive. F. R. ENNOS.

Production of printing surfaces of wood and products thereof. J. K. SNYDER, Assr. to A. C. HORN Co. (U.S.P. 1,717,945—7, 18.6.29. Appl., 29.11.27).—The surface of (A) walnut, (B) oak, and (C) mahogany is etched by treatment with zinc chloride, with sodium hydroxide, and with acetic acid, respectively, the softened grain is brushed out, and the etching material is neutralised, after which the surface is treated with a precipitant and waterproofed by treatment with a metal stearate and a binder in liquid form, when it is ready for use for printing. In (B) and (C) the surface is hardened by treatment with a metal salt after neutralisation of the etching material. L. A. COLES.

Manufacture of [waterproof] paper. Manufacture of waterproofing material. (A) L. KIRSCHBRAUN and A. L. CLAPP, Assr. to FLINTKOTE Co. (B) L. KIRSCHBRAUN (U.S.P. 1,708,926—7, 9.4.29. Appl., [A] 7.2.21, [B] 14.2.21. Renewed [A] 24.9.25, [B] 28.10.26).—(A) Bitumen, which has been emulsified in water with the aid of colloidal clay, is mixed with fibrous stock and caused to deposit and clot thereon by the addition of sodium silicate and aluminium sulphate. (B) Bituminous waterproofing material is reduced to a relatively dry comminuted form, and the particles are enveloped in a protective coating by the addition of sodium silicate and aluminium sulphate with production of a non-adhesive composition, which is then mixed with fibrous stock. These mixtures are formed into sheets and dried. F. R. ENNOS.

Washing of wool. E. C. DUHAMEL (U.S.P. 1,717,440, 18.6.29. Appl., 8.5.25. Fr., 13.5.24).—See B.P. 234,055; B., 1926, 912.

Washing wool and other textile materials. E. C. DUHAMEL (U.S.P. 1,717,375, 18.6.29. Appl., 7.3.25. Fr., 11.3.24).—See B.P. 230,808; B., 1926, 532.

Degreasing of raw wool and other textile fibres. A. ENGELHARDT, Asst. to I. G. FARBENIND. A.-G. (U.S.P. 1,718,548, 25.6.29. Appl., 10.2.27. Ger., 12.3.26).—See B.P. 287,230; B., 1928, 443.

Manufacture of pulp and fibre products. Treatment of cane material. R. A. MARR (U.S.P. 1,717,794—1,717,800, 18.6.29. Appl., [A, c] 2.2.23, [B] 2.3.26, [D] 28.3.24, [E] 27.3.24, [F] 16.4.24, [G] 17.10.22. Renewed [B] 10.10.28, [c] 3.9.26, [D, E] 15.3.29).—See B.P. 266,168; B., 1927, 295.

[Laundry] drum washing and centrifuging machines. M. AURICH (B.P. 313,743, 6.6.28).

Apparatus for use in manufacture of artificial filaments, films, etc. BRIT. CELANESE, LTD., and E. KINSELLA (B.P. 314,099, 17.12.27).

Colouring, sizing, impregnating, or otherwise treating paper. I. G. FARBENIND. A.-G. (B.P. 305,122, 26.11.28. Ger., 31.1.26. Addn. to B.P. 293,000).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

"Saftbraun" dyeing of paper. SCHLATTER.—See V.

PATENTS.

Bleaching or oxidation of fibrous substances and fabric by treatment with ozonised air. I. ACZEL (B.P. 287,904, 24.3.28. Hung., 29.3.27).—Fabric is bleached during passage through a chamber containing a number of electrodes, the fabric being guided between them and thereby subjected to the action of ozone formed by an electric discharge. Loose fibres are bleached similarly while being carried between electrodes on a wire brattice. Such treatment with ozone may also be used for giving a gloss to the textile material or for development after dyeing.

A. J. HALL

Production of fast blue dyeings on the [cotton] fibre. [After-coppering of trisazo dyes.] I. G. FARBENIND. A.-G. (B.P. 285,442, 15.2.28. Ger., 16.2.27).—Cotton dyed with blue trisazo dyes of the type amino-salicylic or aminocresotic acid \rightarrow middle component \rightarrow middle component \rightarrow J-acid or a derivative (cf. B.P. 3673 of 1900; B., 1901, 118) are treated with a copper compound (copper sulphate). The dyeings become faster to washing without changing appreciably in shade.

C. HOLLINS.

Dyeing of cellulose derivatives [with ice colours]. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. E. WALKER (B.P. 310,779, 30.1.28).—Acetate silk etc. is treated with an aqueous dispersion of a 2:3-hydroxynaphthoic arylamide (particularly the *m*-nitro-anilide or the *p*-anisidide), rinsed, and passed through a diazo solution (e.g., diazotised *p*-nitroaniline or 5-nitro-anisidine).

C. HOLLINS.

Production of black colorations on mixed materials containing organic derivatives [esters or ethers] of cellulose. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 311,435, 7.1.28).—Mixed acetate silk (etc.) and cotton, viscose silk, or wool materials are dyed in level black shades by applying a mixture of *p*-amino-diphenylamine and aniline (e.g., in equal parts) and oxidising in the usual manner.

C. HOLLINS.

Manufacture of black dyeings with white or coloured effects on acetate silk. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 311,467, 16.2.28).—Acetate silk (white or coloured) is printed with a wax or fat resist and then dyed with a diphenyl black, e.g., *p*-aminodiphenylamine oxidised with dichromate, the resist being subsequently removed.

C. HOLLINS.

Coloration of materials comprising cellulose derivatives [esters and ethers]. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 311,433, 13.12.27).—Acetate silk etc. is dyed in reddish- or greenish-blue shades with azo dyes made by coupling a diazotised aminoxanthene, particularly Rhodamine 3G extra (Colour Index No. 753), with suitable components, e.g., phenol, *m*-toluidine, or dimethylaniline.

C. HOLLINS.

Coloration of materials comprising cellulose derivatives [with anthraquinone azo dyes]. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and D. H. MOSBY (B.P. 310,827, 30.1.28).—Acetate silk etc. is dyed with azo dyes obtained by diazotising or tetrazotising a mono- or di-aminoanthraquinone and coupling with a suitable component. Examples are: 1-aminoanthraquinone \rightarrow *m*-toluidine (yellow) or anthranilic acid (golden-orange), and 1-amino-2-methylanthraquinone \rightarrow dimethyl-*m*-aminophenol (orange).

C. HOLLINS.

Dyeing of artificial silk from esters and ethers of cellulose and its conversion products. I. G. FARBENIND. A.-G. (B.P. 284,652, 31.1.28. Ger., 2.2.27).—Acetate silk etc. is dyed with azo dyes containing not more than one sulphonic or carboxyl group, made by coupling a diazotised arylamine with fused-ring derivatives of 5-membered ring compounds capable of coupling. Examples are: sulphanilic acid or 2:5-dichloro-aniline \rightarrow 2-methylindole (yellow); aniline-*o*-sulphonic acid \rightarrow 3-hydroxythionaphthene (*S*-oxide of thio-indoxyl; orange) or the corresponding sulphone (greenish-yellow); 6-chloro-*o*-toluidine \rightarrow oxindole-6-sulphonic acid (greenish-yellow).

C. HOLLINS.

Production of green shades on materials made of or containing cellulose esters or ethers. IMPERIAL CHEM. INDUSTRIES, LTD., and A. DAVIDSON (B.P. 312,506, 9.6.28).—Acetate silk etc. is dyed in bright green shades with *NN'*-dimethylindigo.

C. HOLLINS.

Dyeing of cellulose [ester or ether] derivatives [in package or wound form]. BRIT. CELANESE, LTD., G. H. ELLIS, and T. ELLISON (B.P. 311,675, 11.2.28).—In order to secure level dyeing of acetate silk etc. in the form of cops, pirns, cheeses, rolls, etc. an insoluble dye is applied in colloidal suspension.

C. HOLLINS.

Dyeing [with sulphuric esters of leuco-2-aminoanthraquinone]. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 312,404, 18.11.27).—The sulphuric ester of leuco-2-aminoanthraquinone, prepared, e.g., by the method of B.P. 312,243 (B., 1929, 673), is oxidised on the fibre with boiling acid copper sulphate solution to a blue, probably indanthrone.

C. HOLLINS.

Production of pattern effects [by discharge printing] on materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 312,655, 30.1.28).—The fabric is

died with suitable colours (*e.g.*, magenta, violet PDH, aniline \rightarrow phenylmethylpyrazolone, 1:4-diaminoanthraquinone, 1:4-aminohydroxyanthraquinone) and printed with a preparation of a chlorate with ferricyanide or a vanadate as catalyst, with or without the addition of resistant colours (*e.g.*, methylene-blue, rhodamine 6G extra, pigments of the Naphthol AS series, mineral colours, etc.).

C. HOLLINS.

Decoration of fabrics. S. J. SUFFERN, ASSR. to RAMAPO FINISHING CORP. (U.S.P. 1,701,197, 5.2.29. Appl., 14.5.27).—Discharge colours are printed on the fabric simultaneously with the printing of metallic colours thereon.

R. BRIGHTMAN.

Treatment of fibres and fabrics. H. LIVSEY, G. E. HOLDEN, and J. & J. M. WORRALL, LTD. (B.P. 313,980, 20.2.28).—The affinity of cotton or other textile fabric for dyes is increased by impregnating it with a solution of gelatin and steaming it (*cf.* Holden, B., 1928, 854), and is destroyed by treatment with oxidising agents (hypochlorites), but not by soaping or treatment with alkaline reducing solutions.

A. J. HALL.

Waterproofing, more especially for wool and silk goods. W. F. HABERKORN (B.P. 313,771, 29.6.28).—The materials are impregnated at about 70° with an aqueous suspension of aluminium hydroxide prepared by the addition of soda to aluminium sulphate, the amount of hydroxide being at most 5% of the dry weight of the goods.

L. G. LAWRIE.

Production of [photographic] pattern and like effects on materials made of or containing cellulose esters. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 310,773, 30.1.28).—A cellulose acetate fabric is impregnated with a primary aromatic amine (particularly *p*-nitroaniline, nitro-*o*-anisidines, *m*-nitro-*p*-toluidine, *p*-chloroaniline, α -naphthylamine, benzidine, dianisidine, *p*-aminobenzeneazo- α -naphthylamine), and the latter is diazotised. The material is dried, exposed to light, *e.g.*, under a photographic negative, and finally developed with a suitable coupling component.

C. HOLLINS.

Treatment [surfacing] of vegetable fibres and fabrics. Increasing the strength and elasticity of cotton fibres. M. MELLIAND, ASSR. to MELIANA CORP. OF AMERICA (U.S.P. 1,717,991—2, 18.6.29. Appl., [A] 25.7.27, [B] 21.6.26. Renewed [B] 26.11.28. Ger., [A] 25.6.26, [B] 2.7.25).—See B.P. 273,327 and 254,695; B., 1928, 745; 1927, 247.

Treatment of textile materials. C. B. WHITE, ASSR. to VIVATEX PROCESSES, INC. (U.S.P. 1,717,483, 18.6.26. Appl., 15.5.23).—See B.P. 216,090; B., 1925, 706.

Apparatus for dyeing, washing, or bleaching. H. E. PARTRIDGE (B.P. 314,140, 30.3.28).

Dyeing machine with pivoting vat. E. and L. CONSTANT (SOC. E. & L. CONSTANT) (B.P. 314,453, 27.2.28).

Mercerising machines. (SIR) J. F. NORTON & Co., LTD., and D. G. NORTON (B.P. 314,230, 9.7.28).

Machines for treating skeins [with liquids]. SOC. VEUVE BONNET AINÉ & SES FILS (B.P. 289,410, 26.4.28. Fr., 26.4.27).

Manufacture of decorative [compound] sheet [of cellulosic] material [by heat and pressure]. SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 305,189 21.4.28. Fr., 3.2.28).

Dibenzanthrone dyes (B.P. 284,656). **Dyeing with dibenzanthronyls** (B.P. 312,093).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Recovery by dialysis of sodium hydroxide from waste liquors containing colloidal impurities. O. FAUST (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 106—108).—Suitable tanks in which the dialysis of waste liquors may be carried out are described, and price, output, and the life of the membranes used are discussed. A 90% yield of sodium hydroxide can be obtained. From a 17—18% waste liquor, 130—140 kg. of sodium hydroxide may be obtained from one unit of the apparatus in 24 hrs. in the form of a 9% solution, but if a more concentrated solution of the pure caustic soda is required the rate of production is less. The water consumption of the apparatus is 2 litres of water per litre of liquor.

B. P. RIDGE.

Carbamate-carbonate theory of the ammonia-soda process. E. I. ORLOV (Ukraine Chem. J., 1928, 3, 139—140).—An introduction to the following paper by Kiritschenko.

A. FREIMAN.

Carbon dioxide saturation of an ammoniacal solution of sodium chloride and its possible intensification. N. E. KIRITSCHENKO (Ukraine Chem. J., 1928, 3, 141—179).—The processes taking place in the carbonating towers of the Solvay process are discussed both from a purely chemical and from a physico-chemical point of view. It is suggested that the product formed from the carbon dioxide and ammonia is ammonium carbamate, which is being gradually, but at a definite rate according to the law of mass action, converted into ammonium bicarbonate, and not ammonium carbonate, which then reacts with sodium chloride. The author disagrees from the suggestion that the carbonating tower can be divided into three zones in which the different reactions take place. Discussing the chemical reactions from a thermal point of view and applying Le Chatelier's principle, he shows that a high temperature favours the process, but, on account of the rise in the vapour pressure of ammonia and of the rate of decomposition of ammonium bicarbonate, the upper limit has to be kept down or artificial cooling introduced. The process is also discussed from the point of view of applied pressure and the concentration and quantity of the gases introduced.

A. FREIMAN.

Decomposition of water by ferrous hydroxide and the formation of hydrogen in potash deposit works. H. DITZ (Z. Elektrochem., 1929, 35, 392—393).—The observations of Precht (A., 1879, 603; 1881, 227) of the existence of free hydrogen in the Stassfurt deposits and of Goldberg (Diss., Dresden, 1914) that water and ferrous hydroxide interact when heated in an autoclave at 200° to form hydrogen and triferric tetroxide are discussed on the basis of Schikorr's view (A., 1929, 266) that water is catalytically decomposed by metallic iron and ferrous hydroxide.

H. T. S. BRITTON.

Dissociation of carbon monoxide in contact with refractory materials. D. W. HUBBARD and W. J. REES (Trans. Ceram. Soc., 1929, 28, 277—307).—Apparatus is described by means of which a given volume of carbon monoxide could be circulated continuously over catalysts packed in a silica tube heated in an electric furnace. Variations in pressure, due to the reaction $2\text{CO} \rightarrow \text{CO}_2 + \text{C}$, were observed by taking barometric and manometric readings at regular, frequent intervals, while the temperature was slowly raised. The results indicated that dissociation of carbon monoxide is promoted by pure kaolin at 450° and 470—495°; by pure silica at 520—570°; by pure alumina at 260° and 350°; by firebrick material at 410°, 430°, and 500°; by silica brick material at 530—540°; by re-burned firebrick material at 380° and 470°; by calcined clay at 300—400°; and by pure ferric oxide at 300—700°. F. SALT.

PATENTS.

Continuous production of fused caustic alkalis. I. G. FARBERIND. A.-G. (B.P. 306,935, 22.1.29. Ger., 29.2.28).—Caustic alkali lyes of about 50% concentration are heated in inclined tubes rotated at such a speed that the insides are completely coated with the lye. The tubes are constructed of or lined with metal resistant to the alkali, *e.g.*, silver, and so operated that the interiors are filled with steam as free as possible from oxygen. The fused product, free or nearly free from water, issues from the lower ends, whence it flows on to drums or into dishes. L. A. COLES.

Synthetic production of ammonia. R. S. RICHARDSON, ASSR. to NITROGEN ENG. CORP. (U.S.P. 1,707,417, 24.2.29. Appl., 30.12.25).—The incoming gas is passed through the catalyst first in heat exchange tubes only and then in direct contact in the same direction.

R. BRIGHTMAN.

Synthetic production of bodies [ammonia] from their component gases. R. S. RICHARDSON, ASSR. to NITROGEN ENG. CORP. (U.S.P. 1,704,214, 5.3.29. Appl., 24.4.25).—A mixture of nitrogen and hydrogen is passed over a small amount of catalyst at high temperatures and below 400 atm., cooler nitrogen-hydrogen mixture is added to the outflowing gases, and the whole is passed over a much larger quantity of catalyst contained in a second chamber, the pressure being maintained constant during the two operations. W. G. CAREY.

Manufacture of adsorbent silica and like gels. SILICA GEL CORP., ASSEES. of E. B. MILLER and G. C. CONNOLLY (B.P. 287,066, 12.3.28. U.S., 12.3.27).—Hard and tough gels, *e.g.*, of silica, titania, alumina, having a final sp. gr. of about 0.5 are made by washing the hydrogels, submitting them, prior to dehydration, to a gradual heat treatment at 80—160° in the presence of a water-saturated gas or vapour, cooling the product slowly to room temperature, and drying it at 870°.

W. G. CAREY.

Base-exchange bodies [catalysts]. SELDEN CO., ASSEES. of A. O. JAEGER (B.P. 286,212, 16.1.28. U.S., 28.2.27).—The products comprise non-siliceous base-exchanging material prepared by the interaction of at least one compound containing a non-siliceous anion

capable of forming the acidic portion of the product with at least one compound containing a metal amphoteric in at least one stage of oxidation, at least one of the components having catalytic properties and the reaction taking place under conditions such that the products show slight or distinct alkalinity to litmus. One or more of the components may be a complex compound, *e.g.*, a complex ammonia or cyanogen compound, which is subsequently decomposed by heat; the product may be treated with water-glass to cause surface silicification; and diluents, which may have catalytic properties, may be added during the manufacture of the products. In an example, of which 30 are given, an acidified suspension of vanadium pentoxide is reduced with sulphur dioxide and divided into two equal parts, one half being converted into potassium vanadate by treatment with 5*N*-potassium hydroxide at 50—60° and then mixed first with ground cellite, quartz, etc., and finally with the other half; the product, a catalyst suitable, *e.g.*, for use in the oxidation of toluene to benzaldehyde or of sulphur dioxide to trioxide, is collected on a filter and dried at 60—70°.

L. A. COLES.

Manufacture of finely-divided ferric oxide. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 313,999, 21.3.28. Addn. to B.P. 298,926; B., 1929, 53).—A suspension of ferric hydroxide in an aqueous solution of a compound with an alkaline reaction, *e.g.*, sodium carbonate or calcium hydroxide, such suspension being produced, *e.g.*, by treating a ferric salt solution with a quantity of the basic compound in excess of that required for precipitation, is heated at 100—150° under raised pressure.

L. A. COLES.

Composition for producing hypochlorite solutions. M. C. TAYLOR, ASSR. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,716,014, 4.6.29. Appl., 11.3.27).—An aromatic chloroamine is dissolved in an aqueous solution of sodium carbonate, hydroxide, borate, or phosphate to obtain a solution with 10—12% of available chlorine.

A. R. POWELL.

Manufacture of sodium stannate. H. R. McILHENNEY, ASSR. to VULCAN DETINNING Co. (U.S.P. 1,708,392, 9.4.29. Appl., 22.4.26).—Sodium stannate solution containing, *e.g.*, sodium carbonate is evaporated by heating and circulating in a vacuum until the sodium stannate crystallises. After settling, the hot mother-liquor is drawn off and the crystals are drained by suction. The mother-liquors may be further evaporated.

R. BRIGHTMAN.

Production of materials [*e.g.*, fertilisers] in granular form. C. C. SMITH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 314,639, 9.5.28).—Molten salts, *e.g.*, calcium nitrate and/or ammonium nitrate, are brought in the form of fine drops, which may have been allowed to solidify on the surface, in contact with an oil at a lower temperature than and having a b.p. above that of the molten salt, *e.g.*, kerosene. L. A. COLES.

Conversion of salts, especially fertilisers and the like, into globular or similar shaped bodies. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 313,652, 16.3., 23.7., and 14.9.28).—Molten salts, *e.g.*, fused calcium nitrate containing about 12.5% of water, are spurted, *e.g.*, by causing them to flow on to

the surface of a rotating disc, into a cooling liquid which does not dissolve the salt, *e.g.*, carbon tetrachloride, kept in rapid circulation about a vertical axis. Finely-divided solid material, *e.g.*, calcium carbonate, or oils, waxes, etc. may be added to provide the granules with a protective coating. L. A. COLES.

Working-up of potash salts. A. L. MOND. From RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 314,725, 13.9.28).—A quantity of potassium sulphate equivalent to the magnesium sulphate in the crude salts is precipitated and removed by treating an aqueous solution of the salts with ammonia gas in the presence of ammonium salts, *e.g.*, ammonium chloride; magnesium carbonate and/or magnesium ammonium carbonate is precipitated from the mother-liquor by treating it with ammonia and carbon dioxide or with ammonium carbonate; and the mother-liquor from this process is worked up into sodium bicarbonate by further treatment with ammonia and carbon dioxide. The sodium bicarbonate mother-liquor is used for dissolving more of the crude salts, or may be worked up into a mixture of principally ammonium chloride and potassium chloride by evaporation or cooling to low temperatures, and this mixture may be used as a fertiliser or separated into its constituents by treatment with ammonia. The magnesium carbonate tetrahydrate or magnesium ammonium carbonate may be converted into potassium magnesium bicarbonate by treatment with potassium chloride and carbon dioxide, the mother-liquor, which contains ammonium and potassium chlorides, being worked up into a fertiliser. L. A. COLES.

Leaching of raw phosphate. KUNSTDÜNGER-PATENT-VERWERTUNGS-A.-G., Asses. of F. G. LILJENROTH (B.P. 313,036, 30.10.28. Swed., 5.6.28).—Raw phosphate is leached with sulphuric acid etc. under such conditions, *e.g.*, above 90° and using concentrated acid, that calcium sulphate hemihydrate is formed, and this is subsequently converted into gypsum by dilution and/or cooling of the solution. The conversion is facilitated by seeding with short gypsum crystals. L. A. COLES.

Briquetted charge and production of phosphorus and potash therefrom. W. H. WAGGAMAN and H. W. EASTERWOOD, Assrs. to VICTOR CHEM. WORKS (U.S.P. 1,701,286, 5.2.29. Appl., 18.1.24).—A briquetted mixture of finely-ground natural phosphate, a silicate containing potassium, and carbonaceous material is smelted at 1400–1600°. R. BRIGHTMAN.

Production of phosphoric acid. A. NOBLE, Assr. to FEDERAL PHOSPHORUS CO. (U.S.P. 1,695,558, 18.12.28. Appl., 29.2.24).—A mixture of phosphate rock, silica, and coke, containing 15% excess of coke above theory: $\text{Ca}_3\text{P}_2\text{O}_8 + 3\text{SiO}_2 + 5\text{C} = 3\text{CaSiO}_3 + 2\text{P} + 5\text{CO}$, is charged into a feed stack disposed over the hearth of a furnace. Flame impinges on the charge on one side of the hearth and the phosphorus fume and carbon monoxide escape and are burnt on the other, in heat-exchange with the air feed for combustion of the fuel and the charge, the air for the latter passing down the stack with the charge. R. BRIGHTMAN.

Phosphoric acid recovery. E. DU B. MATHEY and A. O. WILLIAMS, Assrs. to AMER. CYANAMID CO. (U.S.P.

1,714,685, 28.5.29. Appl., 6.9.23).—A sintered mass of phosphate rock, sand, and coke, together with sticks of carbonised wood to keep the charge open, is passed through a vertical shaft furnace with an electric arc in the lower portion. The phosphorus vapour is removed by the current of gases evolved from the carbonising wood in the upper zones of the furnace, and may be condensed directly or burned to oxide as desired.

A. R. POWELL.

Manufacture of phosphorus nitride. C. G. MINER (U.S.P. 1,715,041, 28.5.29. Appl., 6.11.22).—A mixture of phosphate rock, sand, and coke, preheated by waste gases from the reduction furnace, is smelted in an electric furnace, and the issuing vapours are led through a condenser to obtain liquid phosphorus and a gas mixture which is burnt to preheat the charge. The liquid phosphorus siphons over into an inclined reaction furnace through which nitrogen is passed; combination of phosphorus and nitrogen is effected by a high-tension discharge at a temperature above the b.p. of phosphorus but below a red heat. The powdery nitride is cooled in an atmosphere of nitrogen or may be transformed directly into ammonium phosphate by treatment with steam in an atmosphere of nitrogen. A. R. POWELL.

Production of copper arsenate. SOC. CHIM. DES USINES DU RHÔNE (Swiss P. 122,587, 19.10.26. Fr., 21.1.26).—Copper arsenite is heated at 600–700° in an atmosphere consisting at least partly of oxygen.

L. A. COLES.

Dry manufacture of calcium arsenite. J. ALT-WEGG and A. M. DUTEL, Assrs. to SOC. DES USINES CHIM. RHÔNE-POULENC (U.S.P. 1,700,756, 5.2.29. Appl., 29.1.27. Fr., 29.7.26).—A dry mixture of 3 mols. of calcium oxide and 1 mol. of arsenic trioxide is treated with dry steam. R. BRIGHTMAN.

Manufacture of magnesium cyanide and its double compound with ammonia. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 314,242, 23.7.28. Addn. to B.P. 300,348; B., 1929, 53).—“Magnesium cyanide diammoniate” is precipitated by treating solutions or suspensions of magnesium compounds, *e.g.*, the hydroxide or nitrate, in an alcohol, *e.g.*, methyl or ethyl alcohol, with ammonia and hydrogen cyanide, and is removed, washed with alcohol, and dried *in vacuo*. The product may be used direct, *e.g.*, as an insecticide, or may be heated *in vacuo* to expel the ammonia. L. A. COLES.

Recovery of cyanide from solutions. L. D. MILLS and T. B. CROWE, Assrs. to MERRILL CO. (U.S.P. 1,701,818, 12.2.29. Appl., 19.8.24).—The cyanide solution is acidified, *e.g.*, by agitation in countercurrent with a stream of sulphur dioxide, and the acidified solution is stripped of hydrogen cyanide by passing over grids in countercurrent with an air stream, the hydrogen cyanide being recovered by similarly directing the air from the disperser upwards through a descending stream of alkaline solution in the absorber, whence the exhausted air is again cycled to the disperser. R. BRIGHTMAN.

Manufacture of titanium tetrachloride. C. DE ROHDEN, Assr. to COMMERCIAL PIGMENTS CORP. (U.S.P. 1,707,257, 2.4.29. Appl., 8.2.28).—Powdered carbon (20–25 pts.) is mixed with powdered titaniferous ore

(60—50 pts.) and titania (20 pts.) in colloidal suspension, and the dried and granulated mixture is treated with chlorine. Colloidal titania is obtained, *e.g.*, by treating 100 pts. of wet titania (60% water content) free from adsorbed sulphuric acid with 15 pts. of concentrated hydrochloric acid. R. BRIGHTMAN.

Isolation of gases [ozone and nitrogen]. E. KUREK (U.S.P. 1,708,067, 9.4.29. Appl., 2.8.24).—An apparatus for removing ozone from ozonised air comprises a flattened spheroidal vessel revolving at high speed about an axis carrying an inlet tube for air, an ozonising apparatus, and tubes for removing the ozone from the peripheral portions of the vessel and nitrogen from the axial portions. It is claimed that the gases separate by the action of the centrifugal force, and that the air is ozonised by the action of sparks emanating from the friction between a steel rod and an abrasive wheel at the axis. A. R. POWELL.

Prevention of explosions in liquid air apparatus. R. G. AUBERT (U.S.P. 1,717,540, 18.6.29. Appl., 28.1.27. Ger., 15.8.25).—A combustible gas, *e.g.*, acetylene, is mixed with the air, and the mixture is liquefied and then filtered to remove the solidified combustible compound before the liquid air reaches the oxygen distillation zone. F. G. CLARKE.

Treatment of [removal of traces of oxides of nitrogen from] gaseous mixtures containing hydrogen. "L'AIR LIQUIDE" SOC. ANON. FOUR L'ETUDE & L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 287,577, 23.3.28. Fr., 25.3.27).—Gas mixtures from which hydrogen is to be extracted by partial liquefaction are passed at 10—40°, with or without previous removal of hydrogen sulphide, over reduced metals which may contain sulphides, *e.g.*, iron which has been used as catalyst in the synthesis of ammonia.

L. A. COLES.

Production of nitrosyl halide [chloride]. G. B. TAYLOR, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,717,951, 18.6.29. Appl., 28.3.27).—Nitrogen peroxide is caused to react with aqueous potassium chloride solution under such conditions that the hydrogen chloride concentration is less than that corresponding to a hydrogen chloride vapour pressure of 1 mm. of mercury. L. A. COLES.

Purification of crude or impure sulphur. MANCHESTER OXIDE Co., LTD., and R. H. CLAYTON (B.P. 314,697, 24.7.28).—Crude sulphur, particularly that recovered from spent oxide etc., is treated while molten, after, if desired, pretreatment with a stream of air or other oxidising gases, with a stream of sulphur trioxide and is then filtered. L. A. COLES.

Preparation of alkali silicates soluble in water. B. F. HALVORSEN, Assr. to NORSK HYDRO-ELEKTRISK KVAELSTOF A/S. (U.S.P. 1,718,292, 25.6.29. Appl., 14.5.25. Norw., 19.5.24).—See B.P. 247,439; B., 1926, 320.

Manufacture of base-exchanging compounds. T. P. HILDITCH and H. J. WHEATON, Assrs. to AMER. DOUGL Co. (U.S.P. 1,717,777, 18.6.29. Appl., 13.8.23. U.K., 14.8.22).—See B.P. 203,158; B., 1923, 1094 A.

Dissolving a mixture of hafnium and zirconium

salts and separating hafnium and zirconium. A. E. VAN ARKEL and J. H. DE BOER, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,718,616, 25.6.29. Appl., 11.5.25. Holl., 6.6.24).—See B.P. 235,217; B., 1925, 713.

Filtering material (U.S.P. 1,702,104).—See I. **Hydrogen and carbon from hydrocarbons** (U.S.P. 1,717,354).—See II. **Fertilisers** (B.P. 294,117).—See XVI. **Removal of silicic acids from liquids** (B.P. 291,435).—See XXIII.

VIII.—GLASS; CERAMICS.

Kilns and kiln firing. IV. **The Woodall-Duckham chamber kiln.** V. **The E.I.C.T. tunnel kiln.** S. R. HIND (Trans. Ceram. Soc., 1929, 28, 261—275, 316—332; cf. B., 1929, 518).—IV. The kiln is a rectangular structure consisting of two parallel rows of eight chambers, each of which works as a down-draught kiln. Each row of chambers constitutes an arched tunnel 19 ft. 2 in. wide, and each chamber has four fire-mouths, which function as "semi-producers." Methods of setting and firing the kiln are described. Data are presented on the nature of the ware fired, the fuel, ashes, and on the thermal efficiency of the kiln.

V. A tunnel kiln constructed by Etudes Industrielles et Constructions Thermique is described. It has the usual three zones for preheating, firing, and cooling, and an over-all length of about 80 m. The principle of recuperation is applied to the walls of the kiln and the gas producers and to the kiln crown, so as to reduce radiation losses. Four gas producers are attached to the kiln, which fires 50—65 tons per day to about 1300—1400°. The kiln is built over an inspection tunnel. Experimental data obtained during a test lasting 72 hrs. are presented, and the thermal efficiency of the kiln is critically examined. F. SALT.

Developments in kilns for burning refractory materials. A. N. TARRANT (Gas J., 1929, 187, 192—193).—A Woodall-Duckham circular tunnel kiln at present being used to fire "green-state" glazed sanitary ware is briefly described. The thermal efficiency is very much greater than that attained in the unrecuperative periodic type of kiln, 1 ton of fuel sufficing to fire 0.86 ton of ware compared with only 0.24 ton in the best types of chamber muffle kiln. The kiln has a mean circumference of 260 ft. (diam. 88 ft.) and a cross-sectional area 5 ft. square. The hollow, revolving tray floor incorporates heating and steaming flues and dips into a sand seal. The ends of the kiln terminate in the loading and unloading shed. During the first 25 ft. of travel the ware is dried by air currents from the open end of the kiln. At the end of this section is a ventilating offtake. The ware then passes through the preheating zone where it is directly heated by waste gases of gradually increasing temperature. At this stage the tray floor is ventilated for the immediate removal of the evolved water. As the temperature reaches 700—800° the collapse of cones automatically seals the ventilating holes so that, during the stages of higher-temperature firing, and of maturing and cooling, the ware is under muffle conditions. The gas firing takes place over a length of 30 ft., with controls at 18 positions on each

side of the kiln. The ware leaves the kiln at a temperature only 5° above that at which it enters.

J. A. SUGDEN.

Comparative effects on an earthenware slip of varying soda-silica ratios in sodium silicate. K. SILK and N. D. WOOD (Trans. Ceram. Soc., 1929, 28, 252—260).—The effect of such variation on the fluidity of an earthenware slip was studied by measuring the time required for a given volume to flow through a 4.1-mm. nozzle. A decrease in the silica-soda ratio increased the fluidity of the earthenware slip; the opposite effect with china clay and ball clay reported by other investigators was confirmed. The curve showing the relation between slop weight and fluidity is approximately a rectangular hyperbola. F. SALT.

Comparison of the properties and industrial durability of lime-bonded and clay-bonded silica bricks. W. J. REES and W. HUGILL (Trans. Ceram. Soc., 1929, 28, 221—249).—Preliminary experiments were made with six different quartzites, using 0.5–5% of ball clay and, for purposes of comparison, 0.5–2% of lime as bonds. Deflocculation of the clay slips increased the bonding properties of the clay. A series of bricks of standard size was then made up with different kinds of clay and with lime as bonds. Bricks made with 3.5% of ball clay bond were approximately equivalent in dry and fired strengths to normal bricks with 2% of lime bond. The degree of quartz inversion was not materially affected by varying the bond, but bricks made without any bond showed a higher degree of inversion than those made with either clay or lime bond. Refractoriness and under-load subsidence were approximately the same for both bonds. Clay-bonded bricks showed slightly higher resistance to spalling, but they were less resistant to slag or corrosive dust than the lime-bonded bricks. No difference was observed between the two types of bricks in the roof of an acid, open-hearth furnace; the lime-bonded bricks were slightly better in the roof of basic furnaces. Clay-bonded bricks were more durable in electric steel-melting furnaces. In coke ovens the two types of bricks proved equally durable, being equally resistant to salt corrosion and mechanical wear. F. SALT.

Deformation study of cobalt oxide-iron oxide-silica mixtures. J. F. SHEPHERD (J. Amer. Ceram. Soc., 1929, 12, 494—497).—Mixtures of the oxides, first of cobalt and iron, then of cobalt, iron, and silica, were made up into pyrometric cone shapes, and the fusion behaviour of the cones was studied. The deformation eutectic temperature of mixed iron and cobalt oxides was 1410° , the composition being 46% Co_3O_4 and 54% Fe_2O_3 ; that of iron oxide, cobalt oxide, and silica was 1150° , the composition being 36% Co_3O_4 , 42% Fe_2O_3 , and 22% SiO_2 . These eutectic mixtures have the same physical properties as has fused cobalt oxide, and might therefore be substituted for it in ground-coat enamels. The theory that the alloy between steel and cobalt oxide is spongy and porous, thus giving the enamel its adhering property, is supported in some degree by the honeycombed structure of some of the cones. F. SALT.

Determination of the permeability of refractory

materials to gas. A. KANZ (Arch. Eisenhüttenw., 1928—9, 2, 843—849; Stahl u. Eisen, 1929, 49, 941—942).—A review of recent work shows the unreliability of the various methods which have been proposed for the determination of this property of refractories. The results of earlier workers agree reasonably well as regards the order of magnitude of the permeability number, but the individual figures are discordant. It is shown that the permeability is dependent on the nature of the pores, the grain size, pressure used in making the refractory, and temperature of firing; it is, however, quite independent of the total porosity, and varies according to the direction in the individual bricks. A. R. POWELL.

After-expansion and true sp. gr. of silica refractories for carbonising plant. A. J. DALE, H. T. S. SWALLOW, and F. WHEELER (Gas J., 1929, 187, 200—203).—In the 1929 report of the Refractory Materials Joint Committee of Inst. Gas Eng. and Soc. Brit. Gas Ind., the results of which are here recorded, a partial answer is given to the question as to whether or not 1500° is a more reasonable temperature than 1410° for the "after-expansion" testing of silica materials in view of the higher temperatures used in present-day settings. Some 60 samples of commercial materials were tested for true sp. gr. and "after-expansion" at 1500° and 1410° . It was found that (1) a testing temperature of 1500° revealed a tendency to permanent volume increase of silica materials of d 2.40 or greater, which was not clearly shown in the 2-hr. test at 1410° . If the sp. gr. was below 2.335 the expansion during test at 1500° was negligible, and if below 2.36 the expansion was less than 0.5%; and (2) in the testing of siliceous materials (below 92% SiO_2) any permanent volume increase due to quartz conversion may be accompanied by a volume decrease due to vitrification and shrinkage of the matrix. Further investigation of this case is required. J. A. SUGDEN.

PATENTS.

Medium for rendering enamel turbid. DEUTS. GOLD- U. SILBERSCHNEIDANSTALT VORM. ROESSLER (B.P. 303,061, 22.12.28. Ger., 27.12.27).—Zirconium oxide can be rendered practically insoluble in enamel melts by preheating it to high temperatures, preferably to 1200 – 1500° . Zirconium oxide and other compounds, either alone or together with similar compounds of titanium and antimony similarly treated, have an opacifying effect equal to that of tin oxide. F. SALT.

Production of walls, furnaces, and installations formed of refractory materials resistant to destructive effects. LABORATOIRE DE PERFECTIONNEMENTS THERMIQUES, Assees. of P. E. J. J. COUTURAUD (B.P. 287,556, 13.3.28. Fr., 24.3.27).—A suspension of graphite (38 pts.) in sodium silicate solution (38 pts. of solution of d 1.32 and 4 pts. of water) is applied by brush or spray to the refractory surface to form a layer protective against ash erosion. [Stat. ref.]

J. A. SUGDEN.

Refractory composition for [ingot mould] hot tops. E. L. MESSLER (U.S.P. 1,717,575, 18.6.29. Appl., 17.9.27).—A mixture composed of 40–60% of flint clay, 10–20% of grog, and plastic clay is claimed.

J. A. SUGDEN.

Production of high heat-resisting substances.

H. M. WILLIAMS, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,713,580, 21.5.29. Appl., 10.1.25).—Coarsely-ground magnesite, silica, zirconia, or alumina is bonded with a slurry composed of the same substance ground very finely in the presence of a deflocculating agent such as sodium hydroxide, carbonate, borate, or silicate.

A. R. POWELL.

Manufacture of carbon electrodes and other refractory articles. CARBORUNDUM CO., Assees. of V. C. DOERSCHUK (B.P. 294,176, 13.7.28. U.S., 19.7.27).—When coke or carborundum etc. is crushed it tends to break up into needle- or plate-like particles. If such material is mixed with a bituminous binder and fed into a mould which is being jolted, the particles orient themselves so that their largest cross-sectional areas lie in a direction at right angles to that of jarring. When removed from the mould and fired at 1000°, the material forms a consolidated mass having a greater heat conductivity in one direction (at right angles to the direction of jolting) than in the other. A carbon electrode may thus be made having a greater lateral than longitudinal heat conductivity, which property increases the life of the electrode by decreasing the rate of oxidation of the upper portion. A suitable jolting machine is described.

J. A. SUGDEN.

Lehrs for annealing glassware. BRIT. HARTFORD-FAIRMONT SYND., LTD. From HARTFORD-EMPIRE CO. (B.P. 314,101, 20.2.28).

[Slip] casting of ceramic pieces [e.g., electric insulators]. A. O. AUSTIN (B.P. 313,325, 25.6.28).

IX.—BUILDING MATERIALS.

Natural weathering, and a comparison of chemical and natural weathering of building stones. E. KAISER (Chem. Erde, 1929, 4, 290—342).—In a study of the effects of natural weathering in a region, the properties of the underlying strata are important, since the nature of these often greatly influences the effects due to climatic weathering; certain oases furnish instances of this kind. The weathering of building stones caused by various gases present in the atmosphere is fully discussed, and the analogy with natural weathering is traced. Analysis of a block of sandstone taken from Regensburg Cathedral showed that of the salts formed by weathering calcium sulphate tended to be concentrated in the outermost layers, whilst magnesium sulphate and chlorides had penetrated deeply into the stone. The presence of alumina and silica in the soluble salts due to weathering was also noted, but it is uncertain in what form they occur. Chemical weathering is intensified by light, and building stones exposed to full light are more rapidly corroded than those in a more shaded position.

H. F. HARWOOD.

PATENTS.

Manufacture of cellular concrete. J. A. RICE, Assr. to BUBBLESTONE CO. (U.S.P. 1,717,319, 11.6.29. Appl., 26.10.26).—A concrete foam-slurry is subjected to gas pressure during at least the beginning of the setting period.

F. G. CLARKE.

Building material. B. F. WILLIAMSON, Assr. to STONE HOMES PROCESS, INC. (U.S.P. 1,716,749, 11.6.29.

Appl., 23.7.23).—A cement mix comprises Florida shell-rock (95% CaCO_3), 90% of which will pass a 1—2-in. mesh, and a binder containing 5—15% each, of the entire mix, of lime and hydraulic cement. When set the product is nail-penetrable and may be sawn.

F. G. CLARKE.

Avoiding dust in hydration of lime and like manufactures. S. TRAVIS, and CALLOW ROCK LIME CO. (B.P. 314,561, 27.3.28).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Atmospheric corrosion of metals. Third (experimental) report to the Atmospheric Corrosion Research Committee (British Non-Ferrous Metals Research Association). J. C. HUDSON (Trans. Faraday Soc., 1929, 25, 177—252; cf. B., 1924, 97; 1927, 301).—Field tests extending over a period of one year were made at five experimental stations, representing various types of atmospheres, on different non-ferrous metals, including copper, arsenical copper, cadmium-copper, two tin bronzes, aluminium bronze, three brasses, nickel, pure and commercial zinc, a nickel-chromium alloy, two copper-nickel alloys, and lead. The corrosion was measured by three independent methods based on determinations of the increase in weight of the metals exposed in Stevenson screens, and of the increase in electrical resistance of wires and the loss in weight of plate specimens under conditions of complete exposure; the decrease in breaking load of the wires used in the resistance tests was also determined. An attempt was made to correlate the results with the meteorological and atmospheric pollution data at each station. In the weight-increment tests, the rate of attack was greater in winter than in summer, the difference being accentuated by increase in atmospheric pollution; the average thickness of the layer of metal corroded varied from 15×10^6 in. per year for arsenical and cadmium-copper exposed at Cardington, to 719×10^6 in. for a "compo" wire (63% Cu, 36% Zn, 1% Pb) exposed at Birmingham. Hence atmospheric corrosion should not seriously affect the mechanical properties of the materials exposed under these conditions. The copper-rich materials gave the smallest weight increments, and this was probably due to the formation of non-deliquescent corrosion products. Both field and laboratory observations support the view that there is a critical humidity for each material, determined by the vapour pressure of its corrosion product, above which condensation will occur on the surface. The increase in electrical resistance is proportional to the weight of metal corroded. Resistance changes were found to correspond to an overall corrosion of layers of metal $78\text{—}721 \times 10^6$ in. thick per year, 80/20 nickel-chromium and tin bronzes being the least, and 70/30 brass and "compo" wire the most affected. The average decreases in the breaking loads for the five stations showed good agreement with the average increases in resistance. The two brasses gave anomalous results as a result of copper redeposition or dezincification. To establish a corrosion-time relationship, continuous

tests by resistance method have been begun in London under conditions of complete exposure and are being continued. In the loss of weight tests of completely exposed specimens, the average thickness of the corroded layer varied from 17 to 408×10^6 in. per annum for 80/20 nickel-chromium at Cardington and 60/40 brass at Birmingham, respectively. The rate of corrosion was much greater for fully exposed specimens than for those exposed in the Stevenson screen, and the values outlined for the relative corrosivity of the five atmospheres correspond closely with the atmospheric pollution data.

M. E. NOTTAGE.

Martensite [iron-carbon] system. K. GEBHARD, H. HANEMANN, and A. SOHRADER (Arch. Eisenhüttenw., 1928—9, 2, 763—771, Stahl u. Eisen, 1929, 49, 940—941).—The metastable martensite system in quenched steels has been investigated by thermal and micrographical methods. Up to 0.1% C the quenched steels consists of homogeneous ϵ -solid solution, with 0.1—0.37% C the structure is eutectoidal consisting of $\epsilon + \delta$, with 0.37—0.9% C the structure consists of $\delta + \eta$ ($+\gamma$), and with more than 0.9% C of $(\delta + \eta) + \gamma$; the phase in parentheses exists as the result of the incomplete establishment of equilibrium conditions. The δ -phase, which has not previously been observed, consists of a crystalline constituent containing 0.37% C; the name "heynite" is proposed for it. The eutectoid line occurs at 470° and the eutectoid composition is 0.23% C. In alloys with more than 0.37% C a peritectoidal equilibrium occurs at 325°, the peritectoidal composition being 0.9% C. The η -phase, or hardenite, is the hardening constituent of quenched steels; it occurs only when the carbon exceeds 0.37%, above which the microstructure of the steels is entirely different from that of low-carbon steels. All the above-mentioned constituents of martensitic steels exhibit characteristic etching properties, details of which are given in the original.

A. R. POWELL.

Determination of the austenite content of steel by measuring the magnetic saturation value, and the mechanism of the annealing process in hardened steel. E. MAURER and K. SCHROETER (Stahl u. Eisen, 1929, 49, 929—940).—If a is the magnetic saturation value of steel annealed at 750—800° and cooled slowly, and b the value for steel quenched from above the A3 point, then the austenite content of the latter is shown to be expressed by the equation $100(a-b)/a = \%$ austenite; the method of determining these values and the necessary apparatus are described together with some results obtained on plain carbon and on alloy steels. It was found that a larger proportion of austenite was retained by quenching in oil than by quenching in water in all steels except those containing 0.95% C and high-carbon alloy steels; these results were confirmed by density measurements and micrographic examination. Annealing of hardened steels at 100° results in decomposition of the martensite with the separation of cementite and a decrease in the magnetic saturation value. At 200—250° the austenite decomposes, and a maximum value of the magnetic saturation is obtained which is higher than that of the fully annealed steel; by adding to this the decrease

observed by annealing at 100° a value is obtained which closely approximates to that calculated from theoretical considerations for pure martensite.

A. R. POWELL.

High-purity magnesium produced by sublimation. H. E. BAKKEN (Chem. Met. Eng., 1929, 36, 345—347).—Crude magnesium has been satisfactorily purified by distillation at 650—800° under a pressure of a few mm. of mercury, but for commercial work the process is expensive on account of the rapid rate of deterioration of the vessels and the difficulties of obtaining satisfactory air-tight welds. Excellent commercial results have been obtained, however, by subliming the metal at 600° under 0.5—1.5 mm. The operation is conducted in a cast-steel heating vessel welded to a thinner wrought-iron condenser within which is a closely fitting removable lining upon which the metal condenses in the form of a coarsely crystalline aggregate. The steel vessel is charged with crude magnesium and heated in a gas-fired furnace for 5—6 hrs. at 600°, the end of the sublimation being marked by a rapid fall of temperature in the condenser. Iron, aluminium, and silicon remain as a residue at the bottom of the vessel; sodium and other very volatile impurities condense at the extreme upper part of the condenser and are readily separated from the magnesium. The impurities remaining in the last-named are 0.003% Si and 0.007% (Al + Fe). The sublimed magnesium has d_{20}^{25} 1.7388, d_{25}^{25} 1.642 (solid) and 1.572 (liquid); it has a volume electrical conductivity of 38.65% and a mass conductivity of 197.7% of that of copper.

A. R. POWELL.

Structure of electro-deposited metals. F. FOERSTER and K. KLEMM (Z. Elektrochem., 1929, 35, 409—426; cf. Foerster and Fischer, B., 1927, 78).—Cresolsulphonic acid lowers the interfacial tension between a solution and a metal, and has therefore some influence on the nature of electro-deposits. Impure cresolsulphonic acid contains unchanged *m*-cresol and also resinous matter, both of which were found to have deleterious results. The impurities may be removed by boiling with animal charcoal. Thus the tendency of tin and cadmium to adsorb the impurities during their deposition from acid sulphate solutions led to a needle-like growth of tin and a spongy deposit of cadmium, whilst the purified addition agent caused smooth layers of cadmium to be deposited. Cadmium tended to separate from acid sulphate and ammoniacal solutions in the form of needle-shaped crystals, and especially from the latter at higher current densities. This could be largely prevented by the inclusion of cresolsulphonic acid in the bath. Less cresolsulphonic acid was necessary in the cadmium baths than in those containing tin. Cadmium showed no tendency to deposit as needle growths from either acid chloride or acid perchlorate solutions, though there was such a tendency from ammoniacal solutions of cadmium chloride; the extent of this, however, was not so great as from the corresponding sulphate solutions. In such solutions cresolsulphonic acid may be used with advantage. The metal used as cathode, and its crystalline structure, had considerable influence on the nature of electro-deposited cadmium, tin, zinc, and lead. The effect of cresolsulphonic acid on the deposition of thallium from a

sulphuric acid solution of thallous sulphate was also investigated. H. T. S. BRITTON.

Solubility of metals in milk. G. N. QUAM (Ind. Eng. Chem., 1929, 21, 703—704).—The loss of weight per unit area of sheets of nickel, tin, chromium steel, aluminium, zinc, and copper in pure milk at 20—100° was determined. The milk was also tested for metals in solution. The loss with tin, chromium steel, and aluminium was nil, and neither chromium nor iron could be detected in the milk. The corrosion of nickel, copper, and zinc reaches a maximum at 75—90° associated with the decreasing solubility of oxygen with rise of temperature. C. IRWIN.

Silica bricks. REES and HUGILL.—See VIII.
Cobalt determination in alloys. HEIM.—See XIII.

PATENTS.

Manufacture of cast iron. INTERNAT. NICKEL CO., Assees. of F. B. COYLE (B.P. 290,267, 8.3.28. U.S., 13.5.27).—Molten cast iron of a composition which would give white or mottled iron when solidified against a non-chilling surface is treated in the ladle with one or more graphitisers such as (a) 0.5% of aluminium and 0.6% of 70% ferromanganese, (b) 1.5% of ferrosilicon and 0.6% of 70% ferromanganese, and (c) 0.5% of ferrosilicon, 0.75% of 70% ferromanganese, and 1.5% of 95% ferronickel. [Stat. ref.] A. R. POWELL.

Apparatus for carburising [case-hardening steel]. P. CHAMBERS, Assr. to SECURITY MANUF. CO. (U.S.P. 1,709,247, 16.4.29. Appl., 3.11.25).—The apparatus comprises a metal carburising chamber contained in a gas- or oil-fired furnace and provided with a long tube in the roof through which the carburising material is dropped into the chamber in the form of a briquette which is vaporised by the heat therein. A. R. POWELL.

Carburiser [for case-hardening iron or steel]. F. C. LANGENBERG, Assr. to E. F. HOUGHTON & Co. (U.S.P. 1,695,337—8, 18.12.28. Appl., 22.3.27).—(A) Carburising material is incorporated with 0.25—10% of an organic, oxygen-containing salt of a carburising oxide and of a salt of a carburising oxide containing nitrogen and oxygen. *E.g.*, charcoal is impregnated with 3% of calcium acetate and 2% of potassium nitrite. (B) Organic salts of metals other than alkali or alkaline-earth metals, and especially the salts of metals of the 8th group, are used as accelerators in carburising compositions. *E.g.*, 70—80% of carbonaceous material and 30—20% of energising carbonates are incorporated with 0.25—2% of cobalt acetate. R. BRIGHTMAN.

Treatment of iron and steel. R. ESNAULT-PELTERIE (B.P. 314,219, 26.6.28).—To harden steel, particularly balls and ball races, the steel is tempered and further hardened by the influence of ammonia at 200—700°. C. A. KING.

Desulphurisation of steel. C. T. HENNIG (U.S.P. 1,717,153, 11.6.29. Appl., 7.8.24).—Steel in the open-hearth furnace is treated with sodium carbonate during the refining process so that the metal is violently agitated or "boiled" and a fluid slag containing soda is obtained. The metal and slag from this treatment are run into a bath of partially refined steel in a second

furnace, and boiling is continued until the metal is free from sulphides and oxides. A. R. POWELL.

Low-carbon chromium steel. H. C. BIGGE and C. R. ELLICOTT, Assrs. to BETHLEHEM STEEL CO. (U.S.P. 1,715,979, 4.6.29. Appl., 15.11.24).—Before casting the finished steel a small quantity of a reducing agent, *e.g.*, silicon, is added to the basic slag covering the metal so as to produce a strongly exothermic reaction and effect thorough deoxidation. The metal is cast as soon as the reaction ceases and before any cooling takes place. The resulting steel is non-porous and free from gas. A. R. POWELL.

Wrought articles of iron-chromium-nickel alloy. H. E. POTTS. From ELECTRO METALLURGICAL CO. (B.P. 314,176, 7.5.28. Addn. to B.P. 297,045; B., 1928, 862).—A rolled alloy containing 20—25% (22%) Cr, 10—15% Ni, 0.7—3% Mn, 0.7—3% Si, and not more than 1% C is claimed. [Stat. ref.] C. A. KING.

Metallic alloy. BARBER ASPHALT CO., Assees. of A. B. DAVIS (B.P. 300,248, 16.4.28. U.S., 10.11.27).—An alloy contains 30% Ni, 40.8% Fe, 21% Cr, 6.2% W, 0.45% Si, 1.55% Mn, and, in addition, small quantities of cobalt and copper as modifiers. C. A. KING.

Magnetic material [iron-nickel-cobalt alloys]. G. W. ELMEN, Assr. to (A, B) WESTERN ELECTRIC CO., INC., and (C) BELL TELEPHONE LABS., INC. (U.S.P. 1,715,646—8, 4.6.29. Appl., [A, B] 30.6.26, and [C] 28.6.28. Renewed [A] 20.12.28).—A magnetic alloy having a constant permeability over a wide range of flux densities contains (A) 65—85% Ni, 5—20% Co, and the balance iron (the preferred composition is 73% Ni, 6% Co, and 21% Fe); (B) 50—65% Ni, 10—30% Co, and 10—30% Fe (preferred composition 60% Ni, 15% Co, and 25% Fe); or (C) 45—80% Co, more than 9% Fe, and more than 4% Ni, together with a small quantity of a fourth element which increases the resistivity. A. R. POWELL.

Ore classifier. W. E. ENDREZZE (U.S.P. 1,714,007, 21.5.29. Appl., 17.9.27).—In an inclined trough open at the top, having a transversely curved bottom, and fitted with a spiral conveyor, classification is effected by passing through the pulp streams of air and water from a series of pipes arranged longitudinally in the middle portion of the bottom. A. R. POWELL.

Reduction of oxides or oxide ores. S. WESTBERG (U.S.P. 1,715,155, 28.5.29. Appl., 27.6.25).—The oxide or ore mixed with a regulated quantity of carbon for reduction is heated in a furnace in an atmosphere of water-gas, or other gas mixture containing hydrogen, in such a manner that the oxide is reduced to metal prior to melting, and the reduced mixture is then melted without coming into contact with carbonaceous material. A. R. POWELL.

Reduction of complex ores. F. O. KICHLINE, Assr. to BETHLEHEM STEEL CO. (U.S.P. 1,717,160, 11.6.29. Appl., 4.12.24. Renewed 3.3.27).—The ore mixed with sufficient reducing agent to reduce the more readily reducible constituent is heated at such a temperature that the desired reduction is effected without melting the reduced metal. The mixture is then heated above the m.p. of this metal and rabbled to cause agglomera-

tion of the molten particles and, after cooling, these are separated by mechanical means from the unreduced portion of the ore, which is then submitted to the usual smelting operation to recover its valuable constituents.

A. R. POWELL.

Mixing apparatus [for refining lead]. H. W. YORK, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,706,722, 26.3.29. Appl., 9.2.27).—A hemispherical lead-melting kettle is provided with an upper framework carrying a centrally disposed pump and a small cylindrical vessel emptying into the main kettle. The lead is pumped from the bottom of the kettle through a pipe which delivers it into the centre of a rotating table in the cylinder. A continuous thin film of lead is thus produced on the table and on to this is dropped the refining agent, *e.g.*, sulphur, through a small funnel. The treated lead and dross formed are returned to the main kettle and the dross is removed from the surface of the lead by means of rakes.

A. R. POWELL.

Bearing metals of the ternary system copper, antimony, lead. S. DEICHES (B.P. 292,936, 25.6.28. Austr., 24.6.27).—Bearing metals of a composition represented on the ternary diagram of the copper-antimony-lead system by any point in the field formed by lines joining the point representing the alloy with 87% Pb and 13% Sb to the points representing the alloys containing (a) 60% Sb and 40% Cu and (b) 48.5% Cu and 51.5% Sb, respectively, are claimed. The preferred alloys contain 30–64% Pb together with small amounts of arsenic, cadmium, or mercury, which induce a peritectic hardening to take place.

A. R. POWELL.

Etching of [metallic] printing forms for intaglio printing, planographic printing, etc. BEKK & KAULEN CHEM. FABR. G.M.B.H. (B.P. 304,685, 15.12.28. Appl., 24.1.28).—The etching reagent is prepared by dissolving 75 pts. of ferric chloride in 25 pts. of water and adding 10 pts. of nitric acid, *d* 1.4, with or without the addition of a solution of iodine in potassium iodide solution.

A. R. POWELL.

Electrodeposition of metals. ELECTRO BLEACH & BY-PRODUCTS, LTD., and [A, C] J. HOLLINS and D. JEPSON, [B, D, E] J. HOLLINS (B.P. 312,598–312,600, 314,470, and 314,558, 25.2.28).—(A) A supplementary or by-pass circulation of electrolyte is arranged directly between the base and top of the anode compartment for washing down sludge accumulating in the compartment. (B) In an electrolyser in which electrolyte overflows from the cathode compartment to the anode compartment over a diaphragm, a rotating perforated diffuser tube is arranged around a vertical central perforated tube through which electrolyte is delivered to the cathode compartment. (C) Sludge is removed from the anode compartment by air injected under pressure and travelling upwards through the anodic material (*cf.* B.P. 312,395; B., 1929, 605). (D) Screens are arranged between the cathodes and anode compartment in order to obstruct the direct path between the cathodes and anode material. (E) Mandrels or cathodes, upon which metal is to be deposited, are mounted upon a rotating common head, and rotate upon their axes. Electrolyte

is supplied through a vertical, central, perforated tube surrounded by a rotating perforated tube.

J. S. G. THOMAS.

[Chromium-cobalt] electroplating process. L. SCHULTE, Assr. to ALLEGHENY STEEL Co. (U.S.P. 1,717,168, 18.6.29. Appl., 29.5.26).—An alloy of chromium and cobalt may be plated on to iron or steel articles from a bath made by mixing the following two solutions: (a) containing 50–75 g. of cobalt sulphate, 30–40 g. of sodium sulphate, and 5–15 c.c. of hydrofluoric acid in 250 c.c. of water, and (b) containing 75 g. of chromic acid, 3 g. of potassium hydroxide, and 10 c.c. of hydrofluosilicic acid (concentrated solution) in 250 c.c. of water. In all cases an excess of (b) must be used.

A. R. POWELL.

Production of coatings on metal articles. O. SPRENGER PATENTVERWERTUNG JIROTKA M.B.H., and B. JIROTKA (B.P. 313,901, 19.3.28. Addn. to B.P. 249,971; B., 1926, 496).—If the concentration of alkali carbonate in the electrolyte is increased to above 10%, heavy metals are deposited from the bath on to aluminium with an oxidised appearance. Copper may be deposited in this manner on magnesium and its alloys.

C. A. KING.

Melting [cupola] furnace. W. STEFFE, Assr. to FREIER GRUNDER EISEN- U. METALLWERKE GES.M.B.H. (U.S.P. 1,717,813, 18.6.29. Appl., 15.3.28. Ger., 21.7.27).—See B.P. 294,027; B., 1928, 715.

Copper-aluminium alloy. E. VADERS (U.S.P. 1,718,502, 25.6.29. Appl., 31.3.27. Ger., 15.4.26).—See B.P. 268,654; B., 1927, 448.

Condensing zinc vapour. F. G. BREYER, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,712,133, 7.5.29. Appl., 10.2.27).—See B.P. 295,115; B., 1928, 758.

Method of uniting radioactive material with a metallic carrier. A. FISCHER (U.S.P. 1,718,899, 25.6.29. Appl., 31.8.27. Austr., 29.9.26).—See B.P. 278,347; B., 1928, 489.

Uniformly hardening the head of railway rails [by its immersion in water]. EISENWERK-GES. MAXIMILIANSHÜTTE (B.P. 290,182, 7.5.28. Ger., 5.5.27).

Electrodeposited protective coatings for vessels [under pressure, *e.g.*, mineral water siphons]. I. AINSTEIN (W. I. EINSTEIN) (B.P. 314,594, 4.2.28).

Condensation of mercury (B.P. 303,013).—See I. Refractory for ingot moulds (U.S.P. 1,717,575).—See VIII. Magnetic alloys (U.S.P. 1,715,646–8).—See X. Nickel anodes (B.P. 314,667).—See XI.

XL—ELECTROTECHNICS.

Electro-deposited metals. FOERSTER and KLEMM.—See X.

PATENTS.

Electric [arc] furnace. W. E. MOORE (U.S.P. 1,718,172, 18.6.29. Appl., 7.9.16. Renewed 3.10.28).—Three arcing electrodes are connected to transformer secondaries, which at a neutral point are connected with the conducting, refractory furnace bottom. Switches are provided in the transformer primaries for

varying the arc voltage between the melting and refining operations. J. S. G. THOMAS.

Transformer. J. ROMP and W. F. BRANDOMA, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,718,080, 18.6.29. Appl., 15.2.28. Holl., 3.3.27).—One of the windings of a transformer is made of wire composed of a copper-silver alloy. J. S. G. THOMAS.

Alternating-current rectifier. H. N. MILLER and E. W. ENGLE, Assrs. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,717,517, 18.6.29. Appl., 3.1.28).—The rectifier comprises a partially reduced chloride, *e.g.*, fused silver chloride, in contact with a metal of the niobium or molybdenum groups. F. G. CLARKE.

[Electrodes for] electrolytic condenser. E. F. ANDREWS, Assr. to ANDREWS-HAMMOND CORP. (U.S.P. 1,717,488, 18.6.29. Appl., 26.2.27).—A bi-polar electrode for use with alternating or pulsating currents has opposite faces, respectively, of nickel and iron. Alternatively, two electrodes having effective surfaces of nickel and iron, respectively, may be employed. J. S. G. THOMAS.

Electrolytic apparatus [for electrolysis of water etc. under pressure]. J. E. NOEGGERATH (B.P. 282,688 and 312,674, 21.12.27. Ger., 21.12.26).—(A) Groups of positive and negative funnel-shaped electrodes or electrode surfaces are contained within an insulating jacket entirely surrounded by electrolyte completely filling a container. Gas conduits are arranged either at the centre or circumference of groups of electrodes, whilst electrolyte channels are arranged at the circumference or centre. End electrodes and one central electrode of each group are mechanically more resistant and have larger cross-sections than the others. Expandable coils are employed for the leading-in conductor or the inlet pipe for circulating electrolyte. (B) An elastic wall, *e.g.*, a diaphragm or piston outside the cell, is subjected on opposite sides to the pressure of the products of electrolysis, *e.g.*, oxygen and hydrogen, respectively, so that differences of pressure are automatically equalised. J. S. G. THOMAS.

Production of electrolytic deposits of varying thickness. WÜRTEMBERGISCHE METALLWARENFABR. (Austr. P. [A] 105,042, 4.6.24, and [B] 107,296, 9.9.25).—(A) The anode and cathode are separated by a non-conducting diaphragm which is perforated with numerous, evenly distributed holes except at the parts opposite to those of the cathode where a thicker deposit is required; here the holes are more numerous or of larger dimensions. (B) The thickness of the deposit is varied not only as in (A), but also by varying the distance between the diaphragm and the cathodes. A. R. POWELL.

Resistor and its manufacture. S. LOEWE, Assr. to RADIO CORP. OF AMERICA (U.S.P. 1,717,712, 18.6.29. Appl., 17.1.27. Ger., 13.2.26).—A thin uniform film of a noble metal is deposited on a heated non-conducting carrier from a fine mist produced by subjecting a solution of the metal to pressure. J. S. G. THOMAS.

[Positive plate for] electric accumulators. H. LEITNER (B.P. 314,123, 24.3.28).—Active material is formed on a nickel plate by introducing the plate into

a 1–2% solution of caustic potash or soda to which is added 0.1–0.2% by vol. of a saturated solution of potassium chloride or chlorate, and then charging the plate as a positive, discharging it, and finally charging it in caustic potash solution of normal strength. J. S. G. THOMAS.

Electron-discharge apparatus. BRIT. THOMSON-HOUSTON CO., LTD., Assces. of H. C. THOMPSON (B.P. 302,307, 22.11.28. U.S., 14.12.27).—An impact-electron-emissive surface, *e.g.*, of barium oxide produced by heating barium peroxide, or a varnish containing lithium borate, is formed upon one of at least three electrodes arranged in an evacuated vessel containing the vapour of magnesium or calcium. J. S. G. THOMAS.

Arc lamp electrodes for heavy currents. COMP. LORRAINE DE CHARBONS POUR L'ELECTRICITÉ (B.P. 285,424, 19.1.28. Fr., 16.2.27).—About 55% of oxides, fluorides, and/or oxyfluorides of the rare-earth metals, *e.g.*, a mixture containing 20 pts. by wt. of oxides of rare earths, 30 pts. of rare-earth fluorides, 2 pts. each of magnesia and anhydrous boric acid, and 1 pt. of thoria, alumina, or blue oxide of tungsten, is incorporated throughout the whole of a carbon electrode, so that the mineralisation is more concentrated at the centre than at the periphery of the electrode. J. S. G. THOMAS.

Manufacture of nickel anodes. INTERNAT. NICKEL CO., W. J. HARSHAW, P. M. SAVAGE, and F. K. BEZZENBERGER (B.P. 314,667, 19.6.28).—An oxide of an element of the nickel group is added to nickel in such proportion that the finished product contains 0.05–0.25% O, preferably 0.24%, and the mixture is worked, *e.g.*, rolled, at 925–1315°. J. S. G. THOMAS.

Manufacture of carbon filaments. A. LEDERER (U.S.P. 1,700,901, 5.2.29. Appl., 14.8.25. Renewed 18.5.28).—Substantially pure carbon in a finely-divided state is mixed with a water-soluble iron salt which yields ferric oxide on ignition, and the mass is dried and heated to decompose the salt. The product is ground into a plastic mass with oleic acid and squirted into threads which are heated at 2500° to obtain a crystalline carbon filament for incandescence lamps. A. R. POWELL.

Carrying out photochemical reactions with dissolved reaction materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 314,267, 20.8.28).—The reaction product is extracted from the solvent after exit from the irradiation chamber, the solvent recharged with fresh reaction material, introduced into the irradiation chamber, and the process repeated cyclically. Thus the process may be applied to the conversion of ergosterol into vitamin-D by irradiation of a solution in alcohol, or to the conversion of cyclohexane into hexoic acid by irradiation of a dilute aqueous solution. Suitable apparatus is described. J. S. G. THOMAS.

Piezo-electric crystals. E. W. C. RUSSELL. From C. B. SAWYER (B.P. 314,680, 6.7.28).—Occluded liquid is removed from the end regions of "composite" piezo-electric crystals, *e.g.*, of Rochelle salt, and a solution yielding an electrically conducting material is introduced into those regions, excess solution being removed. Thus

an aqueous solution of silver nitrate from which silver is deposited on exposure to light may be introduced.

J. S. G. THOMAS.

Electromagnetic chemical balance. H. NAGAOKA and J. IKEBE, Assrs. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,717,462, 18.6.29. Appl., 10.11.24. Japan, 20.11.23).—An adjustable electromagnetic couple is exerted upon the balance lever by a pair of armatures operatively connected to the lever on either side of the fulcrum and energised by current flowing in a pair of solenoids in which they are respectively inserted. Means are provided for determining the couple in terms of the energising current.

J. S. G. THOMAS.

Incandescence electric lamps and the like [with replaceable filaments]. C. TOURNE (B.P. 307,702, 24.8.28. Fr., 9.3.28. Addn. to B.P. 273,293, 278,317, and 280,496).

Incandescence cathode. P. W. DOBBEN, J. G. W. MULDER, and E. OOSTERHUIS, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,718,123, 18.6.29. Appl., 18.7.27. Holl., 14.10.26).—See B.P. 275,449; B., 1927, 786.

Poking and stirring apparatus for electric furnaces. SOC. ELECTROMÉTALL. DE MONTRICHER (B.P. 305,569, 2.10.26. Fr., 7.2.28).

Glow-discharge lamps. STANDARD TELEPHONES & CABLES, LTD. FROM WESTERN ELECTRIC CO., INC. (B.P. 314,464, 28.3.28).

Vacuum tubes. W. S. KIMMIG (B.P. 289,422, 20.2.28. U.S., 27.4.27).

X-Ray apparatus for crystallographic examination etc. S. G. S. DICKER. FROM N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 314,367, 26.3.28).

Impregnating [in vacuo] insulating dielectric materials [with oil]. GEN. ENGINEERING CO. (RADCLIFFE), LTD., and S. TAYLOR (B.P. 313,987, 20.3.28).

Instrument for measurement of minute degrees of luminosity. J. NEALE (B.P. 314,620, 20.4.28).

Heat-exchange apparatus (B.P. 299,100).—See I. **Light hydrocarbons** (B.P. 299,861).—See II. **Bleaching etc. with ozone** (B.P. 287,904).—See VI. **Carbon electrodes** (B.P. 294,176).—See VIII. **Chromium-cobalt plating** (U.S.P. 1,717,468). **Electrodeposition of metals** (B.P. 312,598—312,600, 314,470, and 314,558).—See X. **Improvement of corn** (B.P. 313,979).—See XIX.

XII.—FATS; OILS; WAXES.

Methods for cottonseed analysis. G. S. JAMIESON and R. S. MCKINNEY (Oil and Fat Ind., 1929, 6, [7], 11—13).—In a preliminary report from the Committee of the U.S. Dept. of Agriculture on the sampling and analysis of cottonseed, the official and other methods of determining the oil content of the seed, the moisture, and the free fatty acid in the oil are discussed.

E. LEWKOWITSCH.

Process of stand-oil formation. H. MUNZERT (Farben-Ztg., 1929, 34, 2298—2300).—Linseed oil stand-oil cooked at 290° to the viscosity specification (20° Engler at 50°) for paint materials for the State railways corresponds with the stage at which all α -linolenic acid has

just disappeared; from this point zinc oxide pastes made with the oil no longer become yellow on ageing. On prolonging the heating, the viscosity rises more rapidly than the iodine value falls (cf. Wolff, B., 1924, 916), the oil darkens, and the acid value increases. A stand-oil directly bodied to the standard viscosity is superior to a mixture having the same viscosity made by thinning a stouter stand-oil with raw linseed oil, the latter showing the yellowing due the α -linolenic acid of the raw oil.

E. LEWKOWITSCH.

Iodine values of menhaden fish oil. C. H. BANKES and A. R. LANGE (Oil & Fat Ind., 1929, 6, [7], 31—33).—For determining the iodine value of this oil the Wijs method was found to be more reliable and rapid than the Hanus test; the former required only $\frac{1}{2}$ hr. for complete reaction with oils containing up to 5% of free fatty acids, and 1 hr. for oils exceeding that amount, whereas the Hanus solution gave irregular results and the reaction was not always complete even after 3 hrs. Wijs' solution containing excess chlorine gave normal values after being kept for 2—3 days; excess iodine gave normal values, but rendered the solution less stable. The values by either method were increased by 0.6 for each 10° F. rise in temperature above 70° F.

E. LEWKOWITSCH.

Sodium saponification value. W. NORMANN (Chem. Umschau, 1929, 36, 197—198).—The saponification value and acid value are of more use to the soap maker and refiner etc. if calculated in terms of caustic soda instead of potash. A table of the "sodium values" so calculated for many oils, waxes, and resins is given.

E. LEWKOWITSCH.

Oleates as oil sprays. HOCKENYOS.—See XVI. **Test for rancidity in cacao butter.** COOKE. **Adulteration in butter.** HENVILLE and PAULLEY.—See XIX.

PATENTS.

Manufacture of high-grade soaps from low-grade fats. J. STARRELS (U.S.P. 1,701,703, 12.2.29. Appl., 15.10.23).—Dark low-grade fats, high in fatty acids, are esterified with methyl or ethyl alcohol, e.g., in presence of 1—2% of sulphuric acid, and the esters are distilled in a vacuum, e.g., below 225°, with superheated steam; the distilled esters are used for the manufacture of light-coloured neutral soap, saponification being effected, e.g., in a closed vessel, and the alcohol recovered. Distilled esters obtained from original material of high iodine value, e.g., cotton-seed foots, may be partly hydrogenated, in presence of 0.5% of nickel catalyst at 50—75 lb./in.² and 180° to iodine value about 40°, before saponification. Alternatively, the low-grade fat may be hydrolysed before esterifying and the glycerol recovered.

R. BRIGHTMAN.

Manufacture of soap. A. LETTERMANN (B.P. 288,584, 12.4.28. Ger., 12.4.27).—A pulverisable soap is made by producing a uniform emulsion of fatty oils with an alkaline solution and a water-absorbing substance such as soda, potash, etc. at about 60—70°, and allowing it to remain for a short period (e.g., 4—5 hrs.).

E. LEWKOWITSCH.

Treatment of fatty materials. H. BULL (B.P. 314,269, 25.8.28).—Fatty material containing water is

intimately mixed with substances that can combine chemically with the water present, *e.g.*, anhydrous sodium sulphate or carbonate, burnt gypsum, etc., thus facilitating the subsequent removal of the fat by pressure, extraction, etc.

E. LEWKOWITSCH.

Treatment [separation of pericarp and nuts] of palm fruit. RUBBER CULTUUR MAATSCHAPPIJ "AMSTERDAM" (B.P. 299,061, 19.10.28. Holl., 20.10.27).—After pressing the palm fruit at low pressure the mixture is submitted to a blast of heated air or gas, or dried and then exposed to the air-blast.

E. LEWKOWITSCH.

Manufacture of soap [flakes]. E. FLAMMER and C. KELBER (U.S.P. 1,717,553, 18.6.29. Appl., 16.8.26).—See B.P. 265,714; B., 1927, 304.

Carrying out photochemical reactions (B.P. 314,267).—See XI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Relation between colouring power and covering power of white pigments. F. MUNK (Z. angew. Chem., 1929, 42, 737—738).—Determinations, using ultramarine as tinting substance, of the colouring power of a number of white pigments have shown that there is, in general, no simple relation between colouring power and covering power. Many of the factors influencing these properties are quite dissimilar.

J. S. CARTER.

Pigment and colour index. H. A. GARDNER and S. A. LEVY (Amer. Paint & Varnish Manufs.' Assoc., June, 1929, Circ. No. 352, 499—624).—A revision of Circulars No. 104, 148, and 268, now out of print. Standard methods for the determination of coarse particles, oil absorption, sp. gr., and bulking value of pigments are described in detail, and the results of these tests carried out on 700 white and coloured pigments are tabulated. Photomicrographs of coarse particles of pigments retained by 325-mesh and a colour chart for iron oxide pigments are also included.

S. S. WOOLF.

Clarification of nitrocellulose lacquers. F. KOLKE (Farben-Ztg., 1929, 34, 2405—2408).—Clarification of lacquers by settling, filtration, and centrifuging is discussed. Little or no advantage is derived by the use of water-cooling in centrifuging; the actual rise in temperature during centrifuging is less with a pre-heated lacquer than with cold material, and it is advantageous to warm the lacquer before treatment (max. temperature reached 30°). No additional danger is incurred by the use of high-speed centrifuges fitted with modern braking devices.

E. LEWKOWITSCH.

Drying of oil paints and varnishes. H. WOLFF and W. TOELDT (Farben-Ztg., 1929, 34, 2293—2295; cf. B., 1929, 564).—The rate of drying of oil paints and varnishes can be satisfactorily measured by the authors' dye-spreading test (cf. B., 1929, 255) if the dye solution is diluted with benzene. Drying and weight-change curves are given for several varnishes. The drying of a wood oil varnish was peculiar in that the spread of the drop of dye began to increase again after reaching a minimum value.

E. LEWKOWITSCH.

Cobalt determination in driers, japans, alloys, etc. O. HEIM (J. Oil Col. Chem. Assoc., 1929, 12, 175—176).—The ash of the japan etc. is dissolved in dilute hydrochloric acid, or 10 g. of the material are oxidised with sulphuric acid and hydrogen peroxide until free from organic matter and are then dissolved in dilute hydrochloric acid. Zinc oxide is added very gradually at 50° until only a trace remains undissolved and the liquid is filtered; the filtrate is concentrated to 20 c.c., washed into a separator, and 30 g. of ammonium thiocyanate are added. The solution is exhausted with a 9:1 ether-*n*-amyl alcohol mixture, and the ether extract is shaken with 15—20 c.c. of 10% sulphuric acid and washed. The aqueous solution is neutralised with caustic soda and the cobalt determined by electrolysis, by precipitation as the oxide, or as the 3:5-dimethylpyrazole compound (23.88% Co).

E. LEWKOWITSCH.

Ethyl abietate. A. C. JOHNSTON (Ind. Eng. Chem., 1929, 21, 688—689).—This ester may be prepared, without previous preparation of abietic acid, by the esterification of a solution of commercial wood rosin in alcoholic caustic soda (cf. U.S.P. 1,682,280; B., 1928, 936). It is a viscous liquid distilling at 195—200° at 4 mm., and has f.p. —45° and d_{20}^{20} 1.02. It is practically unsaponifiable, is soluble in all proportions in organic esters, benzene, acetone, etc., and readily dissolves ester gum and dammar gum. Mixed with ethyl alcohol it dissolves nitrocellulose. It may replace at the same time the resin and plasticiser in nitrocellulose lacquers, giving a film of good adhesion. The difficulty of balancing the solvents for nitrocellulose and resin is eliminated.

C. IRWIN.

Influence of mixtures of electrolytes on the flocculation curve of rosin. R. LORENZ (Papier-Fabr., 1929, 27, Fest- u. Auslands-heft, 113—115).—Various proportions of calcium or magnesium sulphate to sodium or potassium sulphate were added to samples of the same size-milk and the effects on the flocculation point of the rosin were observed after various times of treatment. For 2 min. treatment, the concentration of calcium sulphate—indicated by the degree of hardness of the solution—at which flocculation takes place increases with increasing sodium sulphate concentration to a maximum of 32.4°, corresponding with 9.3 g. of sodium sulphate per litre, and then falls. Similar curves are obtained for 15 min. and 2 hr. treatments, but the maximum degree of hardness reached is the lower the longer is the time of treatment, although it corresponds approximately with the same sodium sulphate concentration in each case. Similar results are shown when the sodium salt is replaced by potassium sulphate. With magnesium instead of calcium sulphate somewhat better results are obtained. Thus the maximum degree of hardness for 2 min. treatment is 37—39 and for 15 min. 32—34. The concentrations of potassium sulphate at the maxima, however, are higher than those for sodium sulphate. The results, though of scientific interest, have little technical value.

B. P. RIDGE.

Determination of the m.p. of resins. T. H. DURRANS (J. Oil Col. Chem. Assoc., 1929, 12, 173—175).—

A sample (3 g.) of the resin is just melted (in a sulphuric acid bath) in an ordinary test-tube (diam. 17 mm.), a thermometer is inserted so that the bulb is half immersed, and the resin is allowed to solidify; mercury (50 g.) is poured on to the top of the resin and the tube is reheated in the acid bath, the rise in temperature being maintained at 2°/min. As the resin softens the mercury sinks through it; the m.p. is taken as that temperature at which the resin first appears above the mercury. Good agreement of results (within 2°) is obtained by this method. E. LEWKOWITSCH.

Saponification numbers. NORMANN.—See XII.

PATENTS.

Paint and varnish liquid and its manufacture.

C. M. A. STINE, C. COOLIDGE, and E. B. MIDDLETON, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,700,778, 5.2.29. Appl., 5.9.24).—Quinol, pyrogallol, *p*-aminophenol, or tannic acid is added to a rubber solution containing a drier and pigments to prevent reduction of the viscosity due to a depolymerisation of the rubber. R. BRIGHTMAN.

Cellulose ester solutions and plastics. J. B. PAYMAN, H. SWANN, W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,664, 8.11.27).—Esters (except ethyl glycolate) and ether-esters of glycollic and thioglycollic acids are employed as solvents for cellulose esters in quantity substantially greater than that of the non-volatile plasticiser used. The b.p. should not exceed 190° so that the ester does not remain in the film. Clear lacquers and also enamels may be obtained. C. HOLLINS.

Solutions of cellulose esters, particularly for use as varnishes. E. C. R. MARKS. From AMER. CYANAMID Co. (B.P. 312,469, 4.5.28).—Esters of α -hydroxyacids above C₃, e.g., ethyl α -hydroxyisobutyrate, b.p. 148°, prepared from ketone cyanohydrins, are used as solvents in cellulose ester lacquers. C. HOLLINS.

Nitrocellulose-oxy solutions particularly for use as lacquers or varnishes. I. G. FARBERIND. A.-G. (B.P. 287,137, 10.3.28. Ger., 16.3.27).—Rapid-drying, clear, nitrocellulose lacquers or varnishes of low viscosity and high lustre are made by incorporating in solution an oxyn (e.g., linoxyn) and nitrocellulose; plasticisers, resins, etc. may be added as desired. E. LEWKOWITSCH.

Preparation of surfaces for the reception of cellulose lacquers. I. G. FARBERIND. A.-G. (B.P. 314,266, 20.8.28. Addn. to B.P. 281,310; B., 1928, 531).—The intermediate layer described in the prior patent is treated with an agent, e.g., chrome alum, formaldehyde, having a hardening action on the protein present. E. LEWKOWITSCH.

Hard-rubber coating composition and its manufacture. S. E. SHEPPARD and J. J. SCHMITT, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,701,129, 5.2.29. Appl., 18.3.25).—Hard rubber, e.g., 60 pts., is fluxed with a resin (28 pts.) and a drying oil, e.g., 12 pts. of tung oil, at 200–250°, and dissolved in a solvent containing liquid hydrocarbons of the benzene series. Preferably the rubber is first comminuted by digestion in boiling 15% sodium hydroxide solution for 2–5 hrs. R. BRIGHTMAN.

Coating composition and its manufacture. C. M. A. STINE and J. E. BOOGE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,700,779, 5.2.29. Appl., 18.11.24).—Crude or partly or completely vulcanised rubber is mixed with a volatile thinner, e.g., petroleum distillate, and 0.001–11% of a drier, e.g., cobalt linoleate, spread on a surface, and dried in warm air. R. BRIGHTMAN.

Manufacture of [oily or resinous] condensation products from urea, thiourea, or their derivatives, and an alcohol or ketone. I. G. FARBERIND. A.-G. (B.P. 287,095, 14.3.28. Addn. to B.P. 278,390; B., 1928, 648).—The condensation described in the prior patent is performed in presence of aromatic or hydroaromatic amines or acid amides containing the group NH or NH₂, e.g., benzylamine, acetanilide, *p*-toluenesulphonamide, or a mixture of the last two. C. HOLLINS.

Moulding compositions. K. RIPPER (B.P. 287,177, 15.3.28. Austr., 18.3.27).—Moulding powders, which may be hot-pressed to form clear, colourless, or translucent articles, are produced by refluxing formaldehyde with dicyanodiamide (with or without the addition of other substances condensible to resins with formaldehyde, e.g., urea, thiourea, phenol); the resinous product is precipitated as a powder on being poured into cold water. E. LEWKOWITSCH.

Manufacture of moulded artificial masses by removing the liquid medium from emulsion colloids. K. RIPPER, Assr. to F. POLLAK (U.S.P. 1,701,986, 12.2.29. Appl., 20.5.24. Austr., 22.6.23).—Partly dried (jellified) emulsion colloids are moulded by drying in moulds permeable to the solvent, e.g., water, but impermeable to the colloids present; e.g., the viscous condensation product from formaldehyde and carbamide is dried in a porous mould coated with cellulose ester lacquer. R. BRIGHTMAN.

Manufacture of condensation products of urea and formaldehyde. A. GAMS and G. WIDMER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,718,901, 25.6.29. Appl., 17.2.26. Switz., 10.3.25).—See B.P. 249,101; B., 1927, 563.

Phenol[-formaldehyde] resin composition. H. L. BENDER, Assr. to BAKELITE CORP. (U.S.P. 1,717,600, 18.6.29. Appl., 10.11.26).—See B.P. 280,520; B., 1928, 792.

Enamelling furnaces or the like. INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRIZITÄTS-GES. (B.P. 299,398, 25.10.28. Ger., 25.10.27).

Pigment dyes (B.P. 285,096).—See IV. **Finely-divided ferric oxide** (B.P. 313,999). **Titanium compounds** (U.S.P. 1,707,257).—See VII. **Water-proofing of cartridges** (U.S.P. 1,701,868).—See XXII. **Colouring matters for cosmetics** (U.S.P. 1,702,227).—See XXIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Swelling of rubber. P. STAMBERGER and C. M. BLOW (Nature, 1929, 124, 13).—When carbon disulphide, chloroform, or benzene was used as solvent, the same relative vapour-pressure diminution was observed when the concentration of the jelly was expressed as g. of

rubber per g.-mol. of solvent. The curves expressing the relation between the relative vapour pressure and the concentration bend rapidly at a concentration of 20–30% towards the vapour-pressure axis. This suggests that the process is more complex than that of simple dissolution, but the affinity of the solvent for the rubber can only be due to molecular forces. Although no difference between the vapour pressure of the "liquid" jelly of masticated rubber and that of the solid elastic jelly of untreated rubber was observed, the swelling pressures differed considerably. Hence the forces which cause the imbibition of solvent by the jelly are not uniform in nature (cf. Stamberger, A., 1928, 364). A. A. ELDRIDGE.

Dispersion of "pigments" in rubber. II. E. A. GRENQUIST (Ind. Eng. Chem., 1929, 21, 665–669; cf. B., 1928, 867).—It is believed that the final degree of reinforcement in compounded rubber is governed by (a) the free surface energy of the system, a measure of which is the area of the interface between rubber and "pigment" per unit weight, (b) the intensity of wetting of the pigments by rubber or *vice versa*, and (c) the strength of the rubber matrix. Unworked, smoked sheet rubber after soaking in water has a typical granular structure, but mastication leads to disruption of the globules. Gas black contains both isotropic and anisotropic particles; the latter, which have a greater tendency to aggregation, appear under X-ray investigation to possess hexagonal symmetry similar to that of graphite. Microscopical examination of compounded rubber indicates that the state of dispersion of the ingredients, particularly carbon black, is dependent on the rubber structure, the condition of the proteins and resins, the character of the particles, and the presence of recrystallised rhombic sulphur at the beginning of vulcanisation. D. F. TWISS.

Influence of various accelerators on the surface vulcanisation of rubber by ultra-violet radiation. R. DITMAR (Gummi-Ztg., 1929, 43, 2325; cf. B., 1929, 294).—Exposure to ultra-violet rays induces vulcanisation in films of rubber containing sulphur together with an accelerator of vulcanisation and an activator, *e.g.*, zinc hydroxide; of the accelerators tested, thiuram disulphide proved the most active under these conditions. In the absence of an activator, oxidation occurred concurrently with vulcanisation and, in order to obtain satisfactory results, it would be necessary to suppress oxidation by the incorporation of a red or yellow pigment, *e.g.*, selenium-red. D. F. TWISS.

China clays as rubber-compounding ingredients. W. F. O. POLLETT and T. J. DRAKELEY (India-rubber J., 1929, 77, 973–974; 78, 27–28, 61–62).—Notes from a paper already abstracted (B., 1929, 444). D. F. TWISS.

PATENTS.

Treatment of rubber and like substances. A. BIDDLE, Assr. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,716,478, 11.6.29. Appl., 30.12.25).—A mixture substantially of rubber (10 pts.) as in rubber latex dispersed in 100 pts. of a mixture of petroleum, castor oil, linseed oil, and 75 pts. of a 20% casein glue solution is used as a coating composition. D. F. TWISS.

Rubber composition and its manufacture. H. A. WINKELMANN, Assr. to B. F. GOODRICH CO. (U.S.P. 1,716,474, 11.6.29. Appl., 1.6.25).—Comminuted, vulcanised rubber scrap is masticated in a minor proportion of raw rubber and, after the addition of pine tar, sulphuric acid is dispersed throughout the mixture. On heating an exothermic reaction ensues yielding a heat-plastic material. D. F. TWISS.

Compounding and vulcanisation of rubber and products therefrom. S. M. CADWELL, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,701,946, 12.2.29. Appl., 17.12.23. Renewed 12.7.28).—Rubber is heavy-milled and mixed with sulphur and a zinc compound, *e.g.*, zinc oxide, in the usual way, and carbon disulphide and an amine, *e.g.*, dibenzylamine, are separately diffused in as accelerators. Vulcanisation is effected at 20–25° or in 2 hrs. at 100°. R. BRIGHTMAN.

Preservation of rubber latex. NAUGATUCK CHEM. CO., Assecs. of J. MCGAVACK and R. A. SHIVE (B.P. 288,268, 24.3.28. U.S., 8.4.27).—See U.S.P. 1,699,368; B., 1929, 445.

Recovering cotton or other fibres, fibre stuff, or fibre pulp from rubber-fabric. N. V. VEREEN. NEDERLANDSCHE RUBBERFABR. (B.P. 293,419, 22.6.28. Holl., 6.7.27).

Paint and coating compositions (U.S.P. 1,700,778—9 and 1,701,129).—See XIII.

XV.—LEATHER; GLUE.

Histological structure of skin and its relation to the quality of the finished leather. M. KAYE (J. Soc. Leather Trades' Chem., 1929, 13, 73–87, 118–154).—Mammalian dermis consists of grain and corium, both composed mainly of bundles of slightly twisted collagen fibres held together by encircling threads of reticulin, each fibre being composed of twisted collagen fibrils similarly held together. The grain contains more non-fibrous, cellular structures and more elastin and reticulin than the corium. The collagen fibre bundles are thickest in the corium and are interwoven in waves parallel to the surface, fitting into waves of bundles running at right angles. Isolated elastin fibres form a delicate network in the skin which supports and connects various skin structures and imparts resilience. They consist of a central core, readily digested by tryptic enzymes, surrounded by a more resistant sheath. The quality of a light leather can be roughly judged by their condition. The collagen fibre bundles are surrounded by reticulated sheaths, continuous with each other by connecting reticular fibres. The proportion of reticular tissue becomes greater towards the surface of the grain, terminating at the surface as a dense permeable network into which are fitted the lower cells of the epidermis on the upper side and the looped terminals of collagen and elastin fibres on the other. The reticulated tissue is very resistant to the action of acids or alkalis, but is attacked by bacterial action and gradually weakened and softened by sodium sulphide. The reticular tissue is ruptured owing to bacterial action in stale skins, and this may cause looseness and softness in the finished leather and

destruction of the grain membrane. Leather manufactured from fresh skins in which the reticulin fibres are intact is usually firm and of excellent quality. In dried skins the reticulin fibres encircling the collagen fibre bundles are ruptured mechanically, and the bundles are thus able to separate more readily, but this effect is counteracted by the difficulty of dispersing the coagulated interfibrillary substance. The quality of the finished leather is affected by the type of weaving of the fibre bundles, viz., normal, horizontal, or vertical, their internal structure, packing, and general regularity throughout the leather. Three conditions of internal fibre structure are possible, viz., (a) unopened fibre bundles in which, as a result of insufficient soaking, there is little or no separation of the fibres into fibrils; (b) well-opened fibre bundles, where there is the normal separation into fibrils due to the removal of the interfibrillary substance; and (c) loose fibre bundles where the fibrils are loose, widely separated, and fray out easily when teased from the skin or leather, due to over-soaking, over-liming, or over-bating, or to grease which has penetrated throughout the skin and leather. The fibre bundles may be (i) closely packed, as in heavy leathers, due to tanning the plumped pelt or increasing the substance in strong tan liquors, or, when a fault, the result of insufficient soaking; (ii) evenly separated, as in light leathers, producing flexibility, suppleness, and "long" fibres; or (iii) loosely packed, caused by the loss of hide substance, the presence of excessive amounts of fat in the original skin, the destruction of the reticular fibres which bind together the fibre bundles, or by local looseness at the level of the hair roots. The structure and arrangement of the fibres in good leathers are always regular, and irregularities are due to disease, bad curing, or faulty manufacture. "Horizontal weaving" is the result of insufficient soaking or excessive loss of hide substance. "Vertical weaving" can be caused by using too acid or too astringent tan liquors, or in light leathers by excess acid in chrome-tanning or dyeing. The various defects and conditions are classified and fully illustrated by photomicrographs.

D. WOODROFFE.

[Gelatin compositions for] printing rollers. E. H. RISENFELD and H. WILLSTAEDT (Z. angew. Chem., 1929, 42, 677—682).—The strength of the material used is determined by forming a shaped test piece, in which plates are embedded, by pouring the melted composition into a mould, cooling to room temperature, and keeping for 24 hrs. in an ice chest. The test piece is withdrawn immediately before the trial; hooks attached to the embedded plates serve for suspension and to carry the weights. The extent to which water is absorbed is found to be of no great use as a test, but the extent to which water is given up on exposure to a constant stream of dried air at constant temperature for a definite time is highly characteristic for different compositions. The gel strength of a mixture with 30% of water under standard conditions is also a valuable guide; it diminishes very considerably after heating. The resistance to deformation and the time required to return to normal shape after distortion have been examined, but the results are of a preliminary nature only. The temperature at which the material begins

to flow is measured in the Ubbelohde apparatus (cf. Naumann, B., 1929, 346), and is an important property. Adhesive power is measured by the force required to pull a tablet of given dimensions away from a copper plate on to which it has been previously pressed for a given time at 0° with a definite pressure. Methods for the determination of gelatin, glycerin, sugar, and water have been examined. Gelatin is calculated from the nitrogen content determined by the Kjeldahl method. None of the standard methods gives accurate results for glycerin. The sugar content is determined after complete inversion by the Bertrand method; water is determined by distillation with xylene.

S. I. LEVY.

PATENTS.

Concentration of gelatin and other gelatinising materials. F. J. SHEPHERD, and LIFORD, LTD. (B.P. 313,631, 12.12.27).—Jellies of gelatin or gelatin-containing jellies are shredded or otherwise subdivided, placed in a fabric bag, and/or frozen; the ice is separated by plunging the mass for a limited time into a small quantity of warm water, or by treating the frozen mass with alcohol, or by flotation from the jelly. D. WOODROFFE.

Drying [of hides]. A. W. RANDALL (B.P. 314,738, 17.10.28).

Rendering gelatin insoluble (U.S.P. 1,709,569).—See XXI.

XVI.—AGRICULTURE.

Chernozem and argillaceous soils. M. A. EGOROV and M. M. STRELNIKOVA (Ukraine Chem. J. [Tech.], 1929, 4, 65—82).—Of the two soil types, chernozem is the richer in particles of a diameter larger than 0.25 mm., has the smaller sp. gr., is the richer in humus, nitrogen, phosphorus pentoxide, lime, and ferric oxide than a clay soil, but differs little from it in its contents of silica, alumina, and carbon dioxide. Argillaceous soils decompose hydrogen peroxide much more rapidly than chernozem, but their action on filter paper according to Christiansen's method is equal. A sodium phosphate solution extracts much more organic matter from a chernozem soil, but in the case of an argillaceous soil a greater part of the organic matter belongs to the "crenic and apocrenic acid" fraction. The rate of reversion of phosphorus pentoxide is much greater in the case of chernozem soils. A. FREIMAN.

Significance of the physical and chemical properties of soils in the old and new soil classification. J. HASENBÄUMER (Z. Pflanz. Düng., 1929, 14A, 140—162).—The chemical and mechanical analyses of a large number of soils from different areas are compared with their productivity for individual crops. The extent to which soils can be classified as to their value for particular crops on the basis of these laboratory results is discussed. A. G. POLLARD.

Influences of the development of higher plants on the micro-organisms of the soil. III. Influence of the stage of plant growth on some activities of the organisms. R. L. STARKEY (Soil Sci., 1929, 27, 433—444).—The rate of carbon dioxide production in cropped soils was greater than in fallow soils and was characteristic for each crop. The effect of growing

plants on the carbon dioxide production of soils was paralleled by their effects on bacterial growth (cf. B., 1929, 570), being small at the early stages, reaching a maximum at the period of flowering and fruiting, and subsequently declining. In fallow soils the carbon dioxide production decreases as the growing season advances. The nitrification of soil nitrogen in cropped soils follows the same general course as carbon dioxide production, but the effects on the nitrification of ammonium sulphate were less definitely marked. These accelerated biological processes result from the addition to the soil by the plants of organic matter of narrow carbon : nitrogen ratio. A. G. POLLARD.

Basis of the chemical stimulation of the higher plants. A. NIETHAMMER (Z. Pflanz. Düng., 1929, 14A, 162—169).—The general conceptions of seed stimulation are discussed, with reference to particular substances concerned. Care is necessary to differentiate between true stimulation involving direct effects on the cell plasma, and apparent stimulation which is a secondary phenomenon. Stimulation is not the outcome of differential penetration of the semi-permeable membranes of the seed coating. A. G. POLLARD.

Influence of widening the rows on soil nutrient absorption by the sugar beet. J. SOUČEK (Z. Zuckerind. Czechoslov., 1929, 53, 369—372).—After planting in rows at distances of 35, 45, and 55 cm., the roots were examined at intervals from May to November. The final effect of widening the rows was that the weight both of the roots and of the foliage was distinctly increased, as was also the total weight of dry substance both in roots and leaves. The percentage of sugar in the root on harvesting was highest in the 45 cm. and lowest in the 55 cm. rows, but the total sugar formed in the whole plant was distinctly higher in the 55 cm. rows. The ash content of the whole plant was greatest in the 55 cm. rows. Regarding the nitrogen, P_2O_5 , and Na_2O taken up both by leaves and roots, there was practically no difference for the different rows, though a little more nitrogen was present in the leaves in the 55 cm. than in the 35 cm. rows. J. P. OGILVIE.

Relationship of soil type to the calcium and magnesium content of green bean stems and leaves and of their expressed juices. J. F. FONDER (Soil Sci., 1929, 27, 415—431).—The calcium and magnesium contents of all soil solutions examined, although varying initially to a considerable extent, were reduced to approximately the same value after the growth of a crop of beans. The calcium and magnesium of the bean leaves and stems and the juices expressed from them varied considerably with the nature of the soil. In general, the values tended to increase with the age of the plants and were higher in the leaf tissue than in the stems. The calcium content of the expressed leaf juice was greater than that of the stems. No such regularity occurred in the magnesium content of the juices. Increases in the calcium and magnesium contents of the tissue and juices of leaves and stems were more marked in the early and late periods of growth than intermedially. High calcium content of the soil solution was closely correlated with high calcium content of plants growing in soils of similar texture.

No relationship was apparent between the calcium and magnesium contents of the soil solution and the reaction or texture of the soils. In some cropped soils the calcium and magnesium contents of the solutions recovered their normal values after storage in the air-dry condition for about 80 days. A. G. POLLARD.

Triethanolamine oleate for [insecticide] oil sprays. G. L. HOCKENYOS (Ind. Eng. Chem., 1929, 21, 647—648).—Triethanolamine oleate, made by mixing commercial "triethanolamine" with oleic acid, is used as the basis for the preparation of a miscible oil for oil sprays of the paraffin type. *p*-Dichlorobenzene and carbon tetrachloride may be used in conjunction with such sprays without any deterioration in wetting power or adhesion. A. G. POLLARD.

Drop test of stability of [fungicide] emulsions. G. GOETTSCH (Chem. Weekblad, 1929, 26, 336—338).—The stability of the oil-in-water emulsions used for spraying etc. may be judged by the appearance observed when a drop of the liquid used to form the emulsion is allowed to fall into water under specified conditions. S. I. LEVY.

Classification of [Welsh] soils for purposes of survey. G. W. ROBINSON (Nature, 1929, 123, 980).

Gravitational flow of fertilisers. DEMING and MEHRING.—See I.

PATENTS.

Plant soil. R. H. MORRIS (U.S.P. 1,717,059, 11.6.29. Appl., 20.8.25).—Peat is prepared by distributing alkaline material over the surface and legumes are planted therein. When grown the plants are turned beneath the surface of the bed and the mass is allowed to ferment. After decomposition the material is shredded. A. G. POLLARD.

Manufacture of fertilisers containing nitrogen and phosphoric acid. ELEKTRIZITÄTSWERK LONZA (B.P. 294,117, 16.7.28. Switz., 14.7.27).—Crude phosphates are treated at first at a low temperature and towards the end of the reaction at, e.g., 60—70° with nitric acid of such concentration (preferably 65—75%) that the product contains not more than 2—3 mols. of water per mol. of calcium nitrate. Subsequent decomposition of the product with evolution of oxides of nitrogen is prevented by adding to it not more than 2% of urea. Alternatively, fertilisers prepared in a similar manner but containing a higher proportion of water may be partially dehydrated by treatment with dry air below 40°. L. A. COLES.

Working-up of potash salts (B.P. 314,725). Fertilisers in globular form (B.P. 313,652). Granular materials (B.P. 314,639). Magnesium cyanide compound (B.P. 314,242).—See VII. Aromatic polyhydroxy-compounds containing mercury (B.P. 312,246).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Phase-rule study of the removal of sugar from molasses. I. Ternary system sucrose-water-barium oxide or strontium oxide. K. NISHIZAWA and Y. HACHIHAMA (Z. Elektrochem., 1929, 35, 385—392).—Isotherms of the system involving baryta at 25° and

45° show that the only solid phase besides hydrated baryta and sugar is $\text{BaO}, \text{C}_{12}\text{H}_{22}\text{O}_{11}$ which enters into equilibrium with liquid phases the composition of which varies from 4.0% BaO, 0.16% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ to 1.48% BaO, 31.1% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ at 25°, and from 8.99% BaO, 0.13% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ to 1.25% BaO, 72.52% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ at 45°. At 75°, $3\text{BaO}, \text{C}_{12}\text{H}_{22}\text{O}_{11}$ is the solid phase in equilibrium with liquid phases containing from 34.82% BaO, 0.67% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ to 25.08% BaO, 0.99% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$, at which point $\text{BaO}, \text{C}_{12}\text{H}_{22}\text{O}_{11}$ appears and becomes the stable solid phase until the liquid phase contains 0.48% BaO, 79.06% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$. In the strontia isotherm, $2\text{SrO}, \text{C}_{12}\text{H}_{22}\text{O}_{11}$ exists in equilibrium with liquid phases containing from 5.99% SrO, 0.20% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ to 4.15% SrO, 31.31% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$, and thereafter to 2.26% SrO, 78.63% $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ the solid phase is $\text{SrO}, \text{C}_{12}\text{H}_{22}\text{O}_{11}$. A brief description of the crystal form of the various saccharates obtained is given.

H. T. S. BRITTON.

Purification and decoloration of cane and beet sugar juices by the combined use of sulphur dioxide and activated carbons. G. MEZZADROLI and E. VARETON (*Zymologica*, 1929, 14, 73—104).—Sulphitation of cold sugar juices causes no inversion, and the same is the case at 80° provided that the free acid present is the organic acid of the juice (buffered acidity). Treatment of the cold or hot juice with Norit, even in an acid medium of p_{H} 4—5.2, produces no inversion and diminishes the acidity, provided that the organic acids are adsorbed. Sulphitation and subsequent immediate treatment with Norit does not give rise to inversion, even at 85°, since the Norit adsorbs the acidity and the sulphitation decolorises the juice, renders boiling more regular, prevents formation of froth, and lowers the viscosity. Such treatment does not involve increased consumption of lime for neutralisation, and the effects produced are not annulled by the neutralisation. The results are more marked with dilute than with concentrated juice.

T. H. POPE.

Regeneration of activated carbons [from treatment of beet sugar liquors]. J. KADLEC (*Z. Zuckerind. Czechoslov.*, 1929, 53, 395—399).—Spent decolorising carbon is very irregular in composition, but may contain 16% of mineral matter, of which 24% is soluble in water, and 56% (principally silicates) is insoluble in 2% hydrochloric acid. Part of the salts accumulated during use cannot be completely removed in practice. An example gives the amount of hydrochloric acid (d 1.16) used for regeneration as 124 g. per kg. of carbon.

J. P. OGILVIE.

Determination of roasted sugar beet in admixture with roasted chicory. J. VONDRÁK (*Z. Zuckerind. Czechoslov.*, 1929, 53, 366—368).—During roasting the betaine and choline nitrogen remain unaltered, roasted sugar beet giving about seven times the amount of nitrogen in this form, determined by precipitation with iodine-potassium iodide, as compared with roasted chicory, on which observation the author bases a method for the determination of the former in admixture with the latter.

J. P. OGILVIE.

Application of the [Sandera] conductivity appar-

atus to beet-factory control. K. SANDERA (*Z. Zuckerind. Czechoslov.*, 1929, 53, 378—382).—Using the electrical conductivity apparatus designed by the author (B., 1927, 312, 920), the value in scale divisions for diffusion water was for normal work 12—13, and for a factory in which the waste waters were returned to process 33—34. Diffusion juice was fairly constant between 156 and 164, whilst juice from the single-diffusion vessels rose from 11 to 162. Water from the slices presses gave 18—20, and that from the carbonation scum presses 36—95. Carbonation juice had a value of about 180.

J. P. OGILVIE.

Polarographic measurements for detecting sugar decomposition. K. SANDERA and B. ZIMMERMANN (*Z. Zuckerind. Czechoslov.*, 1929, 53, 383—389).—Heyrovský's polarographic method (A., 1925, ii, 674) was applied to the problem of detecting sugar decomposition on heating in solution or in solid form, and was found highly sensitive in this direction. Curves were obtained indicating that, on heating a 26% solution of sugar from 20° to 100° in a boiling water-bath, decomposition is incipient after 45 min. Sugar crystals heated at 70° showed an alteration which was slight in the dry state but relatively considerable in the presence of moisture.

J. P. OGILVIE.

Mathematical proof of the reliability of the results of the determination of sugar in the sweet slices. J. VONDRÁK (*Z. Zuckerind. Czechoslov.*, 1929, 53, 359—365).—The Gauss method of calculating the true average results from individual deviations when applied to the daily determination of the sugar content of the roots introduced into a beet factory was found in the case of 1414 analyses to give an average result which closely agreed with that actually observed for the campaign.

J. P. OGILVIE.

Molecular constitution of starch and its depolymerisation by heat. A. PICTET (*Woch. Brau.*, 1929, 46, 280—283).—Starch distilled under reduced pressure yields *lavoglucosan*, which is an anhydride of β -glucose. It is also depolymerised when heated with glycerol, yielding, successively, the hexa- and tri-hexosans and α -glucosan. Although the process is reversible, the products of polymerisation differ from those of depolymerisation, di-, tetra-, and octa-*lavoglucosan* being obtained from *lavoglucosan* whilst α -glucosan yields the di- and tetra-hexosans. The hypothesis that the starch molecule contains β -linkings is supported by the fact that at least 30% of *l*-glucosan is obtained from the starch, and that the trihexosan is hydrolysed by emulsin. The production of α -glucosan in the presence of glycerol suggests the contrary, and it is concluded that the resolution of starch under the influence of heat gives no sure indication of the configuration of the glucosan groups. The fact that only molecular association can exist between hexahexosans is considered as an objection to the view of Irvine and others that starch is formed, not by polymerisation, but by an association of either 1, 2, 3, or 6 glucosan groups. Those di-, tri-, and hexa-hexosan polymerides have determined mol. wts. and consist of definite molecules, the atoms of which are bound together by the ordinary valency of the elements. The author finally considers that starch has a high

mol. wt. and is a polyhexosan, i.e., that it is a polymerisation product of a hexosan. C. RANKEN.

Honey. LAMPITT and others; MEES; KRUISHEER.—See XIX.

PATENTS.

Manufacture of dry halogen-calcium-starch preparations. HENKEL & Co., G.M.B.H. (B.P. 294,235, 5.6.28. Ger., 21.7.27. Addn. to B.P. 244,708; B., 1926, 561).—The borax added to the other constituents in the prior patent is replaced by soluble boric acid salts, e.g., sodium metaborate, or by calcium borate, which may be added as such or formed *in situ* by adding calcium hydroxide with the calcium halides and boric acid before solidification. Alternatively, the calcium borate etc. may be added after the remaining constituents have solidified, the mixture being incorporated by passage between rollers. (Cf. B.P. 276,340; B., 1928, 240.)

L. A. COLES.

Simultaneously making sugar and paper pulp from [sugar] cane. H. G. C. FAIRWEATHER. From VAZCANE PROCESS, INC. (B.P. 314,174, 4.5.28).—See U.S.P. 1,688,904—5; B., 1928, 940.

[Cleansing] treatment of sugar cane. R. S. FALKNER (B.P. 314,364, 26.3.28).

Organic phosphate (U.S.P. 1,716,286).—See III.
Cellulose from bagasse (B.P. 287,516).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Proteolytic enzymes of malt. H. LÜERS and L. MALSCH (Woch. Brau., 1929, 46, 265—269, 275—280).—A protease and a peptidase are present in an aqueous solution of green malt. The optimum action of the protease with gelatin as substrate takes place at p_H 4.9—5.0. The action with gelatin is increased by about 50% by the presence of hydrocyanic acid, in which case the activated enzyme has an optimum p_H at 4.6—4.7. There is apparently no relationship between the activation and the time. The peptidase is most active with leucylglycine as substrate at p_H 7.5, and the amount of proteolysis is proportional to the time and to the amount of enzyme present. For the selective adsorption of both enzymes alumina-A is superior to alumina-C_γ or kaolin, the optimal adsorption taking place at p_H 5.0. Alumina adsorbs the protease to a greater extent than it does the peptidase, and adsorbs the protease first from a 20% alcoholic solution. Both enzymes can be elutriated by secondary sodium phosphate, and, with the protease, water alone in some cases is sufficient. By fractional adsorption and elutriation an enzymic solution is obtained containing 12.6% of peptidase and 53.8% of protease. Under p_H 5.0, the peptidase is easily destroyed by the acid medium, and both enzymes are affected by alcohol, the protease being sensitive to very small concentrations. C. RANKEN.

Influence of alcohol vapour and ammonia on the growth of yeast and fungi. P. LINDNER (Woch. Brau., 1929, 46, 283—284).—*Willia belgica* yeast grown in a solution containing 2% of alcohol and mineral nutrients, and into which alcohol vapour and ammonia are led, gives a heavier head of yeast which deposits later on the bottom of the fermentation vessel. In addition, the

yeast cells have a high content of fat. The mycelium of a mucor similarly grown also shows an increased fat secretion. The fat is directly synthesised not from the sugar present in the fermentation medium, but from the resulting alcohol. Thus a yeast rich in glycogen in a drop-culture with pure water shows a secretion of fat in a few days, the fermentation of the glycogen yielding alcohol which in turn is converted into fat. If, however, the water is replaced by yeast water, no fat is formed and the glycogen is used for the synthesis of protein. In this case the protein is directly synthesised not from the sugar and amino-acids, but from the ternary derivatives of the sugar and amino-acid, peptones, etc., which in turn have been synthesised from ammonia. Pyrotartaric acid or glycerol can be used to replace the sugar with equally good results. C. RANKEN.

Composition and determination of barley proteins. II. L. R. BISHOP (J. Inst. Brew., 1929, 35, 316—322; cf. B., 1928, 312).—It is considered that hordein of barley and the "bylin" found by Osborne and Campbell (B., 1896, 665) in malt are identical, and that both hordein and globulin persist throughout malting. The methods for the determination of these proteins in barley have been found satisfactory also when applied to malt. The fineness and evenness of the grinding of the samples are of great importance, as the extraction of the salt-soluble nitrogen markedly increases with a greater fineness of grinding. The hordein nitrogen is affected least, and consequently is more reliable. In a more detailed fractionation of the salt-soluble constituents the copper precipitation previously employed for determining non-protein nitrogen is replaced by the precipitation of the total proteins by a 2.27% solution of trichloroacetic acid, and the clear solution, after centrifuging, is agitated with 2 g. of kaolin. The nitrogen adsorbed on the kaolin represents proteose and peptone nitrogen, whilst the nitrogen in solution is representative of compounds similar to the amino-acids, which are grouped under the term "non-proteins."

C. RANKEN.

Changes undergone by the nitrogenous constituents of barley during malting. I. L. R. BISHOP (J. Inst. Brew., 1929, 35, 323—338).—At the beginning of the malting process immediately after steeping, the two insoluble proteins of the barley endosperm (hordein and glutelin) are broken down at about the same rate to give salt-soluble products; then the rate of disappearance of glutelin falls off. Later, and just prior to the stage at which the malt is kilned, the rate of disappearance of hordein becomes very small and the amount of glutelin may increase slightly. The subsequent increase of the glutelin and the falling off in the rate of disappearance of the hordein may be accounted for by the resynthesis of these proteins in the embryo. During malting, albumin, globulin, and proteose increase but little, the "non-protein" compounds being the chief result of the degradation of the hordein and glutelin. The changes in the nitrogen compounds on the kiln when making pale malts are very slight. Towards the end of the flooring period there is a tendency for a state of balance to be reached between the breakdown of the proteins of the endosperm and their resynthesis in the embryo, with the result

that the same barley in spite of different treatment on the floor yields malts the nitrogen distributions of which are very similar.

C. RANKEN.

Standardisation of the strength of the organism (*Bacterium C*), used in the Chapman biological method for the determination of the preservative power of hops. A. C. CHAPMAN (*J. Inst. Brew.*, 1929, 35, 363).—The tests, which extended over several months and were carried out on different sub-cultures of the original strain, demonstrated the constant virility of the organism. Quantities varying from 0.5 to 2.5 c.c. of a 1% aqueous solution of pure phenol are made up in several test tubes to 10 c.c. with added nutrient agar, and the tubes heated for 10 min. in a steam steriliser. They are then cooled to 40° and inoculated with 3 drops of an 18-hr. broth culture of the organism grown at 37°, and the contents poured into Petri dishes and incubated at 37° for 48 hrs. In all cases the organism grew freely on the 1.5 c.c. plate, whilst its growth was completely inhibited on the plate containing 2 c.c. of the phenol solution.

C. RANKEN.

Identification of hexamethylenetetramine in wines. M. V. IONESCU and C. BODEA (*Bull. Soc. chim.*, 1929, [iv], 45, 466—468).—Treatment of the limpid wine (2—5 c.c.) with 1—2 vols. of 7% aqueous dimethyldihydroresorcinol produces, after 15 min. or on warming, a white crystalline precipitate of methylenebisdimethyldihydroresorcinol, m.p. 187°, when formaldehyde or hexamethylenetetramine is present; this may be identified by its m.p. Natural wines yield no precipitate, but formaldehyde hydrogen sulphite behaves as above. The alcoholic strength of the wine and the concentration of sulphurous acid present do not affect the reaction, which is definite for concentrations of hexamethylenetetramine exceeding 0.1 g. per litre. In rare cases, when traces of acetaldehyde or furfuraldehyde are present, recrystallisation of the precipitate from alcohol will remove the ethylidene-, m.p. 142°, or furylidene-bisdimethyldihydroresorcinol, m.p. 149°, also formed, these remaining in the mother-liquors (cf. A., 1928, 1117).

R. BRIGHTMAN.

PATENTS.

Yeast preparation and its manufacture. R. WILLSTÄTTER and H. SOBOTKA (U.S.P. 1,701,200, 5.2.29. Appl., 19.6.26).—In the processes of the prior patents (U.S.P. 1,538,366 and 1,574,776; B., 1925, 609; 1926, 381) the disaccharides are replaced wholly or in part by dextrose, laevulose, or invert sugar. *E.g.*, 100 pts. of yeast are mixed with 50 pts. of corn syrup or sucrose and liquefied at 40—60°; then 100—150 pts. of syrup or crystalline dextrose, or 100 pts. of malt extract and 50 pts. of lactose, both containing less than 20% of water, are added and the mass is heated in a kneading mill to 85—110°.

R. BRIGHTMAN.

Manufacture of beverages. H. HEUSER, Assr. to UNITED STATES PROCESS CORP. (U.S.P. 1,717,685, 18.6.29. Appl., 8.4.27).—A portion of the beverage of the beer type, which has been condensed, is fermented with yeast and added to a second portion, which has been dealcoholised, in such proportions that the resultant beverage has an alcohol content within the permissible limit.

C. RANKEN.

XIX.—FOODS.

Malted milk. Action of enzymes of malt on milk solids during manufacture. B. G. HARTMANN and F. HILLIG (*J. Assoc. Off. Agric. Chem.*, 1929, 12, 223—238).—In order to obtain the characteristics of genuine malted milk it is necessary to evaporate the mixture of malt-flour infusion and whole milk *in vacuo*. During the manufacturing process only a slight proteolytic digestion occurs. Lipolytic digestion does not occur. Malted milk is diastatically active.

W. J. BOYD.

Kreis reaction for detection of incipient rancidity in cacao butter. T. H. COOKE (*Analyst*, 1929, 54, 411—413).—The Kreis test was found to be useless in this case and did not approach in sensitiveness the faculties of smell and taste of the ordinary observer.

D. G. HEWER.

Dyes as an indication of adulteration in butter. D. HENVILLE and W. M. PAULLEY (*Analyst*, 1929, 54, 413).—Since 97—98% of the margarines examined contained a dye extractable by ammonia, and 94—95% of butter samples did not, the production of a colour on shaking together 10 c.c. of suspected butter fat, 10 c.c. of petroleum spirit, and 10 c.c. of 3% ammonia solution affords a useful indication of adulteration.

D. G. HEWER.

Some scientific aspects of packaging and quick-freezing perishable flesh products. I. More rapid freezing means better preservation. C. BIRDSEYE (*Ind. Eng. Chem.*, 1929, 21, 414—417).—Quick freezing by direct immersion in a liquid refrigerant is preferable to the slow freezing process with a minimum temperature of about -26°, which occupies 12—48 hrs., since there is very little shrinkage due to loss of moisture by evaporation from the surface and the ice crystals formed are too small to cause disruption and compression of the tissues such as occurs in the case of slow-frozen products.

F. R. ENNOS.

Furfuraldehyde and diastase in heated honey. L. H. LAMPITT, E. B. HUGHES, and H. S. ROOKE (*Analyst*, 1929, 54, 381—395).—The tests used for detection of furfuraldehyde were the Fiehe and aniline acetate reactions. A 20-g. sample of honey is dissolved in 20 c.c. of cold water and extracted with 40 c.c. of ether, the ether decanted and evaporated at room temperature, and the residue dissolved in 10 c.c. of ether. To a 2-c.c. portion of this solution are added 2 c.c. of a 1% solution of resorcinol in concentrated hydrochloric acid; a positive test is indicated by a pink coloration in the acid layer, which darkens until after 20 min. there is a deep cherry-red colour at the junction of the acid and ethereal layers. The remainder of the above solution is evaporated at room temperature, and to the residue 2 c.c. of fresh aniline acetate solution (1 c.c. of re-distilled aniline in 4 c.c. of glacial acetic acid) are added. In positive reactions a pink to orange colour appears within 15 min. Both tests are positive with honey containing 5% of commercial invert sugar, which is considered to be present only when thus doubly found. Positive results were only obtained with heated honey when heating had been carried to the point of affecting colour and flavour. Storage of heated honey produced

no development of furfuraldehyde in about 8 months. Heating causes a considerable loss in diastatic activity.

D. G. HEWER.

Determination of honey in honey cake. II. R. T. A. MEES (Chem. Weekblad, 1929, 26, 334—336. Cf. B., 1929, 109; also following abstract.)—The hypothesis that levulose is formed during the baking, when the cake is poor in honey, if the baking powder is alkaline, is confirmed; the suggested minimum levulose content is raised to 25%.

S. I. LEVY.

Determination of honey in honey cake. C. I. KRUISHEER (Chem. Weekblad, 1929, 26, 342—351; cf. preceding abstract.)—The methods of analysis and a very large number of analytical results are set out. Methods are described for determining the extent to which glucose and corn syrup are used in place of honey, and for detecting the presence of artificial invert sugar in honey cake.

S. I. LEVY.

Gossypol: a progress report. E. P. CLARK (Oil & Fat Ind., 1929, 6, [7], 15—19).—The work of the Food Research Division of the U.S. Dept. of Agriculture on gossypol and its derivatives is summarised (cf. A., 1928, 208, 426, 614, 1016). The study of the toxic effect of gossypol on rats is continued: it is concluded that the ready response of the rats constitutes a means of accurately measuring the free gossypol content of cottonseed meals: bound gossypol *per se* appears to have no influence on the growth-rate of rats. Inferior growth rates were shown on a diet containing autoclaved (Menaul process) meal, compared with untreated meals; growth was accelerated by a protein (*e.g.*, casein) supplement, as also by a suitable mineral supplement, indicating that the proper method of utilising cottonseed meal would be as a protein supplement to other feeding-stuffs, a correct balance being preserved between carbohydrate, fat, protein, and mineral constituents.

E. LEWKOWITSCH.

Solubility of metals in milk. QUAM.—See X. **Determination of roasted sugar beet in roasted chicory.** VONDRÁK.—See XVII.

PATENTS.

Improvement of corn. E. SCHLECK (B.P. 313,979, 20.1.28).—Corn for milling is moistened and then subjected to the action of an electric current.

E. B. HUGHES.

Preservation of eggs. C. H. GREEN (B.P. 314,273, 12.9.28).—Eggs are shelled, mixed with 5—10% of glucose, and dried at 15—21°. It is claimed that the product when dissolved in water has the ordinary viscosity and culinary and baking properties of eggs.

E. B. HUGHES.

Manufacture of casein. W. H. SHEFFIELD (U.S.P. 1,716,799, 11.6.29. Appl., 21.8.25).—Acid is added to milk, under conditions which give "gran curd," and the reaction is completed while the mixture passes, with agitation, along a confined path, *e.g.*, a screw-conveyor. The whey is then separated without allowing the curd to settle, and the latter agitated and washed with water as it passes along another closed path.

F. G. CLARKE.

Removal of bleaching chemicals from fruit or the like. S. BLUMENTHAL (U.S.P. 1,717,869, 18.6.29. Appl., 7.12.26).—The fruit is stirred in a solution of

potassium chlorate, drained, and washed in water to remove sulphur dioxide used for bleaching purposes.

E. B. HUGHES.

Artificial sausage casings. A. J. STEPHENS. From N. V. NEDERLANDSCHE PARASINFABR. (B.P. 313,747, 11.6.28).

Freezing of colloidal liquids (B.P. 288,310).—See I.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of mercury in certain pharmaceutical products. A. IONESCU-MATIU and A. POPESCU (J. Pharm. Chim., 1929, [viii], 9, 570—580).—The methods are based on the precipitation of mercuric sulphate by sodium nitroprusside and the observation of the volume of 0.1N-sodium chloride required to effect redissolution.

F. C. HAPFOLD.

Determination of total geraniol in citronella oil. J. ZIMMERMANN (Chem. Weekblad, 1929, 26, 389—391).—Under ordinary conditions of acetylation citronella yields only the diacetate. Discrepancies in the analysis of citronella oil are ascribed to the presence of water and sulphuric acid in the reagents, and to variations of concentration of the alcoholic alkali solutions employed. The fraction of the oil capable of being acetylated may be determined within about 2% from the difference between the esterification numbers of the original and acetylated oils.

H. F. GILLBE.

PATENTS.

Pharmaceutical. M. L. CROSSLEY, Assr. to CALCO CHEM. Co. (U.S.P. 1,716,686, 11.6.29. Appl., 24.3.27).—A magnesium compound is added to a preparation containing cincophen to increase the therapeutic properties of the latter.

F. G. CLARKE.

Manufacture of therapeutic calcium preparations. G. B. ELLIS. From CHEM. WORKS, FORMERLY SANDOZ (B.P. 314,460, 27.3.28).—A preparation for intramuscular injection comprises a concentrated aqueous calcium gluconate solution introduced hot into ampullæ etc. which are hermetically sealed and repeatedly heated and cooled until no further crystallisation takes place.

L. A. COLES.

Solution of hypnotics and sedatives and solvents therefor. W. KROPP, Assr., to WINTHROP CHEM. Co., Inc. (U.S.P. 1,707,863, 2.4.29. Appl., 6.7.25. Ger., 1.9.24).—Difficultly soluble barbituric acid hypnotics or other insoluble medicinals are dissolved in water by mixing with a soluble amide of a lower fatty acid and a soluble urethane.

R. BRIGHTMAN.

Manufacture of a therapeutically active substance from the anterior lobes of the pituitary body. SCHERING-KARLBAUM A.-G. (B.P. 288,132, 24.2.28. Ger., 31.3.27).—The finely-divided fresh or dried glands are extracted with (alkaline) Ringer solution or with 0.1N-sodium hydroxide. The extract, after removal of albumin, may be evaporated or salted out. A solution of the product produces a premature puberty in animals.

C. HOLLINS.

Production of pills and pastilles for the small intestine. S. HERMANN, and PHARMACEUT. WERKE "NORGINE" A.-G. (B.P. 285,091 and 288,542, [A] 10.2. and [B] 11.2.28. Ger., [A] 12.2. and [B] 11.4.27).—The pills etc. are coated with material unattacked by the

gastric juices, but readily removed by the intestinal juices, comprising (A) mixtures of stearic acid with fatty acids of lower m.p., *e.g.*, palmitic acid, or (B) fatty acids of lower m.p. than stearic acid or mixtures of these acids.

L. A. COLES.

Materials for treatment of wounds. MARIA GRÄFIN VON LINDEN (B.P. 285,464, 17.2.28. Ger., 19.2.27).—Muslin, wadding, surgical sewing silk, catgut, etc. are impregnated with 0.1–1.0% of aqueous copper sulphate, copper glyceride, copper dimethylglycine, or the last-named with 0.1% of methylene-blue, whereby the material is sterilised and acquires germicidal properties.

C. HOLLINS.

Anæsthetic gas and its manufacture. M. B. CHENEY, Assr. to CHENEY CHEM. CO. (U.S.P. 1,712,250, 7.5.29. Appl., 29.10.25).—A mixture of 75% by vol. of pure ethylene with 25% of ether vapour is compressed under a pressure of more than 1000 lb./in.²

A. R. POWELL.

Manufacture of thymol. SCHERING-KAHLBAUM A.-G. (B.P. 285,833, 20.2.28. Ger., 22.2.27. Addn. to B.P. 273,684 and 279,856; B., 1929, 236, 316).—Thymol, m.p. 51°, b.p. 234° or 115–118°/12 mm., is separated from *p*-thymol, b.p. 230° or 108–110°/12 mm., in the products of the prior processes by fractional vacuum distillation followed by freezing out.

C. HOLLINS.

Acridine derivatives. G. B. ELLIS. From CHEM. FABR. VORM. SANDOZ (B.P. 283,184, 6.1.28).—Salts of amino- or diamino-acridines with bile acids (other than deoxycholic acid) have strong antiparasitic properties. 3:7-Diamino-5-methylacridinium cholate, and 7:10-diamino-2-ethoxyacridine cholate, *apocholate*, and *cholate* are prepared by mixing aqueous solutions of the sodium salt of the acid and the hydrochloride of the base.

C. HOLLINS.

Manufacture of dihydromorphine. A. BOEHRINGER (C. H. BOEHRINGER SOHN) (B.P. 285,404, 28.1.28. Ger., 15.2.27).—Tetrahydrothebaine, preferably freed from dihydrothebaine and dihydrothebainone, is dealkylated by treatment with boiling 47% hydrobromic acid to give dihydromorphine.

C. HOLLINS.

Manufacture of complex [organic] metallic salts. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 313,541, 14.2.28).—The complex metal compounds of B.P. 295,734 and 213,285 (B., 1928, 799; 1925, 691) are oxidised, *e.g.*, by hydrogen peroxide or air, to give other therapeutic substances. The oxidation of solutions of potassium antimonyl-stanni- and manganipyrocatecholdisulphonates, potassium antimonylpyrogallodisulphonate, diethylamine antimonylpyrocatechuate, and sodium stanniprotocatechuate is described.

C. HOLLINS.

Manufacture of organic antimony compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 313,058, 1.2.28).—A diazo compound and antimony chloride trichloride are added to a tertiary base (pyridine) and the mixture is warmed to 60° with production of the compound, $\text{ArSbCl}_4 \cdot x\text{C}_5\text{H}_5\text{N}$. On addition of water the arylstibinic acid is precipitated. The preparation of benzenestibinic acid and its 3-chloro-4-acetamidoderivative is described.

C. HOLLINS.

Manufacture of stable solutions of salts of stibinic acids. A. CARPMAEL. From I. G. FARBENIND. A.-G.

(B.P. 311,748, 15.2.28).—Salts of organic stibinic acids are stabilised in solution by addition of a multivalent alcohol (glycerol).

C. HOLLINS.

Production of solutions of quinine base adapted for therapeutic use. F. B. DEHN. From CHEM. PHARM. A.-G. (B.P. 312,253, 21.2.28).—See U.S.P. 1,692,900; B., 1929, 453.

Extraction of theobromine from natural products. G. C. A. VAN DORP, Assr. to N. V. SOC. CHEM. IND. "KATWIJK" (U.S.P. 1,718,093, 18.6.29. Appl., 15.3.28. Holl., 22.3.27).—See B.P. 287,507; B., 1929, 149.

Manufacture of [mixed] alkaloid salts. W. SCHOELLER and H. SCHOTTE, Assrs. to CHEM. FABR. AUF ACTIEN, VORM. E. SCHERING (U.S.P. 1,717,585, 18.6.29. Appl., 20.6.27. Ger., 26.6.26).—See B.P. 273,279; B., 1928, 465.

Freezing of colloidal liquids (B.P. 288,310).—See I. Quinazoline derivatives (B.P. 287,179). Di(halo-*genoacylated*) diphenyl ethers (B.P. 286,688).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Mechanical actions on the photographic plate.

A. REYCHLER (Bull. Soc. chim. Belg., 1929, 38, 168–171).—The rubbing of the sensitive silver bromide emulsion on a photographic plate with rods of silver, copper, zinc, glass, or with the finger-nail, skeins of silk, etc., all produced dark markings when the plate was developed irrespective of the chemical nature of the rubbing agent, but depending on the sensitivity of the emulsion. A suggested interpretation of these results is that the friction removes the protective gelatin coating from a molecule of silver bromide, which thus becomes susceptible to reduction by the developer, the resultant grain of silver then forming one pole of an electric couple, round which further reduction takes place. The effect of light, though similar in its results, involves a different mechanism.

B. W. ANDERSON.

PATENTS.

Rendering gelatin insoluble and its application to various arts [photography]. S. DE PROCOUDINE-GORSKY and N. POZNIKOV, Assrs. to SOC. DE PHOTOCHIMIE "ELKA" (U.S.P. 1,709,569, 16.4.29. Appl., 11.11.25. Fr., 19.11.24).—Ripened silver bromide produced by adding 10% silver nitrate solution to an 18% solution of potassium bromide containing 0.75% of gelatin and 15% of alcohol, centrifuging, and washing is mixed in a moist state with photographic gelatin at 45°, and after thorough agitation the mixture is used for coating plates, films, etc. (cf. G.P. 445,513; B., 1928, 427).

A. R. POWELL.

Production of coloured designs and application thereof to manufacture of woven fabrics. F. TWYMAN, C. F. SMITH, and A. HILGER, LTD. (B.P. 314,359, 23.3.28. Addn. to B.P. 227,072).

Pattern effects on materials (B.P. 310,773).—See VI. Concentration of gelatin (B.P. 313,631).—See XV.

XXII.—EXPLOSIVES; MATCHES.

Vapour pressures of glycerol trinitrate and certain glycol dinitrates. W. DEC. CRATER (Ind. Eng. Chem., 1929, 21, 674–676).—Vapour pressures were

determined over the temperature range 15–55° by the dynamical method using carefully prepared materials. The figures obtained for nitroglycerin were higher than those found by previous investigators (0.0015 mm. at 20°). The following are in order of increasing vapour pressure: diethylene glycol dinitrate (0.0040 mm. at 20°), trimethylene glycol dinitrate, ethylene glycol dinitrate, propylene glycol dinitrate. C. IRWIN.

PATENTS.

Production of 2:4:6-trinitro-1:3:5-triazidobenzene. O. TUREK (B.P. 298,981, 14.9.28. Ger., 18.10.27).—1:3:5-Trichloro-2:4:6-trinitrobenzene in acetone is added to potassium azide in aqueous alcoholic acetone, acetone equivalent to the amount added being removed in a vacuum and the temperature maintained at 20–30°. 2:4:6-Trinitro-1:3:5-triazidobenzene, m.p. 131° (decomp. into hexanitrosobenzene), is an explosive (cf. B.P. 298,629, following abstract).

C. HOLLINS.

Explosive charges for detonators, percussion caps, boosters, detonating fuses, projectiles, etc. O. TUREK (B.P. 298,629, 10.9.28. Czecho-Slov., 13.10.27).—The charge is composed of 2:4:6-trinitro-1:3:5-triazidobenzene, either alone or with other explosives. In detonators it is used in a compressed state as the main charge, being covered by a layer of an azide of a heavy metal, e.g., lead azide, which in turn may be covered by a further layer of the main constituent (cf. B.P. 298,981, preceding abstract). S. BINNING.

Blasting cartridges. E. C. R. MARKS. From SAFETY MINING CO. (B.P. 313,333, 5.7.28).—A blasting cartridge containing liquid carbon dioxide is described. S. BINNING.

Waterproofing [of cartridges]. S. L. WILLIS and W. H. WOODFORD, Assrs. to REMINGTON ARMS CO., INC. (U.S.P. 1,701,868, 12.2.29. Appl., 12.5.25).—A composition obtained, e.g., by heating 37.5 pts. of tung oil, 1 pt. of manganese resinate, and 8 pts. of lead oxide at 220° and adding 95 pts. of carbon tetrachloride and a small amount of tung oil, is used for sealing and impregnating the top wad and crimp of loaded paper-shot cartridges. R. BRIGHTMAN.

XXIII.—SANITATION; WATER PURIFICATION.

Purification and sterilisation of water by superchlorination. R. ADLER (Gas- u. Wasserfach, 1929, 72, 675–678).—The superchlorination process requires that after the expiration of 30 min. an excess of 1 mg./litre of chlorine shall be present, and that this excess shall never fall below 0.3 mg./litre. It is added before filtration and effects also the entire removal of iron. In order to facilitate the formation of hypochlorous acid (which is the active compound) waters containing much organic matter are passed after filtration through manganese dioxide. This treatment also removes manganese salts if such are present. The excess chlorine is removed by activated carbon, the reaction $C + 2H_2O + 2Cl_2 = 4HCl + CO_2$ occurring in the adsorbed film. The satisfactory carrying out of this treatment depends on a fixed relation between the carbon surface and water velocity. The carbon surface is adversely affected by colloids whether originally present in the water or resulting from the chlorination. They can be removed

by periodical washing with water to which alkali has been added. The adsorbed alkali is rendered innocuous by treatment with calcium chloride solution. If phenols are present in the water the objectionable odour of chlorophenols is destroyed by the carbon treatment.

C. IRWIN.

PATENTS.

Non-poisonous colouring matters for cosmetics. J. CULMANN and E. AHRENS, Assr. to G. SIEGLE CORP. OF AMERICA, INC. (U.S.P. 1,702,227, 12.2.29. Appl., 20.6.27).—The red aluminium lake of eosin is used alone or in admixtures with Lake Red C or the aluminium lake of rhodamine. R. BRIGHTMAN.

Fumigant. W. MOORE (U.S.P. 1,702,168, 12.2.29. Appl., 3.2.26).—Ammonium carbonate (1 pt.) is dispersed in *p*-dichlorobenzene (4 pts.) and naphthalene (0.5 pt.). R. BRIGHTMAN.

Toxic agent. E. P. KAMPA and P. N. HYMAN (U.S.P. 1,707,723, 2.4.29. Appl., 6.6.27).—A solution of tetrachlorodinitroethane in ethylene dichloride or other volatile hydrocarbon solvent is used as fumigant. R. BRIGHTMAN.

Sludge-digesting apparatus. J. R. DOWNES, Assr. to S. F. MILLER (U.S.P. 1,717,100, 11.6.29. Appl., 12.8.26).—The digesting sludge supports a floating cover fitting fairly closely to the inside of the tank. The edge of the cover projects downwards into the sludge, and the underside slopes upwards from the edge to the centre where a gas-collecting chamber is fitted.

C. JEPSON.

Subdivided activated sludge system for sewage treatment. K. IMHOFF (U.S.P. 1,717,780, 18.6.29. Appl., 18.6.27. Ger., 26.4.26).—Sewage is purified in separate stages by passing it in succession through a series of tanks in which aeration and settlement tanks are combined in one unit. The aeration is obtained by using submerged paddles, and the currents produced help to recirculate the settled sludge. C. JEPSON.

Manufacture of aromatic polyhydroxy-compounds containing mercury. A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 312,246, 18.1.28).—A mixture of mercuric chloride and a polyhydroxybenzene (resorcinol, 2:6-dihydroxytoluene, pyrogallol) is dissolved in water, especially in presence of sodium or ammonium chloride, and acidity is neutralised by addition of sodium carbonate. The neutral solution may be used directly as a disinfectant, insecticide, or fungicide, or may be evaporated to dryness. C. HOLLINS.

Preventing the formation of [boiler] scale. J. ALSBERG, Assr. to SUPERHEATER CO. (U.S.P. 1,717,905, 18.6.29. Appl., 14.8.24).—The precipitation of crystalline scale-forming materials in the preheater, prior to introduction into the boiler, is prevented by the addition of a colloidal chestnut extract to the cold feed water.

C. JEPSON.

Apparatus for testing the chlorine content of water, and for like purposes. W. PATERSON (B.P. 314,155, 13.4.28).—Determinations are made by comparing by transmitted light the coloration produced, e.g., by *o*-tolidine and chlorine, with a series of previously prepared, coloured glass slips corresponding to varying proportions of the reagents. C. JEPSON.

Filtering material (U.S.P. 1,702,104).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 13, 1929.

I.—GENERAL; PLANT; MACHINERY.

Rapid determination of dust in air. L. C. MCNAIR and J. F. HIRST (J.S.C.I., 1929, 48, 127 T).—The method depends on the retention of the dust in sugar and the subsequent measurement in a centrifuge tube.

Registering manometer for permanent control. R. GUILLERY (Rev. Mét., 1929, 26, 329—333).—The apparatus is described with reference to photographs and scale plans, with full details for which the original must be consulted. A. R. POWELL.

PATENTS.

Apparatus for continuous chemical, heat, or other treatment of materials. E. HAYWARD (B.P. 314,916, 5.3.28).—In the autoclave with worm conveyor for solid materials described in B.P. 295,975 (B., 1928, 814) the worm is not provided with a through shaft, but is divided into sections with dog-clutch teeth, the sections resting on the bottom of the autoclave tube and driving each other. Only one stuffing box need be provided for the short driving shaft at one end. Slide valves are described for regulating the admission and outlet of material. B. M. VENABLES.

Operating industrial furnaces and furnaces adapted thereto. R. WARSITZ (B.P. 292,146, 14.6.28. Ger., 15.6.27).—A reverberatory or similar furnace is heated largely by radiation from a mass of gas-freed solid fuel at one side of the hearth, and streams of air and/or gas are forced through the solid fuel from several directions to maintain a central zone opposite the work chamber at a white heat, the solid fuel acting as a surface-combustion agent and also as a combustible. Above the white-hot zone the preheating and removal of volatile matter take place; below it the fuel is consumed by the lowermost supply of air, preferably with formation of carbon monoxide. After passing over the goods the gases leave through another mass of solid fuel which also radiates heat to the work chamber and causes the formation of water-gas from any water vapour present in the products of combustion. The water-gas is reburnt by another supply of air immediately after leaving the mass of fuel, and the heat produced utilised in a regenerator or recuperator. B. M. VENABLES.

Preventing formation of deposits on heat-exchange apparatus exposed to hot gases. LABORATOIRE DE PERFECT. THERMIQUES, Assees. of P. E. J. J. COUTURAUD (B.P. 284,218, 24.1.28. Fr., 24.1.27).—The fire side of the tubes etc. is painted with a mixture of graphite and a fire-resisting heat-conductive binder such as a soluble silicate. B. M. VENABLES.

Linings for furnaces, kilns, and the like. S. TRAVIS, and CALLOW ROCK LIME CO., LTD. (B.P. 314,772,

27.3.28).—In a shaft furnace the lining above and below the zone where comparatively rapid destruction of bricks takes place is in a single thick layer of bricks or blocks, but the destructive zone has a double layer, to enable the inner one to be renewed without collapse of the other zones. Sight-holes are formed in the outer layer in order to determine the condition of the inner layer. B. M. VENABLES.

Cooling drums. W. G. SCHRÖDER (B.P. 290,976, 9.5.28. Ger., 21.5.27).—The drum is double-walled, the cooling agent flowing in the annular space, which is provided with obstacles such as chain links, rods with collars, vanes, strips, etc. to cause the medium to become agitated without materially increasing the resistance to its flow. The inner and outer walls of the drum may also have relative rotation with the same object. B. M. VENABLES.

Drying apparatus. A. W. F. CAPPS (B.P. 314,907; 5. and 9.1.28).—The air or other drying medium first passes over the material to be dried (preferably counter-current) in a compartment on one side of a heat exchanger and then returns on the other side of the heat exchanger so that moisture abstracted from the material is condensed on the return pass and gives up its latent heat to the air in the drying compartment. The air is heated initially to start the drying, and also continuously, preferably between the two passes, to make up for losses. B. M. VENABLES.

Dryer. P. T. LINDHARD, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,718,243, 25.6.29. Appl., 26.1.28).—The material passes downwards through the annular spaces left between two series of superposed conical baffles, the outer series being inverted and truncated. Means are provided to adjust one series vertically with respect to the other, and to give them relative movement horizontally. The drying medium passes through the material as it works downwards. B. M. VENABLES.

Drying chambers with heating floor. SOC. ANON. DE COMMENTRY, FOURCHAMBAULT & DECAZEVILLE (B.P. 296,440, 25.8.28. Fr., 1.9.27).—The combustion chamber and flues are below the floor of the drying chamber. The flues (not less than two) are arranged symmetrically about the longer axis of the chamber and the gas passes from them into the chamber. B. M. VENABLES.

Manufacture of finely-distributed or colloidal mixtures of bodies insoluble in one another [metals and oils]. M. POLANYI and S. VON BOGDANDY (B.P. 286,316, 3.3.28. Ger., 4.3.27).—One substance is vaporised or cathodically atomised and brought into contact with the other, liquid, substance of which the surface is continually being renewed, preferably by allowing it to

circulate through a centrifugal bowl to the centre of which the atomised or vaporised substance is admitted. The process is described as applied to the production of colloidal suspensions of metals in oils.

B. M. VENABLES.

Filter element. G. J. LIPSCOMB (U.S.P. 1,716,786, 11.6.29. Appl., 10.5.28).—A cartridge for a vacuum filter is composed of a number of annular filtering elements each comprising a sandwich of a pair of perforated sheets and a filtering material, the former having projections which embed in the latter.

B. M. VENABLES.

Mixer. L. LAING (U.S.P. 1,718,745, 25.6.29. Appl., 1.6.27).—A receptacle is provided with a vertical central stirring shaft carrying mixing blades which rotate near the bottom, and a stem near the wall carries a baffle blade which can be adjusted vertically.

B. M. VENABLES.

Mixing foam with plastic materials. E. M. RICKARD and J. A. RICE, Assrs. to BUBBLESTONE CO. (U.S.P. 1,716,932, 11.6.29. Appl., 26.10.26).—A number of helical agitators are arranged on a rotating shaft within a container. At any point of the shaft there are two helices of different diameter and opposite hand, and at intervals along the shaft the hand reverses, *i.e.*, an inner helix is the same hand as the outer one next along the shaft.

B. M. VENABLES.

Vacuum distillation. C. R. BURCH, F. E. BANCROFT, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 315,186, 19.7.28).—In a high-vacuum distilling apparatus as described in B.P. 303,078 (B., 1929, 269) the evaporation takes place from heated trays which are nested to form their own baffle (there is an extra heated baffle at the top) so that drops of distilland have no opportunity of reaching the condensing surface, which is the walls of the vessel. The distillate vapour leaves the edges of the trays in an upward direction, which assists the complete evacuation of the lower part of the chamber. [Stat. ref.]

B. M. VENABLES.

Fractionating column. R. E. WILSON, R. D. HUNNEMAN, W. H. BAHLKE, and F. M. ROGERS, Assrs. to STANDARD OIL CO. (U.S.P. 1,716,939, 11.6.29. Appl., 31.1.25).—A tower is provided with internal angle rings to support the fractionating plates and with openings in the shell to insert the plates, which are in sections and bolted together through downwardly depending flanges.

B. M. VENABLES.

Deodorisation or like treatment of fumes and gases. J. W. VERNON (B.P. 314,187, 16.5.28).—Gases, such as those evolved in the production of artificial silk, containing hydrogen sulphide or other sulphur compounds, are treated with sulphuric acid of concentration not less than 82%, preferably in the form of a mist or spray. The gases may first be washed with water at 95°, and subsequently treated with the acid, which may either be cold or may also be heated to 80–95°. After removal of the sulphur by filtration the acid is concentrated if necessary and returned for further use. Coal gases may be similarly treated, the unsaturated hydrocarbons other than ethylene being

first removed by sulphuric acid of lower concentration than 82%. A. B. MANNING. ³

Washing of gases and vapours. I. G. FARBERIND. A.-G. (B.P. 293,003, 26.6.28. Ger., 29.6.27).—A wetting agent (0.1–0.2%) is added to the aqueous washing liquor. C. HOLLINS.

Carrying out exothermic chemical reactions under pressure and at a high temperature. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, Assces. of SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE ET PROD. CHIM.) (B.P. 305,534, 23.1.29. Fr., 6.6.28).—In the apparatus described in B.P. 268,721 (B., 1927, 688) the gases after having served as a heat-protecting screen to the pressure-resisting wall are brought out of the apparatus and further heated before returning to the reaction zone. The application of the invention to ammonia synthesis is claimed.

B. M. VENABLES.

Laboratory apparatus for heating or evaporating liquids. H. J. S. SAND, and BROWN & SON (ALEMBIC WORKS), LTD. (B.P. 314,923, 2.4.28).—A still, water oven, or similar laboratory apparatus is made in two parts so that the lower heated part (water bath) may be easily cleaned or repaired. The upper rim of the lower part is in the form of a channel with mouth upwards, and the lower edge of the upper part is inserted into this. The channel forms a water seal kept replenished by condensate running down from the upper part. The outer wall of the channel is higher than the inner, so that excess condensate runs back to the water bath.

B. M. VENABLES.

Fire-extinguishing compound. CLAYTON INSTALLATIONS, LTD., and J. E. HACKFORD (B.P. 315,123, 10.8.28).—A mixture of sodium sulphite or metabisulphite with sodium bicarbonate, in about equal proportions, is added to an acid solution containing, *e.g.*, 10% of alum and 1% of sulphuric acid, with a foaming agent.

H. ROYAL-DAWSON.

Crushing or pulverising machine. W. R. HUME (U.S.P. 1,719,549, 2.7.29. Appl., 18.7.27. Austral., 9.10.26).—See B.P. 289,721; B., 1928, 506.

Filtering or like device. H. A. THOMPSON, Assr. to SWINNEY BROS., LTD. (U.S.P. 1,719,346, 2.7.29. Appl., 12.1.29. U.K., 26.10.28).—See B.P. 308,166; B., 1929, 499.

Absorption refrigerating machines. SULZER FRÈRES SOC. ANON. (B.P. 295,371, 14.5.28. Switz., 11.8.27).

Bearings for spindles of centrifugal separators. AKTIEBOLAGET SEPARATOR (B.P. 299,977, 24.10.28. Swed., 3.11.27).

Driving means for mixing or similar apparatus for liquids. M. H. CARPMAEL. From SNIA-VISCOSE (B.P. 315,164, 24.10.28).

Separation of dust from boiler flue gases. J. T. BARON and J. B. CLARKE (B.P. 314,886, 30.3.28).

Separating liquids (U.S.P. 1,716,957).—See III. **Leaching solid materials** (B.P. 301,306).—See VII. **Melting finely-divided materials** (B.P. 297,813).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Relation between bitumen content, caking power, and structure of bituminous coals. W. MÜHLSTEDT (Brennstoff-Chem., 1929, 10, 241—247).—A number of coals have been submitted to the Bochum crucible test, and their caking indices (Kattwinkel, B., 1926, 257) and oily and solid bitumen contents (Fischer, B., 1925, 233) have been determined. Each method of examination revealed differences between the coals, but no definite relationship between the different series of results could be recognised. It was, for example, impossible to relate the caking index of a coal or of a blend of a coal with a non-caking material with its bitumen content. Other factors, the mode of action of which could not be determined by the methods employed, play an important part in the coking process. The coals have also been separated into fractions of increasing sp. gr., and these examined separately. In general the caking power of the coal fell off with a decrease in the proportion of the lightest constituents. There were, however, exceptions to this rule, due principally to the different caking power of these constituents in different coals. Extraction of coal with benzene brought about a very considerable decrease in the proportion of lighter constituents; the fraction of $d < 1.28$ almost completely disappeared. Oxidation of the coal at 120° had the same effect, but to a small degree, and also decreased the amount of benzene-soluble bitumens. Owing to the varying ash content of "dull" coal, an accurate separation of "bright" coal, "dull" coal, and fusain on the basis of sp. gr. differences was not possible. The influence of the various factors which have been investigated on the coking process is discussed. A. B. MANNING.

Evaluation of bituminous coals for gas works from their analyses. R. MEZGER (Brennstoff-Chem., 1929, 10, 237—241).—Proximate and ultimate analyses and determinations of the calorific value must be supplemented by other data in order to assess accurately the value of a coal for gas production. Large-scale experimental retorts have the disadvantage of high initial cost as well as the large quantity of coal and the length of time necessary for a test. Laboratory methods of assaying coal for gas-works purposes are divisible into two classes, (a) those in which all the products of carbonisation are separately determined, and (b) those in which only the volume and calorific value of the gas and the yields of coke and tar are determined. Of the former that due to Schlöpfer (Monats-Bull. Schweiz. Ver. Gas- Wasserf., 1929, 1, 5; 2, 47), and of the latter that due to Geipert (B., 1926, 938), are recommended. A. B. MANNING.

Determinations of ignition points of cokes. G. AGDE and M. SCHNITSPAHN (Brennstoff-Chem., 1929, 10, 257—261, 282—287; cf. B., 1927, 930).—A series of cokes have been prepared by carbonising two coals at temperatures ranging from 500° to 1100° and at two different rates of heating, and determinations have been made of their ignition points, the temperatures at which they begin to reduce carbon dioxide (reduction point), and their adsorptive capacities for hydrogen and carbon dioxide. The ignition points were deter-

mined by passing air at 15 litres/hr. through 2 c.c. of coke, of 2—3 mm. particle size, in a quartz tube the temperature of which was raised at 5°/min. The point at which the rise in temperature of the coke showed a sudden increase was taken as the ignition point. Certain of the cokes produced at 900—1100° showed no ignition point. This was due to the presence of an insufficient amount of easily accessible amorphous carbon, as is shown by the observed relationship between the ignition points and the amounts of gas adsorbed by the cokes, which give a measure of the available amorphous carbon. No difficulties arose in the determinations of the reduction points, which require the presence of only very small quantities of available amorphous carbon in order to give consistent results. A. B. MANNING.

Effect of pre-oxidation on primary distillation products of coal. VI. The tars. J. T. DONNELLY, C. H. FOOT, and J. REILLY (J.S.C.I., 1929, 48, 101—105 r).—Coal in batches varying from 700 to 800 g. was oxidised at 190° and the products of oxidation were collected and determined. Samples of coal were oxidised to varying degrees of oxidation, depending on the "total oxygen used"; the "total oxygen used" was calculated from the weights of carbon dioxide and monoxide and water formed, thus: "total oxygen used" = increase in wt. of coal + wt. CO₂ + wt. CO + wt. H₂O. Distillations at 600° were carried out on the oxidised samples and on untreated coal, the resulting tars being accurately determined. The tars were then compared by a method of selective solvent extraction. The results show that pre-oxidation seriously influences the tar yield, gradually destroying the heavier tar and causing more gas and volatile tar to be formed.

Transformation of methane or coke-oven gas by electric discharges under reduced pressure. F. FISCHER and K. PETERS (Brennstoff-Chem., 1929, 10, 108—113).—By subjecting methane to a high-tension electric discharge at 15—50 mm. pressure it is almost completely converted into acetylene. If undiluted methane is used some carbon deposition occurs and the discharge tube is soon short-circuited. It is preferable therefore to use a diluted gas, e.g., coke-oven gas. With the latter (CH₄ 23.4%, H₂ 52.3%) the optimum yields of acetylene, up to 9.2% in the exit gases, were produced at 40—50 mm. pressure in a cylindrical tube with the electrodes 40—50 cm. apart, using a transformer giving 5—7 kilovolts as a source of potential. The gas was admitted at both ends of the discharge tube and withdrawn from the centre, the gas current thereby helping to cool the electrodes and prevent any deposition of carbon thereon. The efficiency of conversion, expressed as kw.-hrs./m.³ of acetylene produced, fell with the rate of passage of the gas from 178 at 58 litres/hr. to 31 at 367 litres/hr.

A. B. MANNING.

Formation of liquid hydrocarbons from acetylene. I. Polymerisation of acetylene. F. FISCHER, F. BANGERT, and H. PICHLER (Brennstoff-Chem., 1929, 10, 279—282).—The passage of acetylene over activated charcoal or silica gel at 600—700° leads at first to the formation of hydrogen, methane, and carbon. The carbon, which is deposited on the material used, appears

to form a suitable condensation catalyst, for the production of liquid hydrocarbons increases with time of passage of the gas until 70% of the acetylene is converted into liquid products (cf. Zelinski, B., 1924, 242). The ratio of light oil to heavy oil in the product is approximately 1:1. The light oil (boiling up to 150°) contains no paraffins, 70% of aromatic and 30% of unsaturated hydrocarbons. With diluted acetylene, such as can be produced from coke-oven gas (cf. preceding abstract), the total yield is smaller (40%), although that of the light oil fraction remains about the same as with the undiluted gas. Addition of carbon dioxide to the gas favours the formation of the lighter oil at the expense of the heavier. The life of the catalyst is lengthened, and an exhausted catalyst can be regenerated, by the addition of carbon dioxide to the acetylene. By the use of acetylene-carbon dioxide mixtures even with an empty tube 70% of the acetylene can be converted into oil. The efficiency of the process is not increased by the use of lower pressures.

A. B. MANNING.

Detection of coal-tar pitch in natural and petroleum asphalt mixed with colophony. F. J. NELLENSTEYN and J. C. M. SAUERBIER (Z. angew. Chem., 1929, 42, 722).—The diazo tests for the presence of phenols, always present in coal-tar pitch, is unreliable in the presence of colophony, but good results are obtained by Millon's reagent. The asphalt mixture (10 g.) is boiled for 20 min. with 25 c.c. of *N*-sodium hydroxide; the solution is filtered, rendered feebly acid with nitric acid, evaporated to 5–10 c.c., and treated with 5 c.c. of Millon's reagent. If coal-tar pitch is present in amounts exceeding 1% a colour develops after heating for 30 min. on the water-bath (cf. Chapin, B., 1920, 652 A).

A. R. POWELL.

Wood tar. I. Y. POSTOVSKI and B. G. PERETZ (J. Chem. Ind. Moscow, 1928, 5, 625–628).—The dehydrated tar (chiefly from pine and aspen wood) contained C 79.95, H 6.23, O 13.82%; ether dissolved 86%, leaving carbon. The fatty acids (2.1%) consisted of formic, acetic, propionic, butyric, and valeric acids. Of the phenols (30% of tar) 20% was liquid. Pyridine (0.1%) was present. Of the neutral substances 5% (probably naphthylfulvene) was volatile with steam, the remainder being hydroaromatic substances of high mol. wt. The tar contained 48.8–55% of water and 0.1–0.3% of inorganic substances. Its use as fuel is suggested. The calorific values of the crude and heat-dehydrated tar are 3970 and 7550 g.-cal., respectively.

CHEMICAL ABSTRACTS.

Silica gel for the extraction of high-sulphur oils from crude petroleum. G. E. WOODWARD (Ing. Eng. Chem., 1929, 21, 693–695).—The sulphur content of naphtha solutions of Inglewood crude petroleum was reduced 18% and 37% respectively by stirring with a commercial silica gel and Holmes gel respectively. Holmes gel removed most colour. 90% of the adsorbed material was removed from the gel by washing with naphtha, then benzene, and finally ether, and the remainder was recovered by treatment with sodium hydroxide.

D. WOODROFFE.

Anomalies in the measurement of the viscosity of mineral oils containing suspended paraffin wax.

L. EMANUELI and E. DA FANO (Giorn. Chim. Ind. Appl., 1929, 11, 261–263).—An extra dense mineral oil (cylinder oil) was found to exhibit viscosity in disagreement with Poiseuille's law, abnormally high efflux times being observed at the lower pressures. This behaviour is attributed to the presence of particles of solid paraffin wax in the oil.

T. H. POPE.

Utilisation of natural gases for the ammonia process. LIANDER. **Ammoniacal and spent liquors.** BAILEY. **Manganese carbide.** FISCHER and BANGERT.—See VII. **Protection of light alloys by tars.** AUBERT and PIGNOT. **Corrosion of metals by benzene.** FUJIO.—See X.

PATENTS.

Utilisation of coal. H. S. REED and R. D. LAMIE (U.S.P. 1,696,731, 25.12.28. Appl., 16.10.25).—Coal, e.g., slack, is delivered by a multiple-feed screw to an externally heated, rotating, ball-mill retort, divided into a series of chambers so that the coal particles become successively smaller with their passage through the retort. The powdered coal is delivered direct to a furnace with primary and secondary air, or is briquetted.

R. BRIGHTMAN.

Treatment of artificial fuels. H. E. WETHERBEE, Assr. to R. F. GRANT and H. M. HANNA (U.S.P. 1,696,511, 25.12.28. Appl., 22.10.25).—Carbonaceous particles are agitated in presence of water and 1–2% of oil or other binding material capable of displacing water to give an adherent film. The excess of water is drained off and fuel particles are briquetted by pressure after, e.g., oxidation of the film of drying oil.

R. BRIGHTMAN.

Combustion of pulverised fuel and means therefor. E. W. GREEN, G. R. UNTHANK, and D. DUNN (B.P. 314,614, 13.4.28).—A stream of pulverised fuel is evenly mixed in a whirl chamber and is then discharged from an annular conduit into the furnace. The stream is directed outwards by a deflector on the end of the central tube, through which a current of air is supplied to eliminate the rarefaction so produced. Streams of air supplied through perforated plates surrounding the central tube mix with the deflected stream of fuel, the volumes and velocities of the several air streams being such as to bring about complete combustion of the fuel.

A. B. MANNING.

Production of carbon. T. EWAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 314,163, 19.4.28).—Carbon monoxide obtained as a by-product in the industrial manufacture of hydrogen from water-gas is used for the production of carbon. After treatment of the water-gas with steam in the presence of a suitable catalyst at 500° the resulting gas mixture is washed with water or ammonia liquor to remove carbon dioxide and sulphur compounds, and the residual carbon monoxide is absorbed under pressure in an ammoniacal cuprous solution. On releasing the pressure and heating the solution, carbon monoxide free from catalyst poisons is produced and is used for the catalytic production of carbon. The cuprous solution should contain no halogen; an ammoniacal solution of cuprous formate, acetate, or carbonate gives good results.

A. B. MANNING.

Activation of carbon. J. DRUCKER and H. THIENEMANN, Assr. to I. G. FARBENIND A.-G. (U.S.P. 1,709,611, 16.4.29. Appl., 25.10.26. Ger., 10.11.25).—Carbon is activated by treatment with sulphur vapour at 800–1000°, until 40–80% of the carbon is converted into carbon disulphide. R. BRIGHTMAN.

Manufacture of activated charcoal. E. F. SCHELLER (U.S.P. 1,704,765, 12.3.29. Appl., 2.5.27. Ger., 3.5.26).—Carbonaceous matter is mixed with sodium monoxide and after reaction the mass is heated at 500–700°. Of the carbon thus obtained 0.1 g. will produce 60% decolorisation in 100 c.c. of standard molasses solution. R. BRIGHTMAN.

Carbonising and burning bituminous fuel. HUMPHREYS & GLASGOW, LTD., Asses. of J. M. RUSBY and W. I. BATTIN (B.P. 301,796, 28.9.28. U.S., 5.12.27).—The fuel passes down a vertical retort on to a travelling grate which carries the carbonised fuel into the combustion chamber of a boiler. The lower zone of the column of fuel in the retort is intermittently air-blasted, the blow gases passing through an offtake at the top of that zone to the combustion chamber. The fuel in the upper zone of the retort is carbonised by recirculating part of the gas generated in the retort through the lower and upper zones in succession, this operation alternating with the air-blasting periods. The excess coal gas generated is led off and collected. A. B. MANNING.

Carbonisation of coal and other solid carbonaceous substances. CONTINUOUS COAL CARBONISATION, LTD., C. B. WINZER, and R. NISBET (B.P. 314,665, 13.6.28).—Coal is carbonised in narrow ovens, through which it is carried within vertical containers. The latter are divided into two compartments by a central partition, and are constructed of two halves pivotally connected at their upper ends and provided with latches for locking the two parts together. They are open at the top, and are charged from hoppers, a pair of which are arranged in series, the lower being of capacity about equal to that of each container. The containers are carried on rollers running on girders which extend through the oven. At the discharge end of the oven mechanism is provided for swinging the two halves of the container apart and so discharging the carbonised fuel. A. B. MANNING.

Distillation of solid carbonaceous material. C. POSTEL, Assr. to AMER. SHALE REDUCTION CO. (U.S.P. 1,717,808, 18.6.29. Appl., 11.6.23).—The material is passed continuously through an inclined jacketed chamber which is externally heated. The jacket is filled with molten metal and the chamber is rotated so that the metal bath moves continuously past the source of heat, thereby ensuring uniform heating of the material to be distilled. A. B. MANNING.

Intermittently operating carbonising chambers. WOODALL-DUCKHAM (1920), LTD., and (Sir) A. McD. DUCKHAM (B.P. 314,659, 9.6.28).—Each chamber is provided with a bottom offtake in addition to the usual top offtake, the former being normally closed by a seal in a lower foul main; it opens when the pressure in the lower part of the carbonising chamber exceeds a predetermined maximum and closes again when this

pressure has been released. An uptake provided with a by-passed cut-off valve connects the upper and lower foul mains. Liquor sprays within the uptake maintain the required level of liquid in the lower main.

A. B. MANNING.

Vertical retorts for carbonisation of coal and like materials. E. WEST, and WEST'S GAS IMPROVEMENT CO., LTD. (B.P. 314,231, 9.7.28).—The retort walls are built up of superposed sections, each comprising (a) a ledge course which projects beyond the retort and serves to support the floor tiles of each combustion chamber, (b) a key course, and (c) chamber tiles extending from the key course to the next ledge course. The sections of each course are grooved and tongued so as to interlock with the courses above and below. The ledge course is designed to overlap the external horizontal joint between that course and the chamber tiles below, whilst the horizontal joints above and below the key course are covered by the floor tiles of the combustion chambers. A. B. MANNING.

Destructive hydrogenation of solid carbonaceous material. A. RULE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 314,213, 22.6.28).—Coals, lignites, etc., which on hydrogenation under 200 atm. pressure yield viscous products containing a high proportion of solid material, are hydrogenated under pressures of at least 500 atm. and at a temperature of 420°. A suitable oil or tar may be added as a vehicle. Mobile products are formed having a ratio of total oil to matter insoluble in benzene of at least 10:1. Hydrogenation under the conditions mentioned is generally complete in 1 hr.

A. B. MANNING.

Manufacture of fuel gas and similar combustible gases and apparatus therefor. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 314,239, 18.7.28. Addn. to B.P. 214,544; B., 1924, 549).—In carrying out the process described in the main patent the gasifying medium (air or steam) is preheated in a device arranged in the shaft of the producer above the fuel bed. The preheater may consist of a number of separate hollow members, constructed, e.g., of silicon carbide brick, and open below so that the preheated medium impinges directly on the top of the charge. An amount of steam and air sufficient to keep the charge of small fuel in motion is introduced through the grate from below. In an alternative arrangement a tube preheater is arranged in the upper part of the shaft with a solid heat-accumulator beneath it; the fuel is then preferably introduced through the latter, and thereby dried and partially gasified. A. B. MANNING.

Apparatus for the manufacture of water-gas. TULLY, SONS, & CO., LTD., and A. V. TULLY (B.P. 314,601, 4.4.28).—The apparatus comprises a gas producer, a superposed retort surrounded by a chequerwork heating jacket (cf. B.P. 271,765; B., 1927, 548), and a cyclone dust extractor, the outlet of which is connected to the stack valve. The dust extractor, which is adapted for the recovery of the sensible heat of the blow gases, serves to superheat the steam for the down run. When part of the water-gas is enriched with oil in the chequerwork jacket the dust extractor serves as a fixing chamber for the oil vapours. A. B. MANNING.

Direct production of ammonia. C. OTTO (U.S.P. 1,696,446, 25.12.28. Appl., 15.8.27. Ger., 9.8.26).—Distillation gases from coal gas-producing furnaces pass to a gas cooler, and the condensate is fed partly to the hydraulic main and partly is used to irrigate and cool the gases in the pipe leading to the cooler.

R. BRIGHTMAN.

Gas purification process. G. E. SEIL, Assr. to KOPPERS Co. (U.S.P. 1,701,825, 12.2.29. Appl., 26.12.25).—The hydrogen sulphide is absorbed by an alkaline solution, liberated from it by an air current, and oxidised by means of an aqueous medium containing a bacillus capable of oxidising hydrogen sulphide.

R. BRIGHTMAN.

Regeneration of washing liquids employed in removing carbon dioxide and other soluble constituents from gases. K. GORDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 314,842, 11.4.28).—The gases are scrubbed under pressure with water free from oxygen and the dissolved gases are removed by releasing the pressure in stages, or mainly at ordinary pressure, the final stage being below atmospheric pressure; the water is then returned to the absorption process without aeration.

W. G. CAREY.

Production of compressed gas. J. THIECKE, MINIMAX, A.-G., and H. HERZOG (B.P. 314,604, 4.4.28).—Compressed gas for compressed-air brakes, pneumatic hammers, paint or cement guns, and other industrial uses is produced by burning a mixture of about 85 pts. of ammonium nitrate and 15 pts. of charcoal in a closed vessel. Owing to the slow speed of combustion a safe and uniform development of pressure is obtained. The mixture may be compressed in the form of a cartridge and have a suitable priming embedded in it. A waterproof coating of shellac is provided.

A. B. MANNING.

Operating with gases containing carbon monoxide at elevated temperatures. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 314,129, 30.12.27).—Apparatus for carrying out processes, e.g., the synthesis of methyl alcohol, involving the use of gases containing carbon monoxide at high temperatures and pressures is constructed of iron in all parts except those maintained at temperatures between 150° and 250°. These are constructed or coated with materials, e.g., copper, silver, special steels (cf. B.P. 231,285; B., 1925, 425), which do not form metal carbonyls under the working conditions. Carbon monoxide is excluded from the apparatus, and is preferably replaced by hydrogen, while it is being heated up or cooled down.

A. B. MANNING.

Treatment of tarry matter, heavy hydrocarbon residues, and the like. A. S. KNOWLES, Assr. to TAR & PETROLEUM PROCESS Co. (U.S.P. 1,717,884, 18.6.29. Appl., 12.6.25).—The tar or other heavy liquid hydrocarbon is sprayed on to a bed of loose material, principally carbon, within a closed chamber heated externally from below. The liquid flows through the bed, in the lower layers of which it becomes coked. The gases and vapours evolved escape through an outlet in the top of the chamber, whilst the solid residue is removed as coke.

A. B. MANNING.

Purification of tar acid bearing oils. S. CAPLAN, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,710,764, 30.4.29. Appl., 29.9.24).—The oil is agitated with about 1.5 vols. of 1% aqueous ammonia or other buffer agent at 50°, separated from the aqueous layer, and agitated at 50° with about 1.5 vols. of water.

R. BRIGHTMAN.

Treatment of benzene to eliminate corrosive properties. T. R. TRAINOR, Assr. to J. A. KELLY (U.S.P. 1,711,367, 30.4.29. Appl., 30.4.27).—The benzene is treated successively with lime water and sodium bicarbonate solution while circulating in a closed system.

R. BRIGHTMAN.

Separation of oils from mixtures with solid substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 314,405, 23.1.28).—Oily residues obtained in the low-temperature carbonisation, cracking, extraction, or destructive hydrogenation of carbonaceous materials are mixed with about 30% of a liquid in which the oil is soluble and with a small quantity of a wetting agent, and are centrifuged. Aromatic or hydroaromatic hydrocarbons, tar oil fractions, etc. may be used as the solvent, and alkylated sulphonic acids of aromatic hydrocarbons, fatty acids or their salts, etc. as the wetting agent. It is sometimes advantageous to work in an alkaline medium.

A. B. MANNING.

Production of low b.p. and other hydrocarbons and derivatives thereof by the destructive hydrogenation of coals, oils, etc., and treatment of the residues. I. G. FARBENIND. A.-G. (B.P. 288,148, 28.3.28. Ger., 31.3.27).—The oily residues from hydrogenation processes are treated while still hot with hot gases or vapours, especially steam, to recover the oils. The material, for example, may be passed through a pipe by means of a worm conveyor, with or counter-current to the hot gases or vapours. The de-oiled residues may be carbonised and gasified, the hot gases being used in the oil recovery treatment; the carbonised residues may be used for the production of activated carbon. The oils recovered may be used as a vehicle in the hydrogenation of a further quantity of coal etc.

A. B. MANNING.

Cracking of hydrocarbons. G. H. TABER, JUN., Assr. to SINCLAIR REFINING Co. (U.S.P. 1,705,077, 12.3.29. Appl., 26.11.27).—The gas-vapour mixture from the cracking operation passes through a fractionating tower, vapour from which passes to a condenser and receiver. Vapours from the receiver are scrubbed with a higher-boiling oil fraction from the fractionating column. Vapour from the scrubber passes to the gas-holder, whilst the scrubbing oil is returned to the fractionating column and stripped by the ascending vapours from the still.

R. BRIGHTMAN.

Cracking of hydrocarbons. M. J. TRUMBLE (U.S.P. 1,696,658, 25.12.28. Appl., 2.9.24. Renewed 24.10.28).—Hydrocarbon oil is cracked under pressure by flowing in a thin film over the external surface of a furnace flue. The volatile products escape through a dephlegmator and are condensed. The phlegms, together with a relatively large volume of circulating solvent of b.p. above the cracking temperature used, and after settling from carbonaceous matter, are returned with raw oil

to the cracking still, the solvent removing the residual carbonaceous products from the flue surface, and carrying them into the settling chambers.

R. BRIGHTMAN.

Distillation of shale. H. S. REED and R. D. LAMIE (U.S.P. 1,696,730, 25.12.28. Appl., 16.10.25).—Oil shale is continuously distilled in a closed ball-mill retort, divided into compartments such that the size of the shale particles diminishes successively as the shale advances through the retort, until spent shale is discharged from the final chamber as a fine powder.

R. BRIGHTMAN.

Manufacture of paraffin. C. A. WARD, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,718,672, 25.6.29. Appl., 20.11.25).—Slack-wax, produced by filter-pressing chilled paraffin slop, is distilled to produce a sweatable wax.

J. S. G. THOMAS.

Production of liquid polymerisation products [fuels] from gases containing hydrocarbons. C. EPNER (B.P. 294,100, 8.6.28. Ger., 16.7.27).—Methane or other hydrocarbon gas (e.g., coke-oven gas) is subjected to the silent electric discharge (e.g., 20,000 volts at 10,000 cycles) at such a rate (2 litres per hr. through an ozoniser 4 cm. diam. \times 15 cm.) that formation of lubricating oils is avoided. The methane content of a coke-oven gas falls from 50% to 10%, with production of fuel oils. The gas is preferably preheated to avoid condensation in the ozoniser. The products may with advantage be subsequently hydrogenated.

C. HOLLINS.

Anti-knock fuel. G. HAMMOND, Assr. to FUEL DEVELOPMENT CORP. (U.S.P. 1,713,530, 21.5.29. Appl., 15.3.26).—Butyl alcohol together with commercial alcohol, or ethylene glycol and water, is added to gasoline.

R. BRIGHTMAN.

Lubricating oil. K. G. MACKENZIE and R. HASKELL, Assrs. to TEXAS CO. (U.S.P. 1,705,298, 12.3.29. Appl., 21.8.24).—Anti-chattering properties are imparted to lubricating oil by adding about 2% of oxidised paraffin wax.

R. BRIGHTMAN.

Operating molten-slag gas producers. H. J. F. PHILIPON, Assr. to SOC. ANON. "L'AIR CHAUD" (U.S.P. 1,720,290, 9.7.29. Appl., 31.1.23. Belg., 8.3.22).—See B.P. 194,648; B., 1923, 702.

Gas purification process. H. A. GOLLMAR, Assr. to KOPPERS CO. (U.S.P. 1,719,177 and 1,719,762, 2.7.29. Appl., 7.3.27 and 5.11.26).—See B.P. 280,165 and 286,633; B., 1929, 8.

Gas purification process. D. L. JACOBSON, Assr. to KOPPERS CO. (U.S.P. 1,719,180, 2.7.29. Appl., 5.11.26).—See B.P. 286,663; B., 1929, 8.

Gas analyser. A. B. CUNNINGHAM, Assr. to REPUBLIC FLOW METERS CO. (U.S.P. 1,719,593, 2.7.29. Appl., 19.5.23).—See B.P. 288,510; B., 1928, 508.

[Apparatus for] combustion of [small] fuel. W. R. SMALL (B.P. 314,469, 9.8.28).

Liquid fuel oil burners. TODD OIL BURNERS, LTD., and E. CLARK (B.P. 315,056, 21.5.28).

Low-pressure liquid fuel burners. S. E. MAJOR and E. F. BAKER (B.P. 294,577, 23.7.28. Austral., 26.7.27).

[Down-draught] gas producers [with depth tubes]. H. C. READING (B.P. 315,090, 4.7.28).

Operating furnaces (B.P. 292,146). Deodorisation of gases (B.P. 314,187).—See I. Cleaning etc. agents (B.P. 313,861).—See III.

III.—ORGANIC INTERMEDIATES.

Synthetic carbamide from ammonia and carbon dioxide. G. A. YAKOVKIN (J. Appl. Chem. Russia, 1928, 1, 70—77).—Carbamide was obtained in 40% yield from liquid ammonia saturated with carbon dioxide under pressure, without supercooling, followed by heating at 160—170° and increasing the pressure to 75—80 atm.

CHEMICAL ABSTRACTS.

Thermal reactions of pure organic substances under high hydrogen pressures. F. HOFMANN and K. LANG (Brennstoff-Chem., 1929, 10, 203—205).—A number of pure hydrocarbons have been treated with hydrogen at 400—500° under pressures of 200—250 atm. Benzene was unchanged except for the production of 1.2—2.3% of diphenyl. Diphenyl itself was transformed to the extent of over 90% into benzene. With a suitable choice of temperature and time of heating, up to 50% of toluene was converted into benzene, the methyl group appearing partly as methane and partly as ethane. Small amounts of diphenyl, monomethyl-diphenyl, and ditolyl were also formed. In the absence of high-pressure hydrogen, toluene remained stable at 470°. *cyclo*Hexane was unchanged when heated with hydrogen at 460° and 212 atm. pressure. At a slightly higher temperature, however, gaseous hydrocarbons and methylcyclopentane were produced. In the absence of hydrogen, *cyclo*hexane was decomposed at the lower temperature. *cyclo*Hexene was partly hydrogenated to *cyclo*hexane and partly converted into polymerisation and other products; among the latter methyl- and dimethyl-*cyclo*pentane and *dicyclo*hexyl were identified. Ethylene was almost completely converted into saturated hydrocarbons. Although at 490° pyridine remained stable in the absence of hydrogen, with hydrogen under 240 atm. it yielded 90% of ammonia and gaseous hydrocarbons. In the residual oil, besides unchanged pyridine, nitriles and higher tertiary bases were identified.

A. B. MANNING.

The π of sulphonation. C. COURTOT (Rev. Gén. Mat. Col., 1929, 33, 177—183).—The π of sulphonation is defined by Guyot (B., 1919, 811 A) as the limit of concentration of sulphur trioxide at which no further sulphonation of a given organic molecule at a given temperature can take place. The quantity of acid, X, containing a % of sulphur trioxide, required for the monosulphonation of 1 mol. of an organic compound, RH, is given by $X = \text{SO}_3 (100 - \pi) / (a - \pi)$. Before the sulphonic acid can be separated, it is necessary to remove the acid of π concentration by the very inconvenient methods of liming out or salting out. In order to eliminate the water produced during the reaction, Guyot proposed passing through the sulphonation

mixture an excess of the vapour of the organic compound, the water being carried off thereby, and complete utilisation of the acid for sulphonation being assured. A method is described for the monosulphonation of benzene, toluene, naphthalene, fluorene, diphenyl sulphide, and chlorobenzene by means of sulphur trioxide in chloroform solution, 90% of the theoretical yield being obtained. The preparation of the disulphonic acid is sometimes complicated by the formation of a loose combination of the disulphonic acid with additional sulphur trioxide, which, however, on keeping gradually gives up sulphur trioxide to the monosulphonic acid present, the yield of naphthalenedisulphonic acid, for example, being 65% after 24 hrs. L. G. LAWRIE.

1:4-Dioxan. E. W. REID and H. E. HOFMANN (Ind. Eng. Chem., 1929, 21, 695—697).—1:4-Dioxan has m.p. 11°, b.p. 101.1°, d_{20}^{20} 1.0338, n_D^{20} 1.4232, is miscible in all proportions with water, stable, and is little affected by acids, alkalis, sodium, or oxidising agents. It is a solvent for "wet" cellulose nitrate, resins, oils, waxes, dyes, and many organic compounds. Its boiling range and the solubility of cellulose nitrate and acetate respectively in the system 1:4-dioxan-ethyl alcohol-toluene have been determined, and are shown graphically. It is recommended for use in lacquer manufacture. D. WOODROFFE.

Detection of isopropyl alcohol. REIF.—See XX.

PATENTS.

Production and use of cleaning, emulsifying, and wetting agents. K. LINDNER (B.P. 313,861, 14.12.27).—High-boiling petroleum, brown-coal tar, etc. mixed with aliphatic or aromatic compounds containing hydroxyl, thiol, ketonic, carboxylic acid or ester or chloride groups, are sulphonated with chlorosulphonic acid etc. in the cold. *E.g.*, refined mineral oil of viscosity 7.5° Engler is mixed with isopropyl alcohol and sulphonated with chlorosulphonic acid at 35°. [Stat. ref.] C. HOLLINS.

Process of separating liquids [alcohols]. E. H. HAABESTAD, Asst. to B.A.S. Co. (U.S.P. 1,716,957, 11.6.29. Appl., 31.7.25).—A mixture of monohydric alcohols (slightly soluble in water) containing 4 to 6 carbon atoms with a volatile substance soluble in the alcohols but insoluble in water is treated with water, the aqueous solution of the alcohols is heated, and the vapours are condensed; the alcohols are separated, and the remainder of the condensate and still residues are again treated with water etc. B. M. VENABLES.

Manufacture of acetic acid from acetylene. J. KÁRPÁTI and M. G. HÜRSCH (B.P. 287,135 and 294,226, 2.3.28. Hung., [A] 16.3.27, [B] 21.7.27).—(A) Acetylene is passed with oxygen, or acetylene and oxygen are passed alternately, at 35—40° into acetic acid containing mercury sulphate and as oxidation catalyst cellulose or a wood sawdust which has been heated in nitrogen and activated with steam. (B) The three reagents (acetylene, oxygen, and acetic acid containing the catalysts) are circulated through separate systems having as common portion the reaction chamber, where the acetic acid is sprayed downwards in countercurrent first to acetylene (until absorption ceases), and then to oxygen alternately. C. HOLLINS.

Manufacture of aliphatic acid [acetic] anhydrides. BRIT. CELANESE, LTD., S. J. GREEN, and R. R. WIDDOWSON (B.P. 314,555, 28.1.28).—For the anhydridisation of acetic acid vapour tungstic acid and/or a tungstate of calcium or magnesium is used as catalyst at 550—650°. C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. H. DREYFUS (B.P. 314,132, 27.3.28).—Acetic acid vapour (about 3 pts.) is passed with hydrogen chloride (1 pt.) through a fireclay tube at 200—250°, and afterwards over sodium acetate to remove mineral acid. Other hydrogen halides may be used. C. HOLLINS.

Manufacture of suspensions and emulsions. I. G. FARBERIND. A.-G. (B.P. 287,464, 19.3.28. Ger., 18.3.27).—The products from the sulphonation of wool fatty acids with phenols (B.P. 307,776 and 286,252; B., 1929, 442, 608) are used as emulsifying agents and for cleansing (*e.g.*, removal of oil from cloth by emulsification). C. HOLLINS.

Manufacture of esters [from vinyl acetate]. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H., W. O. HERRMANN, and H. DEUTSCH (B.P. 314,646, 14.5.28).—Vinyl acetate reacts with alcohols or phenols in presence of acid catalysts (*e.g.*, sulphuric acid) to give other acetic esters and acetaldehyde. Cooling is sometimes necessary. The preparation of ethyl, *n*-butyl, glycol, and phenyl acetates is described. C. HOLLINS.

Production of acidyl derivatives [O-esters] of hydroxyalkyl ethers of polyhydric alcohols. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 313,892, 19.12.27).—The monohydroxyalkyl ethers of alcohols containing 3 or more hydroxyl groups are heated with an acylating agent, particularly an acid anhydride. Thus sorbitol β -hydroxyethyl ether (from sorbitol and ethylene oxide) with acetic anhydride gives a viscous liquid which is a rapid swelling agent for cellulose nitrate. Glycerol β -hydroxyethyl ether yields with phthalic anhydride a plasticiser for "glyptal" resins, and with succinic acid a viscous product which hardens on further heating to an insoluble, infusible, elastic resin. C. HOLLINS.

Production of acetaldehyde from acetylene. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 313,864, 12.3.28).—Acetylene, pure or diluted, is passed into concentrated aqueous solutions of zinc chloride or other salts showing acid reaction (*e.g.*, chromic chloride, ferric chloride), at 110—140°, with or without addition of small quantities of compounds of metals of groups 1, 2, 4, 6, and 8 of high at. wt. (*e.g.*, mercury, uranium). C. HOLLINS.

Manufacture of acetone. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 313,897, 16.3.28).—Acetaldehyde and steam at about 400° are passed over a catalyst sufficiently rapidly to minimise the decomposition of the acetone formed. Suitable catalysts are zinc oxide and other compounds of metals the acetates of which decompose with production of acetone, deposited on aluminium rings. A 93% conversion is obtained when the vapours of 300 c.c. of 30% aqueous acetaldehyde are passed per hr. over 1 litre of catalyst. C. HOLLINS.

Manufacture of organic bases and pest-destroying agents therefrom. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,934, 14.1. and 25.8.28).—Aliphatic hydrocarbons containing two or more double linkings, *e.g.*, butadiene, isoprene, piperylene, etc., are condensed with ammonia or amines in presence of an alkali metal at low temperatures to give bases containing one or more butenyl groups. The products are used in aqueous solution or suspension, with soap-like substances if desired, for destruction of pests. Butadiene sodium (or potassium) and ammonia at -80° to -15° give tributenylamine, b.p. $82-87^{\circ}/4-6$ mm., and a base of higher b.p. Butadiene gives products of the following b.p. when condensed similarly with methylamine (b.p. $165-166^{\circ}$), piperidine (b.p. $178-180^{\circ}$), aniline (b.p. $235-237^{\circ}/752$ mm., and by-product, b.p. $150-160^{\circ}/10$ mm.), methylaniline (b.p. $234-236^{\circ}$), ethylaniline (b.p. $243.5-244.5^{\circ}$), ethylenediamine (b.p. $160-260^{\circ}$), cyclohexylamine (b.p. $150-250^{\circ}$). Dimethylbutadiene and piperidine similarly give a product of b.p. $205-208^{\circ}$. Piperylene and piperidine with sodium at $80-100^{\circ}$ yield 1-*n*-pentenylpiperidine, b.p. $195-196^{\circ}$; isoprene and methylamine or *n*-butylamine give products of b.p. $193-197^{\circ}$ and $233-238^{\circ}$, respectively. C. HOLLINS.

Production of salts of halogen-substituted aliphatic tetra[alkyl]ammonium bases. E. GLÜCKSMANN (B.P. 297,839, 27.9.28. Ger., 29.9.27).—Halogenated tetra-alkylammonium salts (*e.g.*, triethyl- β -bromoethylammonium bromide) are converted into other salts by double decomposition especially with formation of insoluble metal salts. C. HOLLINS.

Manufacture of condensation products from urea, thiourea, or their derivatives, and an alcohol or a ketone. I. G. FARBENIND. A.-G. (B.P. 290,192, 22.3.28. Addn. to B.P. 278,390; B., 1928, 648).—Metals of the alkali, alkaline-earth, or zinc group react with the products of B.P. 287,095 (B., 1929, 691) to give metal compounds which are viscous liquids or resins completely soluble in water and useful for keeping casein, gelatin, etc. in soft condition. Sodium, calcium, zinc, and magnesium are employed in the examples. C. HOLLINS.

Manufacture of lead tetraethyl. II. W. DAUDT, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,705,723, 19.3.29. Appl., 15.10.26).—Magnesium ethyl chloride is used; *e.g.*, the solution from 30 pts. of ethyl chloride and 10 pts. of magnesium in 125 pts. of ether containing 1 pt. of methyl iodide and a crystal of iodine is added to a suspension of 60 pts. of lead chloride in 125 pts. of ether at $0-50^{\circ}$, and the mass immersed in 600 pts. of water. R. BRIGHTMAN.

Production of the [normal and] acid sulphates of α -naphthylamine. T. WARBURTON (B.P. 315,078, 19.6.28).— α -Naphthylamine is mixed with the calculated amount of sulphuric acid in a mixing or grinding machine. C. HOLLINS.

Manufacture of new aromatic *N*-aminoalkyl-aminoaldehydes and derivatives thereof. I. G. FARBENIND. A.-G. (B.P. 288,972, 3.4.28. Ger., 16.4.27).—The method of B.P. 17,135 of 1898 is applied to ω -dialkylaminoalkylanilines. *p*-Nitrotoluene-2-sulphonic

acid is reduced with zinc dust to the hydroxylamine, concentrated hydrochloric acid and formaldehyde and finally β -diethylaminoethylamine hydrochloride are added; after two days the product is salted out and hydrolysed to give 4- β -diethylaminoethylaminobenzaldehyde, b.p. $157-159^{\circ}/1$ mm. The *N*-methyl- (b.p. $166-168^{\circ}/2$ mm.), 2-chloro- (b.p. $120-125^{\circ}/1.5$ mm.), and 3-methoxy- (b.p. $144-146^{\circ}/6$ mm.) derivatives, 2- β -diethylaminoethylaminobenzaldehyde, b.p. $130-134^{\circ}/2$ mm., and *N*-methyl-*N*- γ -dimethylamino- β -butylaminobenzaldehyde, b.p. $152-154^{\circ}/1$ mm., are similarly prepared. C. HOLLINS.

Manufacture of 3 : 7-tetra-alkyldiaminoxanthones. IMPERIAL CHEM. INDUSTRIES, LTD., E. H. RODD, and H. H. STOCKS (B.P. 314,826, 3.3.28).—3 : 7-Tetra-alkyldiaminoxanthones are converted into corresponding xanthiones by the action of sulphur in boiling xylene, and thence into xanthenes by boiling with acid. C. HOLLINS.

Manufacture of hydroxypyridine compounds. SCHERING-KAHLBAUM A.-G., Assees. of C. RÄTH (B.P. 288,628, 3.4.28. Ger., 14.4.27).—A 2-halogenopyridine having no hydrolysable group in the 5-position is boiled with alkali at 175° or heated in a closed vessel with hydrochloric acid at 150° . 2-Hydroxypyridine and 5-nitro-2-hydroxypyridine, m.p. 184° , are thus prepared. C. HOLLINS.

Preparation of pure quinoline from crude coal-tar quinoline. O. WAGNER (G.P. 451,590, 31.10.25).—Air is led through boiling crude quinoline containing copper oxide or copper to oxidise dihydric phenols, sulphur compounds, etc. Monohydric phenols are removed by distillation with aniline (azeotropic mixture, b.p. $182-185^{\circ}$), and the quinoline bases are then distilled. C. HOLLINS.

Intermediates of the 1 : 9-anthrathiazole series. R. N. LULEK, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,706,981, 26.3.29. Appl., 18.3.27).—1-Chloroanthraquinone-2-carboxylic acid (or its aldehyde) is heated with sodium polysulphide and ammonia at 100° in a closed vessel to yield the anthrathiazole-carboxylic acid. C. HOLLINS.

Manufacture of condensation products of the benzodiazine [quinazoline] series. I. G. FARBENIND. A.-G. (B.P. 288,159, 30.3.28. Ger., 31.3.27).—2 : 4-Dihalogenoquinazolines are condensed with 2 mols. of the same or different amines. *E.g.*, 2 : 4-dichloro-6-nitroquinazoline is heated with an aqueous solution of J-acid (2 mols.) at $35-85^{\circ}$, and the product isolated as sodium salt. 2 : 4-Dichloro-6-nitroquinazoline is also condensed with H-acid (1 mol.) and then aniline (1 mol.). The products on reduction give diazotisable amines. The reduced product from J-acid (2 mols.) may be condensed with 2 : 4-dichloroquinazoline and the resulting compound condensed with a further molecule of J-acid. C. HOLLINS.

Operating with gases containing carbon monoxide (B.P. 314,129).—See II. [Thioindigoid dye] intermediate (B.P. 288,215).—See IV. Wetting etc. agents (F.P. 630,615).—See VI. Froth flotation (B.P. 314,822).—See X. Sulpho-acid products (B.P.

308,280).—See XII. **Varnishes** (B.P. 314,571).—See XIII. **3-Nitro-2-aminopyridine-5-sulphonic acid** (Austr. P. 107,595).—See XX. **Photographic desensitisers** (B.P. 314,144).—See XXI.

IV.—DYESTUFFS.

Direct cotton dyes from 2 : 7-diaminofluorene. G. T. MORGAN and H. A. HARRISON (J.S.C.I., 1929, 48, 125—126 T).—In the fluorene series the base corresponding in chemical constitution with benzidine is 2 : 7-diaminofluorene (m.p. 163°) and accordingly the latter has been tetrazotised and coupled with 1-amino-8-naphthol-3 : 6-disulphonic acid (H-acid) and with 1-amino-8-naphthol-2 : 4-disulphonic acid (2S acid). The resulting tetrakisazo dyes have been examined in regard to their affinity for unmordanted cotton. The diaminofluorene-H-acid dye is greener in shade and somewhat superior in affinity to the benzidine-H-acid dye (Chlorazol Blue B). It is slightly inferior in fastness to sulphuric acid, but somewhat superior in fastness to washing. The diaminofluorene-2S-acid dye is a considerably redder blue than the dianisidine-2S-acid dye (Chlorazol Sky Blue FF). It is slightly inferior in affinity and fastness. The fluorene dyes are not affected beneficially by after-coppering. There is evidently a close analogy between the di-*p*-diamines of the diphenyl and fluorene series as regard their capacity for furnishing direct cotton dyes, but the colours of the fluorene series exhibit no outstanding advantages over the commercial dyes of the diphenyl group.

Crystallisation phenomena in dye salts. H. WAGNER (Kolloid-Z., 1929, 48, 248—252).—Some of the properties of Pigment Scarlet 3B have been investigated. The substance dissolves readily when warmed with 50 times the amount of water, giving a turbid solution which when kept for 24 hrs. deposits long needles of the dye. The particles in solution exhibit the Brownian movement. Addition of barium chloride precipitates a well-defined insoluble salt, which is carmine-red: this comes down in a very fine granular form, the crystalline nature of which is revealed only by X-ray examination. The dye is salted out by concentrated solutions of sodium or calcium chloride with the formation of a gel, but the existence of fine needle-like crystals of the dye is easily recognisable in the gel. Very dilute solutions of sodium chloride cause the separation of long needles. The gels formed by high concentrations of salt are unaltered by shaking, but those formed by lower concentrations are liquefied by shaking: the latter do not set again when left at rest, and are said to be "partially thixotropic." The partially thixotropic gels gradually become crystalline on ageing. E. S. HEDGES.

Chrome colours on wool. NOELTING, BATTEGAY, and LICHTENBERGER.—See VI.

PATENTS.

Manufacture of aminoanthraquinone derivatives [dyes for cellulose acetate]. SOC. CHEM. IND. IN BASLE (B.P. 295,257, 7.8.28. Switz., 4.8.27).—Aminoanthraquinones (e.g., 1 : 4 : 5 : 8-tetra-aminoanthraquinone, 1 : 4-aminohydroxyanthraquinone) are solu-

bilised by treatment with an aldehyde (except formaldehyde) in presence of bisulphite. The aldehydes used are benzaldehyde, furfuraldehyde, acetaldehyde, crotonaldehyde. The products may be isolated by evaporation or salting out, and are soluble dyes for acetate silk. C. HOLLINS.

Preparation of [benzanthrone] dyes, and dyeing and printing of textile fibres etc. W. M. TODD, J. S. WILSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 314,589 and 314,763, 30.12.27).—(A) Amino-dibenzanthrone is boiled with hydrochloric acid, sodium nitrite, and copper sulphate to give a black vat dye. (B) Nitro- or amino-dibenzanthrone is converted by pyridine-sulphuric anhydride, pyridine, and copper into a sulphuric ester of the leuco-compound, which is treated on the fibre with nitrous acid and copper sulphate, or is boiled with hydrochloric acid, sodium nitrite, and copper sulphate to produce the black vat dye in substance. C. HOLLINS.

Manufacture of 2-cyanobenzanthrone [and vat dye therefrom]. KALLE & Co. A.-G. (Swiss P. 122,904, 16.1.25. Ger., 17.11.24. Addn. to Swiss P. 119,978).—See B.P. 243,026; B., 1927, 101. 2-Chlorobenzanthrone, m.p. 190°, obtained from 3-acetamidobenzanthrone by chlorination, hydrolysis, and removal of the amino-group, is converted by cuprous cyanide in pyridine at 220° into 2-cyanobenzanthrone, m.p. 275—276°, which on alkaline fusion gives a fast blue vat dye. C. HOLLINS.

Manufacture of nitrogenous vat dyes [of the dibenzanthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 314,593, 4.2. and 11.6.28).—Nitro- or nitroso-dibenzanthrones or their reduction products are converted into grey-blue vat dyes by boiling with sodium acetate, potassium carbonate, or sodium formate, in presence of copper or copper compounds. C. HOLLINS.

Manufacture of nitrogenous [vat] dyes [of the pyranthrone series]. I. G. FARBENIND. A.-G. ([A] B.P. 285,502, 18.2.28. Ger., 18.2.27. [B] Addn. B.P. 314,652, 31.5.28).—(A) Di-, tri-, or tetra-bromopyranthrone is condensed in nitrobenzene, preferably in presence of sodium acetate and copper carbonate, with aminoanthraquinones etc., e.g., α -aminoanthraquinone (violet-black), 1-amino-4-methoxyanthraquinone (black), 4-amino-1 : 1'-dianthraquinonyl (dark violet), to give vat dyes. (B) The process proceeds more rapidly and smoothly in presence of copper or copper oxide. C. HOLLINS.

Stabilised diazo preparations. I. G. FARBENIND. A.-G., Asses. of K. SCHNITZSPAHN (G.P. 453,985, 19.6.25. Addn. to G.P. 438,743; B., 1929, 647).—Magnesium chloride, partly or completely anhydrous, is added as a stabiliser in the process of the prior patent. E.g., *p*-nitroaniline hydrochloride is mixed with nearly anhydrous magnesium ammonium chloride ($\text{MgCl}_2 \cdot 2\text{NH}_4\text{Cl}$), dry sodium hydrogen sulphate, and dry sodium nitrate. C. HOLLINS.

Manufacture of mordant [chrome-printing diazo] dyes. I. G. FARBENIND. A.-G., Asses. of W. NEELMEIER and W. REBNER (G.P. 454,177, 25.3.26).—A *p*-phenylenediamine is coupled with 2 mols. of salicylic or *o*-cresotic acid, and the resulting dye is sulphonated.

Diazotised *p*-aminobenzeneazosalicylic acid coupled with salicylic acid and sulphonated with 20% oleum gives a chrome-printing orange. C. HOLLINS.

Insoluble azo dyes [ice colours and pigments]. Soc. CHEM. IND. IN BASLE (F.P. 630,903, 26.2.27. Switz., 19.3.26).—2:3-Hydroxynaphthoic arylamides are coupled in substance or on the fibre with diazotised *ar*-tetrahydro- β -naphthylamine for reds. C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 287,479, 23.2.28. Ger., 21.3.27).—Arylamides of 2:3-hydroxynaphthoic or acetoacetic acid are coupled in substance or on the fibre with diazotised dichloro- or dibromo-*m*-toluidines, *e.g.*, 2:4-, 4:5-, or 4:6-dichloro- or 4:5-dibromo-*m*-toluidine. The coupling components in the examples are 2:3-hydroxynaphthoic 4- and 5-chloro-*o*-anisidides, α - and β -naphthylamides, 2:5-dimethoxyanilide, 5-chloro-*o*-toluidide, and bisacetoacetylolidine. The dyeings are fast to kier-boiling and light. C. HOLLINS.

Manufacture of insoluble azo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (F.P. 630,644, 10.3.27. Ger., 24.3.26).—Arylamides of 2:3-hydroxynaphthoic or acetoacetic acid, or hydroxynaphthacarbazoles, are coupled in substance or on the fibre with tetrazotised diaminocarbazoles (non-sulphonated). Examples are: 2:8-diaminocarbazole ($N=5$) with 2:3-hydroxynaphthoic anilide (blue) or 2-hydroxy- β -naphthacarbazole (black) or bisacetoacetylolidine (yellow-brown); 2:8-diamino-3:7-dimethylcarbazole with 2:3-hydroxynaphthoic α -naphthylamide (blue-black); 3:7-dichloro-2:8-diaminocarbazole with 2:3-hydroxynaphthoic α -naphthylamide (violet). C. HOLLINS.

Manufacture of azo dyes [blue to violet ice colours and pigments]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 313,865, 12.3.28).—A *p*-aminodiphenylamine is treated with excess of nitrous acid to form a nitrosodiaz compound, which is then coupled in substance or on the fibre with a 2:3-hydroxynaphthoic arylamide. Violet to garnet shades are obtained, which may be converted into blues or dark violets by removal of the nitroso-group, *e.g.*, by treatment with hot alkaline sulphide solution, preferably with addition of soap etc. The following derivatives of 4-aminodiphenylamine are mentioned in the examples: 2'- and 4'-methoxy-, 2'- and 4'-ethoxy-, 2', 3', and 4'-methyl-, 2'- and 4'-phenoxy-, 2', 3', and 4'-chloro-, 4'-methoxy-3-methyl-, 6'-methoxy-3'-methyl-, 4'- and 5'-chloro-2'-methyl-, 6'-chloro-3'-methyl-, 3'-chloro-4'-methyl-, 4'-chloro-2'-methoxy-, 4'-chloro-3-methoxy-2':6-dimethyl-, 2':3- and 3':4'-dichloro-, 2':3-dimethoxy-, 2':3-dimethyl-, also *p*-aminophenyl- β -naphthylamine. In addition to the usual arylamides the following are mentioned: 2:3-hydroxynaphthoic *o*-phenoxyanilide, *p*-benzyloxyanilide, *o*- and *p*-ethylanilides, 5-chloro-2-ethylanilide, *m*-benzoylanilide, 3-methoxy- β -naphthylamide, 4-dimethylamino-sulphonyl-*o*-anisidide, 4-ethylsulphonyl-*o*-toluidide; bis-2:3-hydroxynaphthoic derivatives of 2:5-diamino-*p*-tolyl methyl ether, 4:4'-diamino-2:2'-dimethyldiphenylmethane, 4:4'-diaminobenzophenone; also 6-bromo-2:3-hydroxynaphthoic *o*-anisidide, α -naphthylamide, anilide, 5-chloro-*o*-anisidide. C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 287,908, 27.3.28. Ger., 29.3.27).—2:3- or 2:5-Dichloro-*p*-toluidine is coupled in substance or on the fibre with 2:3-hydroxynaphthoic arylamides, *e.g.*, the anilide (scarlet-red), 4-chloro-*o*-toluidide (blue-red), 5- or 4-chloro-*o*-anisidide (red), etc. Good fastness to kier-boiling and light is claimed. C. HOLLINS.

Manufacture of azo dyes [for wool and viscose silk] and their application. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 314,672, 27.6.28).—Yellow wool dyes of good milling fastness are prepared by coupling a tetrazotised 4:4'-diaminodiphenyl di- or tri-sulphide with 2 mols. of a sulphonated pyrazolone, *e.g.*, 1-*p*-sulphophenyl-3-methyl-5-pyrazolone or 1-(2:5-dichloro-4-sulphophenyl)-3-methyl-5-pyrazolone. The dyes also give level shades on viscose silk. C. HOLLINS.

Manufacture of azo dyes [for wool and lakes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 314,768, 1.3.28).—Butyl anthranilate is diazotised and coupled with sulphonated coupling components, *e.g.*, 1-*p*-sulphophenyl-3-methylpyrazolone (yellow on wool; yellow barium lake), phenyl- γ -acid (red-brown), H-acid \leftarrow *p*-nitroaniline (black). [Stat. ref.] C. HOLLINS.

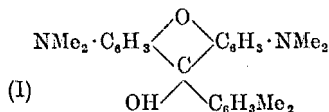
Blue tetrakisazo dyes for cotton. H. JORDAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,712,732, 14.5.29. Appl., 19.9.25).—Dyes of the type [monoacyl-*p*-arylenediamine or its sulphonic acid \rightarrow non-hydroxylated middle component]₂ \rightarrow carbonyl-J-acid give after removal of the acyl group reddish- to greenish-blue shades on cotton and may be diazotised and developed on the fibre with β -naphthol to improve fastness to washing. The green-blue dye [*p*-aminoacetanilide-3-sulphonic acid \rightarrow Cleve acid]₂ \rightarrow carbonyl-J-acid, hydrolysed, is specially claimed. C. HOLLINS.

Conversion of complex metal compounds of *o*-hydroxyazo dyes into related compounds. I. G. FARBENIND. A.-G., Assees. of H. KRZIKALLA and H. KÄMMERER (G.P. 453,949, 1.4.26).—Prechromed azo dyes derived from *o*-aminophenols are subjected to ordinary chemical reactions; *e.g.*, nitro-groups may be reduced to amino- or azoxy-groups, and amino-groups may be acylated, without destroying the metal complex. Examples are: acetylation of prechromed 4-chloro-*o*-aminophenol \rightarrow H-acid for a redder wool dye; reduction of the nitro-group (to amino-group) in 4-nitro-*o*-aminophenol-6-sulphonic acid \rightarrow β -naphthol for a blue to grey wool dye. C. HOLLINS.

Manufacture of [thioindigoid] vat dyes and intermediate products. I. G. FARBENIND. A.-G. (B.P. 288,215, 4.4.28. Ger., 4.4.27).—5-Halogeno-4-methylthioindigos show good fastness to boiling. 6-Chloro-2-cyano-*m*-toluidine, obtained by chlorination of the cyano-*m*-toluidine hydrochloride, is converted into 4-chloro-2-cyano-3-methylphenylglycolic acid, which by hydrolysis of the nitrile group, heating in dilute sulphuric acid, and oxidising yields 5:5'-dichloro-4:4'-dimethylthioindigotin (red-violet). C. HOLLINS.

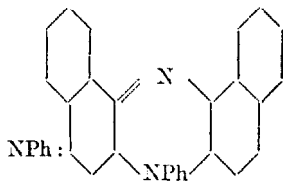
Manufacture of xanthen dyes. IMPERIAL CHEM. INDUSTRIES, LTD., E. H. RODD, and H. H. STOCKS (B.P.

314,825, 3.3.28).—3 : 7-Tetra-alkyldiaminoxanthenes are condensed with aromatic halogen compounds in presence of sodium to give carbinols; the salts of the products are dyes having a rhodamine shade. *E.g.*, 3 : 7-tetramethyldiaminoxanthone, sodium, and 4-chloro-*m*-xylene interact in benzene at 70° give the carbinol (I); 2-chloronaph-



thalene, *o*-chlorotoluene, and chloroanisoles react similarly. C. HOLLINS.

Manufacture of azine dyes. SOC. ANON. DES MAT. COL. ET PROD. CHIM. DE ST. DENIS, R. LANTZ, and A. WAHL (F.P. 629,669, 22.2.27, and B.P. 285,840, 22.2.28. Fr., 22.2.27).—Oxazines are heated with primary arylamines to give azines. 9-Phenylimino- $\beta\beta'$ -dinaphthoxazine and aniline yield 9-phenylimino-7-phenyl- $\beta\beta'$ -dinaphthoxazine (attached formula); the 5-anilino-derivative and the $\alpha\beta'$ -isomeride are similarly



prepared. 9-Anilino-5-phenylimino- β -naphthoxazine and aniline yield a corresponding azine. C. HOLLINS.

Manufacture of coloured [resinous or plastic] compositions or solutions of the same. [Dyeing of acetate silk.] J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 314,451, 28.12.27).—Insoluble colours are incorporated with water-soluble resinous plastics. *E.g.*, the sodium salt of an acetaldehyde-phenol-chloroacetic acid condensation product is mixed with 1 : 4-diaminoanthraquinone and a little water in a roller-mill, and dried and ground; the violet powder so obtained gives with water an inky liquid, or may be used for printing or dyeing acetate silk. Condensation products of phenolsulphonic acid with formaldehyde or sulphur, alkali casein, and the like are also suitable. C. HOLLINS.

Dyes [for acetate silk for domestic use]. S. M. TOOTAL (B.P. 314,149, 5.4.28).—Insoluble acetate silk dyes are suspended in solid soluble gelatin and glycerol, in a container of harder gelatin if desired, with or without powdered soluble dye in the same container. C. HOLLINS.

Condensation products [thiohydrins] of the anthraquinone series [dyes for acetate silk]. G. KRÄNZLEIN and M. CORELL, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,710,992, 30.4.29. Appl., 15.12.26. Ger., 17.12.25).—See B.P. 263,178; B., 1928, 398.

Condensation products [dyes] of the anthraquinone series. F. ACKERMANN and P. SCHETELIG, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,719,792, 2.7.29. Appl., 18.5.25. Switz., 2.8.24).—See B.P. 237,872; B., 1925, 839.

Manufacture of azo dyes derived from 2-hydroxynaphthalene-3-carboxylic acid arylamides. H. WAGNER, Assr. to GENERAL ANILINE WORKS, INC. (Reissue 17,361, 2.7.29, of U.S.P. 1,622,690. Appl., 21.2.29).—See B., 1927, 469.

Vat dyes [of the thioindigoid series]. E. HOFFA, H. HENYA, E. THOMA, and O. HIRSCHL, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,710,981, 30.4.29. Appl., 23.5.27. Ger., 29.5.26).—See B.P. 271,906; B., 1928, 849.

Preserving difficultly-soluble dyestuffs in a state of fine division. F. RISSE and E. FISCHER, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,719,944, 9.7.29. Appl., 5.4.27. Ger., 22.4.26).—See B.P. 269,918; B., 1928, 666.

Photographic desensitisers (B.P. 314,144).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Use of dispersing agents for washing textiles with hard water. F. MÜNZ (Z. angew. Chem., 1929, 42, 734—736).—In presence of a suitable protective colloid actual precipitation of calcium and magnesium soaps may be avoided. A particularly suitable substance appears to be "Intrasol," a material which contains sulphonic derivatives of higher fatty acids in addition to esters of sulphuric acid.

J. S. CARTER.

Analysis of union materials [containing artificial silks]. L. L. LLOYD and E. PRIESTLEY (J. Soc. Dyers and Col., 1929, 45, 201—204).—The well-known method for separating wool and cotton by treatment with caustic soda is not suitable for the analysis of wool materials containing artificial silk because the last-named fibre is substantially soluble in the alkali. Artificial cellulose silks, including viscose, Chardonnet, and cuprammonium silks, are more rapidly hydrolysed than is cotton by dilute sulphuric acid, and this forms the basis of new methods of analysis. For wool and cellulose artificial silk unions, 0.5—2.0 g. is conditioned under standard conditions, immersed and pressed for 20 min. at 50° in 50 c.c. of sulphuric acid of *d* 1.345, the acid decanted, the residual wool fibre well washed with water, dilute ammonia, then water, well dried between filter paper, and conditioned as before; the loss of weight is equal to the artificial silk originally present in the material. In wool, cellulose artificial silk, and cotton unions the artificial silk is first determined as described above, and then the cotton in the residual mixture of wool and cotton by similar hydrolysis using sulphuric acid of *d* 1.345 for 24 hrs. at 25° whereby the cotton is separated from the wool which remains unattacked. The results obtained by these methods agree closely with those obtained by methods of dissection. The above methods are not suitable for the analysis of unions containing real silk since this loses considerably in weight and becomes brittle, but methods suitable for real silk unions which may also contain cotton, but less satisfactory when the silk is undischarged or is heavily coloured with basic dyes, have been devised utilising the discovery that only the cellulose artificial silks are rendered water-soluble by

treatment for 15 min. at 50° with hydrochloric acid of $d_{1.105}$. Cellulose acetate silk in union materials may be determined by extraction with acetone. Cellulose artificial silks are equally soluble in 1% solutions of caustic soda and caustic potash (cf. Hall; B., 1929, 596), the solubilities of Chardonnet, viscose, and cuprammonium silks after boiling for 1 hr. being 13–14%, 6–10%, and 4–7%; the solubility of Chardonnet silk under similar conditions in 20% caustic soda was 5–6%.
A. J. HALL.

Cellulose fibre-length. G. K. BERGMAN and A. BACKMAN (Papier-Fabr., 1929, 27, 449–456).—The fibre lengths of unbleached commercial sulphite- and sulphate-pulps have been determined, using the conceptions "average fibre-piece length" and "equilibrium mean length." The significance and measurement of these quantities, and changes produced in them by mincing, bleaching, etc. of the wood pulp are discussed.

B. P. RIDGE.

Absorptive power of cellulose. S. R. H. EDGE (J.S.C.I., 1929, 48, 118–121 r).—The adsorption of water, acids, alkalis, salts in general, and alum in particular by the wood and cotton cellulose pulps as used by papermakers are investigated experimentally. Attempts to find the isoelectric point of cellulose, as has been done for gelatin, by investigating the water adsorption at different p_H values failed, none of the methods tried being sufficiently accurate. In general the results give no evidence for the amphoteric nature of cellulose, and whilst they do not disprove its amphoteric nature they are adequately explicable on the assumption of colloidal adsorption of salts and acids in the skin layer of the fibres. The great importance of the traces of ash-producing constituents always found in the purest commercial celluloses for the adsorptive phenomena found is proved.

Measurement of the viscosity of celluloses. A. KÜNG and E. SEGER (Papier-Fabr., 1929, 27, 433–436).—A comparatively rapid method of measurement of the viscosity of cellulose after its conversion into xanthate by the viscose method, but without ageing or ripening, is described. The cellulose, cut up into 1-mm.² pieces, is mixed thoroughly, and sufficient is weighed out to give 0.9 g. of dry material. This is steeped in 10 c.c. of 17.5% sodium hydroxide solution at 20° for 2 hrs., squeezed out to a weight of exactly 3 g., and treated with carbon disulphide vapour in a special bottle. The latter has a hollow stopper in which the necessary amount of carbon disulphide is placed, and vapour from the solvent escapes into the interior of the bottle through a small hole and there converts the alkali-cellulose into xanthate, which is subsequently dissolved out by sodium hydroxide solution. The viscosity of the viscose solution thus obtained is measured in an Ostwald viscosimeter, and the whole procedure can be completed in 9½–10 hrs. as against the several days necessary for the ordinary viscose conversion method.

B. P. RIDGE.

Viscosity of viscose. I. Change in viscosity of viscose during ripening. G. KITA, S. IWASAKI, S. MASUDA, and K. MATSUYAMA (Kolloid-Z., 1929, 48, 270–276).—Measurements of the change in the

viscosity of viscose during ripening have been made, using different kinds of cellulose and both the falling-sphere and the Ostwald viscosimeters. The following results were obtained. The viscosity change when measured with the falling-sphere viscosimeter is marked by a fall over the first few days, and is followed by a slow rise, which suddenly becomes steep shortly before coagulation sets in. The rate of change depends on the kind of cellulose employed. The behaviour of diluted viscose, measured with the Ostwald viscosimeter, varies greatly according to the kind of cellulose, but either is of the type mentioned above or the minimum is followed directly by a sharp rise. The ability to form threads varies in the same way as the viscosity measured by the falling-sphere method. The viscosity of viscose and the properties of the thread spun from it are related to the content of α -cellulose and to the viscosity of the solution in ammoniacal cupric oxide.
E. S. HEDGES.

Rôle of dielectric constants, polarisation, and dipole moment in colloid systems. IV. Swelling of cellulose acetate in single organic liquids. I. SAKURADA (Kolloid-Z., 1929, 48, 277–283).—The degree of swelling of acetone-soluble cellulose acetate in a number of organic liquids has been measured. All the solvents and swelling media for cellulose and acetate have relatively high values of molecular polarisation and dipole moment. Substances which are free from or poor in dipoles are neither solvents nor swelling agents for cellulose acetate.
E. S. HEDGES.

Mechanical wood pulp. P. KLEM (Papier-Fabr., 1929, 27, 467–469).—Mechanical wood pulp consists of a mixture of substantially unchanged wood fibres with fibres which have been broken, torn, or ground into smaller fragments and form the so-called meal-stuff. The relative proportions of these two constituents affect the properties of papers made from the pulp, and their determination is a matter of importance. The Hurum apparatus for this purpose is described. It consists essentially of a glass cylinder fitted with a stirrer, and a metal base with a two-way tap through which water may be admitted to or removed from the cylinder. Sieves made from coarse or fine cloth are placed in the cylinder with the pulp and the meal-stuff is washed through whilst the long fibres are retained. With increasing meal-stuff content the degree of grinding and the tearing length of the pulp as a whole increase. The strength of a mixture of mechanical wood pulp and cellulose (as shown by tearing length) may exceed that of either of these materials alone.
B. P. RIDGE.

1:4-Dioxan. REID and HOFMANN.—See III.

PATENTS.

Process of laundering and detergent. Process of dry-cleaning. L. HAGER and J. POPPERMAN, ASSRS. to PINE-O-PINE Co. (U.S.P. 1,710,974–5, 30.4.29. Appl., [A] 4.5.26, [B] 13.7.25).—(A) Rosin is treated with caustic soda solution in amount at least sufficient to saponify it, and mixed with excess of pine oil. The goods may be washed in a solution of 0.4–1% of soap containing 0.4–0.8% of the emulsion produced by diluting the detergent with 8 vols. of water. (B) The detergent may be diluted with 2–480 vols. of gasoline for dry cleaning.
R. BRIGHTMAN.

Manufacture of sized fibrous product. H. McC. SPENCER (U.S.P. 1,714,052, 21.5.29. Appl., 12.3.26).—Sized paper is made by suspending the pulp in water, adding a peptised colloidal solution of aluminium hydroxide (cf. U.S.P. 1,513,566; B., 1925, 41) and a rosin emulsion, and agitating the mixture.

F. R. ENNOS.

Products of cellulose by decomposition of vegetable fibres. O. C. STRECKER (U.S.P. 1,705,424, 12.3.29. Appl., 25.2.27. Ger., 30.9.24).—Vegetable fibre is heated at 70–100° with a solution containing a soluble metallic derivative of a phenol, naphthol, hydrogenated phenol or naphthol, or sulphonic, carboxylic, etc. derivatives thereof, a further amount of metallic salt being added during the digestion to maintain the original alkali concentrated and prevent composition of the lye by acids. Alternatively digestion may be effected under pressure, *e.g.*, at 150–160° for 4–6 hrs.

R. BRIGHTMAN.

Increasing the tensile strength of artificial silk. I. BOLOGNESI (B.P. 313,981, 13.3.28).—All mechanically and molecularly associated water is removed by continuously treating the material with an anhydrous dehydrating organic liquid in which it is insoluble, *e.g.*, light petroleum, which, after distilling and condensing in a suitable apparatus is allowed to drip on to the artificial silk and is then periodically withdrawn therefrom. The artificial silk is finally freed from the dehydrating agent in a drying chamber. F. R. ENNOS.

Fining of freshly spun artificial silk threads. NORDDEUTS. VERWALTUNGS-GES.M.B.H. (B.P. 283,481, 5.1.28. Ger., 10.1.27).—Threads spun from viscose or cuprammonium cellulose which, after or during coagulation, have been brought to a highly swollen state by solvent or swelling agents, *e.g.*, zinc chloride or 70% sulphuric acid either alone or mixed with sulphates, glucose, etc., are stretched positively by guiding over a number of rotating rollers of acid-resisting material, of successively increasing peripheral speed; the threads are finally washed. F. R. ENNOS.

Manufacture of artificial threads or filaments from cellulose esters or ethers by the dry-spinning or evaporative method. BRIT. CELANESE, LTD., R. W. PAYNE, and R. P. ROBERTS (B.P. [A] 314,404 and [B] 314,414, 23.12.27).—Artificial filaments presenting reduced lustre or a dull, matt, or lustreless appearance are produced by dry spinning (A) either a solution of a cellulose ester or ether in a volatile solvent into an evaporative atmosphere containing one or more organic precipitants for the cellulose derivative, *e.g.*, alcohols, hydrocarbons, esters, or ethers, or a solution of the ester or ether in a mixed solvent containing one or more volatile solvents and one or more of the above precipitants, (B) a solution of the ether or ester in volatile solvents containing water; the spinning conditions are adjusted so that the precipitating action is exerted on the substance composing the filaments, while still substantially wet. F. R. ENNOS.

Manufacture of threads, ribbons, films, etc. from solutions of cellulose esters or ethers. RUTH-ALDO CO. INC., Assees. of H. L. BARTHELEMY (B.P. 282,790, 28.12.27. Fr., 28.12.26).—Solutions of cellulose esters or ethers in

organic solvents are coagulated in an aqueous solution or emulsion of (a) alcohols, polyalcohols, hydrocarbons or fatty acids of high mol. wt. or their esters, or animal and vegetable oils, and (b) an emulsifying agent such as alkali oleate, ricinoleate, or stearate, Twitchell's reagent, or gum tragacanth. F. R. ENNOS.

Removal of sulphur from articles made from viscose. I. G. FARBERIND. A.-G. (B.P. 293,833, 5.7.28. Ger., 13.7.27).—Viscose artificial silk is treated with a solution obtained by extracting the crude cellulose xanthate with an alcohol or an aqueous alcohol used directly, after dilution, or after evaporation to dryness and re-dissolving in water or alcohol.

F. R. ENNOS.

Cellulose composition for esterification [nitration]. Composition for conversion into cellulose derivatives [nitrates]. G. A. RICHTER, ASSR. to BROWN CO. (U.S.P. 1,700,595–6, 29.1.29. Appl., 3.3.27).—(A) Mercerised cellulose fibre is sheeted on a paper machine and regenerated cellulose is deposited upon it from a cellulose xanthate solution. The whole may then be nitrated. (B) A mixture of mercerised and unmercerised pulp is sheeted in a paper machine, and is then suitable for nitration. C. HOLLINS.

Working-up of ethylcellulose. I. G. FARBERIND. A.-G. (B.P. 288,143, 22.3.28. Ger., 31.3.27).—Ethylcellulose containing at least two ethoxy-groups is dissolved at an elevated temperature in a solvent which will not dissolve the ether in the cold, *e.g.*, benzene, light petroleum, and the solution, after purifying while hot, is cooled, whereby the ethylcellulose separates. It may then be worked up into plastic masses, while still wet, with solvent at an elevated temperature, and afterwards cooled. F. R. ENNOS.

Manufacture and treatment of cellulose esters. H. DREYFUS (B.P. 314,918–9, 28.3.28).—Cellulose is esterified by means of (A) an alkoxyacetic acid, or (B) partly an alkoxyacetic acid and partly an aliphatic acid. In each case a mixture of acid (methoxy- or ethoxy-acetic, or acetic) and anhydride in presence of sulphuric or hydrochloric acid and/or ferric chloride etc. is used, and acylation may be followed by partial hydrolysis. The new esters may be spun and dyed in the same manner as cellulose acetate. C. HOLLINS.

Homogeneous esterification of cellulose. RUTH-ALDO CO., INC., Assees. of H. L. BARTHELEMY (B.P. 282,788, 28.12.27. Fr., 28.12.26).—Cellulose or nitrocellulose esters with a low percentage of nitrogen, after a preliminary softening treatment with hot vapours of glacial acetic acid, are acetylated with acetic anhydride and sulphuric acid in at least three stages, each fraction of the acetylating agent being added only after the previous one has reacted to equilibrium. In this way local reactions are counteracted, and homogeneous acetylcelluloses are obtained. [Stat. ref.]

F. R. ENNOS.

Esterification of cellulose. RUTH-ALDO CO., INC., Assees. of H. L. BARTHELEMY (B.P. 282,794, 28.12.27. Fr., 28.12.26).—Esters of low viscosity, high plasticity and tensile strength, and good elasticity and keeping properties are made by subjecting cellulose to a pre-

liminary oxidation with an aqueous solution composed of (a) a direct oxidising agent, *e.g.*, alkali peroxide, perborate, or hydrogen peroxide, (b) a base such as alkali or magnesium hydroxide, and (c) an alkali resin, a soap, or sodium or ammonium sulphurinate, followed by esterification. F. R. ENNOS.

Moisture-proof [cellulose] material. DU PONT Cellophane Co., INC. (B.P. 283,109, 23.12.27. U.S., 3.1.27).—A sheet of regenerated cellulose is coated with a composition consisting of 30–70% of a base such as a cellulose ether or ester, 30–60% of a gum or resin, 2–6% of a wax, *e.g.*, paraffin, and 5–30% of a plasticiser such as an aryl or alkyl phosphate or phthalate, the mixture being applied as a solution in a mixed solvent containing alcohol, toluene, butyl acetate, and ether or ethyl acetate. F. R. ENNOS.

Treatment of plant fibres. C. MASSE, Assr. to Soc. CIVILE DES PROC. MASSE (U.S.P. 1,719,066, 2.7.29. Appl., 10.2.27. Fr., 22.2.26).—See B.P. 266,344; B., 1927, 839.

Making bands of artificial fibres. I. G. FARBERIND. A.-G. (B.P. 286,603, 29.2.28. Ger., 5.3.27).

Deodorisation of gases (B.P. 314,187).—See I. **Suspensions etc.** (B.P. 287,464).—See III. **Wool fat** (B.P. 314,586–7).—See XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching [cotton] without chemicking. Report by C. SUNDER on Sealed Note No. 2178, 21.5.12, of A. DONDAIN (Bull. Soc. Ind. Mulhouse, 1929, 95, 225–229).—Dondain has proposed a modification of the process described in G.P. 240,037, in which air (oxygen) is used for aerating the kier liquor during the boiling of cotton materials for the purpose of obtaining a white which does not require subsequent chemicking, consisting of replacing the air by nascent oxygen produced by electrolysis of the kier liquor itself. Sunder criticises the modified process unfavourably, pointing out that cotton is liable to deterioration when treated with a hot caustic alkali in the presence of oxygen, that the electrolytic device prevents filling the kier completely with liquor, although this is generally desired, and that it is doubtful whether more than a half-bleach can be obtained, although the aeration of a kier liquor does largely destroy its brownish colour. A. J. HALL.

[Properties of] chrome colours on wool. E. NOELTING (Sealed Note No. 2367, 2.1.20. Bull. Soc. Ind. Mulhouse, 1929, 95, 217–219). Report by M. BATTEGAY and J. LICHTENBERGER (*Ibid.*, 219–221).—The prevailing theory that during the oxidation of azo dyes derived from chromotropic acid on wool by treatment with a dichromate the oxidised dye forms a colour lake with the resulting chromium oxide is disputed, it being suggested that the dye remains unoxidised but forms a complex salt with the chromium oxide formed during oxidation of the wool. Such complex salts, which are easily soluble in water, have been prepared apart from the wool fibre by treating chromotrope dyes with chromium fluoride and formate; the preparation of similar compounds with aluminium instead of chromium was only partly successful. The usual red solu-

tions of Chromotrope 2R and 2B become deep blue on addition of chromium fluoride or formate, and the chromium is then not precipitated by addition of sodium carbonate, whilst the acidified solution dyes wool a bluish-black shade similar to that obtained by after-chroming wool dyed with the same chromotropes in the usual manner; complete exhaustion of the dye-bath is obtained and the method satisfactorily used with various commercial chromotrope dyes. Chromotropic and H-acids, but not 1:8-diamino- or 2-hydroxynaphthalene-3:6-disulphonic acids, also form water-soluble complex salts with salts of chromium, aluminium, and iron. Certain azo dyes derived from *o*-aminophenols behave similarly to the chromotropes. The report draws attention to a number of patents relating to the manufacture of wool dyes containing chromium and to recent work in which the chromium lakes of dyes derived from chromotropic acid were obtained in crystalline form. A. J. HALL.

Effect of light on coloured [cotton] fabric. III. (MISS) E. HIBBERT (J. Soc. Dyers and Col., 1929, 45, 204–205; cf. B., 1928, 708, 854).—Anthracene (in substance) is converted into anthraquinone by exposure to light, and more easily and rapidly in cotton fabric impregnated with anthraquinone from alcoholic solution. Purpurin fades rapidly on cotton, wool, and cellulose acetate silk. Extraction of the faded cotton by carbon tetrachloride yielded a colourless substance, m.p. 130°, considered to be a eutectic mixture of phthalic acid and phthalic anhydride. The faded parts of cotton fabric impregnated with phthalic acid and anhydride have, after extraction with carbon tetrachloride, a greater affinity for methylene-blue than similarly impregnated but unexposed parts. After exposure to light of cotton impregnated with sodium oxalate, the cotton also has an increased affinity for methylene-blue. A. J. HALL.

Printing with indanthrene and other vat dyes. FRÈRES KOEHLIN (Sealed Note No. 2141, 30.12.11. Bull. Soc. Ind. Mulhouse, 1929, 95, 221–223). Report by V. SILBERMANN (*Ibid.*, 224).—A printing process which allows the printed vat dyes to be fixed at any time after printing and also the simultaneous printing of vat dyes and oxidation colours such as Aniline Black and Paramine Brown, consists of printing fabric with a thickened paste of the vat dye, drying, padding with an alkaline reducing solution containing caustic soda or sodium carbonate and Rongalite, then steaming for about 10 min. in an air-free Mather-Platt, and soaping. Alternatively, the alkaline reducing solution may be applied to the fabric before printing with the vat dyes. Silbermann reports favourably on the process, but points out that it is not possible to print simultaneously with Aniline Black if the fabric is first prepared with the reducing solution. A. J. HALL.

Printing sulphur dyes without attacking the printing rollers. P. HOLM (Sealed Note No. 1960, 27.12.05. Bull. Soc. Ind. Mulhouse, 1929, 95, 354–357). Report by A. WOLFF (*Ibid.*, 358–359).—A satisfactory printing paste containing sulphur dyes may be prepared using caustic soda and arsenious oxide instead of a hyposulphite or glucose, and it has the advantage that it does not attack copper rollers; phenol is added to

the paste to dissolve the reduced dye. The amount of arsenious oxide required is dependent on the purity of the sulphur dye, but 60 pts. of dye require 20 to 40 pts. of oxide. The printing pastes may be resisted by means of pastes containing zinc and ammonium chlorides. Wolff reports favourably on the process. A. J. HALL.

Singeing of cotton and the formation of oxycellulose. G. E. HOLDEN (J. Soc. Dyers and Col., 1929, 45, 205; cf. B.P. 295,203; B., 1928, 745, and B., 1929, 92).—Comparison of the fibre tips cut from singed and non-singed cotton pile fabrics showed that the tips from the singed fabric had a decreased affinity for Chlorazol Sky Blue FF, an increased affinity for methylene-blue, and a lower viscosity in cuprammonium solution, thus indicating the formation of oxycellulose in singeing processes. Fabric not uniformly moist at the time of singeing is found to have a greater affinity for dyes after singeing in those parts containing the greater amount of moisture. The singeing of faultily-dyed cotton fabric frequently assists levelness in a subsequent stripping process. A. J. HALL.

PATENTS.

Wetting, cleansing, and emulsifying agents. COMP. NAT. DE MAT. COL. & MANUF. DE PROD. CHIM. DU NORD RÉUNIES (ÉTABL. KUHLMANN) (F.P. 630,615, 9.3.27).—Naphthalene is sulphonated (*e.g.*, with 29% oleum) and condensed with acetone at 120–125°. The acetone may be replaced by acetone oil, especially that boiling below 200°. C. HOLLINS.

Colloidal mordant bath and its manufacture. L. R. PARKES (U.S.P. 1,710,000, 23.4.29. Appl., 14.12.27).—Solutions of a metallic salt of a weak acid and of a fixing agent are mixed, the more dilute solution being added to excess of the more concentrated; *e.g.*, 25 c.c. of 0.1*N*-sodium oleate are added to 100 c.c. of 0.2*N*-aluminium acetate. R. BRIGHTMAN.

Dye set [for fixing dyes on textiles]. N. M. CROUCH (U.S.P. 1,702,155, 12.2.29. Appl., 9.2.26).—An aqueous solution of a mixture (1 oz. for every 2 lb. of textile) of equal pts. of aluminium acetate, magnesium chloride, and lead acetate is used as a fixing bath (1 oz. per gal.) for dyed textiles. C. HOLLINS.

Manufacture of dye preparations [for acetate silk dyeing]. SOC. CHEM. IND IN BASLE (Swiss P. 122,569, 22.5.26).—Insoluble azo or vat dyes are pasted with a solution of humic ester substances made alkaline with ammonia. The pastes may be diluted with water and give clear shades on acetate silk in spite of the dark colour of the bath. C. HOLLINS.

Dyeing of cellulose esters and ethers. IMPERIAL CHEM. INDUSTRIES, LTD., and E. A. SPEIGHT (B.P. 314,651, 30.5.28).—Black shades are obtained direct on acetate silk by means of disazo dyes of the type: amine of the benzene series $\rightarrow \alpha$ -naphthylamine or Clevic acid \rightarrow 1:5-aminonaphthol or 1:5-naphthylenediamine. As first components are mentioned aniline, *o*- and *p*-toluidines, *m*-xylydine, *p*-chloroaniline, 5-chloro-*o*-toluidine. C. HOLLINS.

Dyeing of artificial [regenerated cellulose] silk. COURTAULDS, LTD., C. M. WHITTAKER, and T. M. HARRISON (B.P. 314,671, 26.6.28).—Level dyeings on

viscose silk are secured by addition to the dye-bath of 1% of sodium chloride and 1% of β -naphthol (on weight of liquor used). Dyed material may be levelled by a similar treatment. C. HOLLINS.

Coloration of fabrics made of or containing cellulose derivatives [esters]. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 314,208, 2.5.27).—Penetration of knitted or woven fabrics of acetate silk etc. is secured by applying the dye in an organic solvent (*cf.* B.P. 282,036; B., 1929, 169) by means of a spray. C. HOLLINS.

Drying and carbonising textile fabrics. J. H. WALSH, ASSR. TO JOHNS-MANVILLE, INC. (U.S.P. 1,714,062, 21.5.29. Appl., 16.3.25).—A web of fabric is moved through a series of vertical runs in a heat-insulated chamber divided into two compartments, in the first of which it is subjected to blasts of heated air impinging perpendicularly against its surface, and in the second to direct radiant heat and highly heated still air alternately applied. F. R. ENNOS.

Colour printing paste. J. G. KERN and C. J. SALA, ASSRS. TO E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,705,818, 19.3.29. Appl., 9.2.27).—Technical "trichthanolamine" [a mixture containing 65% of tri-(β -hydroxyethyl)amine] is added to vat dye printing pastes to facilitate reduction and to give uniform prints. C. HOLLINS.

Composition for imparting to mercerising lye the property of easily wetting cotton fabrics and yarns. NAAML. VENN. J. A. CARP'S GAREN-FABRIEKEN (B.P. 305,664, 4.2.29. Holl., 10.2.28).—Caustic soda lyes used in mercerising readily wet out cotton after the addition of a mixture of one or more phenols and one or more saturated aliphatic or aromatic alcohols, particularly benzyl alcohol, the increase in wetting-out power being largely dependent on the ratio of the phenols to alcohols. Liquors thus treated may be used in mercerising grey cotton materials not previously scoured. *E.g.*, 18 kg. of cresol and 2 kg. of benzyl alcohol are added to 1000 litres of caustic soda (*d* 1.26–1.32). A. J. HALL.

Treatment of fabrics [of acetate silk etc.]. BRIT. CELANESE, LTD. (B.P. 299,058, 19.10.28. U.S., 21.10.27).—Acetate silk fabrics are impregnated at 40° with a 1–5% solution of basic aluminium acetate acidified with acetic acid, and may then be ironed at 230–240° without sticking and without increase of lustre when ironed damp. C. HOLLINS.

Improving vegetable fibrous material. HEBERLEIN & Co. A.-G. (B.P. 301,392, 26.11.28. Ger., 28.11.27).—In producing special effects on cellulosic material by subjecting it to two or more successive treatments with swelling agents such as concentrated acids or salt solutions, coagulation of the cellulose between the treatments is avoided by omitting to wash out with water the first swelling agent before treatment with the second, or by effecting such washing with an inert organic liquid which does not produce coagulation; increased or new effects are thus obtained. *E.g.*, cotton fabric is embossed with a pattern as described in B.P. 268,389 (B., 1928, 332), then drawn during about

30 sec. at ordinary temperature through an ammoniacal solution of copper oxide (20 g. of copper per litre), squeezed, and directly mercerised with caustic soda, whereby a fabric is obtained in which the embossed portions have retained their original character, but the remainder has become stiff and linen-like. A. J. HALL.

Dyeing or colouring products made with cellulose acetate. G. H. ELLIS, ASSR. to CELANESE CORP. OF AMERICA (U.S.P. 1,719,324, 2.7.29. Appl., 25.9.23. U.K., 27.1.23).—See B.P. 219,349; B., 1924, 906.

Machines for treating skeins. SOC. VEUVE BONNET AINÉ ET SES FILS (B.P. 301,053, 29.6.28. Fr., 25.11.27. Addn. to B.P. 289,410).

Dyes, dyeing, etc. (B.P. 314,589 and 314,763). **Coloured compositions etc.** (B.P. 314,451).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Contact sulphuric acid process. A. O. JAEGER (Ind. Eng. Chem., 1929, 21, 627—632).—Converter design for the contact sulphuric acid process is discussed with especial reference to the Selden Company's (Jaeger-Bertsch) new converter, which is cooled by a double countercurrent, automatically regulated heat-exchange system, and compensated for converter shell cooling. The catalyst used contains as active principle non-siliceous base-exchange substances (of zeolite type) in which vanadium oxide is present in the non-exchangeable nucleus; this catalyst has great resistance to high temperatures and long life, and is unaffected by gaseous poisons for platinum. In tests with 135 litres of burner gas (7% SO₂) per hour per 200 c.c. of contact mass, conversions of 97—98.8% were obtained; at 200 litres per hour the conversion was practically unchanged, and even at 300 litres it decreased by less than 1%. With a 5% burner gas the conversion was 99.5% and with 9% SO₂ above 97%. E. LEWKOWITSCH.

Utilisation of natural gases for the ammonia process. H. LIANDER (Trans. Faraday Soc., 1929, 25, 462—472).—The reactions into which methane, chosen as typical of natural gases, and air, steam, or carbon dioxide respectively may enter are discussed from the viewpoint of Nernst's heat theorem. Two series of experiments, dealing with the reaction between methane and oxygen, with and without a catalyst, at temperatures ranging from 550° to 1150°, are described, one set being static, whilst in the other the gases were passed continuously through the reaction chamber. The results suggest that natural gases may be employed with advantage in the production of the initial gases for the ammonia process, and it is further suggested that a suitable mixture of natural gases, air, and steam might be passed over an efficient catalyst at 1000° or above. Parts of the natural gases might first be burned with air and the products then led with more natural gas and steam over the catalyst. The carbon dioxide and any undecomposed methane could be removed by the Bosch method. H. T. S. BRITTON.

Analysis and constitution of ammoniacal and spent liquors. T. L. BAILEY (65th Ann. Rep. Alkali etc. Works, 1928, 22—26).—Removal of sulphide pre-

vious to determination of the oxygen absorption value cannot be carried out by means of soluble lead salts or even lead carbonate. It is shown that with the latter reagent dihydric phenols are removed. The method recommended is to acidify with 30% sulphuric acid, neutralise with calcium carbonate, and precipitate with zinc sulphate solution. Tests show that this procedure gives accurate results. It has been found that polyhydric phenols do not react with iodine in a solution made definitely acid with sulphuric or hydrochloric acid. The iodine method can therefore be used for the determination of thiosulphate and sulphide if carried out as prescribed. The difference between oxygen value as determined and that due to known constituents is probably due to substances of the pyrocatechol type. A determination of total sulphur indicated the absence of organic sulphur compounds in spent liquor. Ammoniacal liquor contained a small proportion of sulphur other than sulphate, sulphide, thiocyanate, and thiosulphate. C. IRWIN.

Determination of total and available lime. N. SHILKIN (Chem. Eng. Min. Rev., 1929, 21, 354—355).—Total lime is determined by precipitation as oxalate which is redissolved in 1 c.c. of nitric acid; the solution is evaporated to dryness and the residue ignited, gently at first, finally over a blast burner, and weighed as lime. Available lime is best determined by the method of Shaw and others (B., 1928, 365). A. R. POWELL.

Determination of copper and nitrite in solutions of cuprammonium hydroxide. E. BUTTERWORTH and H. A. ELKIN (J.S.C.I., 1929, 48, 127—128 T).—Modifications in the usual methods for the determination of copper and nitrite in cuprammonium hydroxide are described. The presence of the nitrite is shown to be responsible for inaccuracies in the thiosulphate titration for copper. Decomposition of the nitrite by acidification with sulphuric acid obviates the inaccuracy. A modification of the nitrometer method for determination of nitrite consists in the neutralisation of the alkali with dilute sulphuric acid (25% by vol.) before the addition of the concentrated acid.

Formation of a manganese carbide decomposable by water from manganese oxide and methane at relatively low temperatures. F. FISCHER and F. BANGERT (Brennstoff-Chem., 1929, 10, 261—265).—Manganese carbide is formed when methane is passed over the oxide (either Mn₂O₄ or MnO₂) at 1050—1200°. The same carbide is formed at lower temperatures, 800—1000°, if 10% of barium oxide is added to the manganese oxide, or if the reaction is carried out under 6—12 mm. pressure. Addition of alumina has the same effect, but in a smaller degree. The addition of nickel, chromium, or iron oxide favours the formation of a carbide which is stable to water but is decomposed by acids. The former carbide reacts with water, 90% of its carbon content being thereby converted into gaseous hydrocarbons; the average composition of the gas evolved is C_nH_m 2.3%; H₂ 52.8%; C_nH_{2n-2} 44.9%. The carbide is shown to have the constitution (Mn₅C₂)_x. A. B. MANNING.

Synthetic carbamide. YAKOVKIN.—See III. **Decomposition of beryl.** ILLIG and others.—See X.

Exchange reactions in permutits. UNGERER. Base exchange in orthoclase. BREAZEALE and MAGISTAD.—See XVI.

PATENTS.

Treatment of filter liquor [from ammonia-soda process]. H. AHLQVIST (U.S.P. 1,696,224, 25.12.28. Appl., 17.11.26).—Filter liquor from sodium hydrogen carbonate produced in the ammonia-soda process passes to a still successively through a heat-exchanger and a preheater in countercurrent direct contact with the still vapours. Dilute ammonia liquor and ammonia and carbon dioxide are withdrawn from the heat exchanger, which can be water-cooled, waste liquors escaping from the foot of the still. The preheated filter liquor is treated with milk of lime in passing from the preheater to the still.

R. BRIGHTMAN.

Conversion of ammonium chloride into ammonia and hydrochloric acid. J. KESSLER (U.S.P. 1,718,420, 25.6.29. Appl., 18.5.26).—Ammonium chloride vapour is passed into fused ammonium bisulphite, and the hydrochloric acid produced collected. A rapid current of air free from carbon dioxide is passed through the retort to expel residual hydrochloric acid, and ammonia is liberated by passing superheated steam through the heated mass.

F. G. CLARKE.

Preparation of alkali sulphates. CHEMIEVERFAHREN G.M.B.H. (B.P. 300,630, 16.10.28. Ger., 18.11.27).—An alkali chloride is treated with magnesium sulphate in the presence of an aqueous ammoniacal solution of ammonium chloride, in such concentrations that solid alkali sulphate is formed free from magnesium salts.

H. ROYAL-DAWSON.

Manufacture of sodium bicarbonate and ammonium chloride. W. GLUUD and B. LÖPMANN (U.S.P. 1,710,636, 23.4.29. Appl., 10.1.25. Ger., 21.2.24).—A solution of the chlorides and bicarbonates of sodium and ammonium is treated with sodium chloride in presence of another readily soluble sodium or ammonium salt other than the chloride. *E.g.*, ammonium bicarbonate is added at 30° to a solution of ammonium nitrate saturated with sodium chloride; the precipitated sodium hydrogen carbonate is removed, and after adding the equivalent amount of sodium chloride and stirring at 20° the ammonium chloride precipitated is collected.

R. BRIGHTMAN.

Decomposition of calcium carbonate and manufacture of calcium carbide. G. PATART and H. NIELSEN (B.P. 314,819, 30.12.27).—A mixture of calcium carbonate with sufficient coal to yield the carbon necessary for the production of calcium carbide on subsequent heating in an electric furnace is heated in a rotary retort as described in B.P. 276,497, 287,037, and 287,381 (B., 1928, 356, 395) to produce coke free from graphitic carbon, and in the presence of a current of combustion gases containing less carbon dioxide than that normally present in combustion gases; *e.g.*, the gases are diluted with carbon monoxide from the electric furnace and/or with stripped distillation gases from the retort.

L. A. COLES.

Manufacture of calcium hypochlorite. (A) A. GEORGE and M. C. TAYLOR, (B) A. GEORGE, (C) J. A.

GUYER and M. C. TAYLOR, (D) R. B. MACMULLIN and A. GEORGE, Assrs. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,718,284—7, 25.6.29. Appl., 4.8.26).—(A) A slurry consisting of a eutectic solution of calcium hypochlorite and alkali chloride, together with lime and an alkali compound which reacts with lime and chlorine to give calcium hypochlorite and alkali chloride, is chlorinated. (B) The above alkali compound is added to milk of lime, which is then chlorinated. (C) A solution of caustic alkali is chlorinated, and calcium chloride treated with the alkali hypochlorite obtained. (D) A mixture of caustic alkali with excess of lime is chlorinated in the presence of water, and calcium hypochlorite recovered from the product.

F. G. CLARKE.

Manufacture of barium peroxide. B. LAPORTE, LTD., I. E. WEBER, and V. W. SLATER (B.P. 315,151, 6.10.28).—Barium oxide suitable for peroxide manufacture is made from dense barium carbonate containing a small proportion of an alkali salt, prepared, *e.g.*, by adding a small proportion of an alkali salt to barium carbonate precipitated otherwise than by alkali carbonate.

W. G. CAREY.

Production of lead oxide. G. B. PRICE, and QUIRK, BARTON & Co., LTD. (B.P. 315,128, 16.8.28).—A vessel for the melting and oxidation of lead is provided with an oscillating, horizontal surface sweeper having attached combs, a screening plate to prevent admixture of the molten lead with the suspended litharge, and beaters rotating in a vertical plane, arranged, *e.g.*, in two pairs on opposite sides of the pan, the two members of each pair rotating in opposite directions.

L. A. COLES.

Working-up of calcined zirconium-lime products. KALI-CHEMIE A.-G., Assecs. of RHENANIA-KUNHEIM VEREIN CHEM. FABR. A.-G. (B.P. 300,271, 2.11.28. Ger., 11.11.27).—The products are freed from a portion of the lime by extraction with aqueous sulphurous acid, the residue is dissolved in hydrochloric acid, and the solution treated in the cold with the calcium sulphite liquor obtained in the first process, basic zirconium sulphite being separated by gradual heating.

W. G. CAREY.

Leaching of solid raw materials [phosphates] with acid solutions. KUNSTDÜNGER-PATENT-VERWERTUNGS-A.-G., Assecs. of F. G. LILJENROTH (B.P. 301,306, 23.10.28. Swed., 26.11.27).—A system of leaching and countercurrent decantation (*e.g.*, a Dorr plant) is combined with an absorber for acid vapours. *E.g.*, raw phosphate and nitric acid are leached together, and the mud of calcium sulphate (precipitated by ammonium sulphate) and insoluble matter is pumped up the series of washing settlers, countercurrent to a wash liquor of ammonium sulphate. A concentrated solution of ammonium phosphate overflows from the lowest settler for treatment elsewhere. A more dilute solution from the lowest settler but one is caused to absorb nitrous fumes and the resulting acid liquor serves to leach the phosphate as above.

B. M. VENABLES.

Manufacture of neutral sodium phosphate. C. F. BOOTH and A. B. GERBER, Assr. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,700,972, 5.2.29. Appl., 19.7.24).—Sodium phosphate dodecahydrate free from alkali is crystallised from a solution of *d* 1.30 in which the

ratio of the phenolphthalein to the methyl-orange titration is 0.432 to 0.451.

R. BRIGHTMAN.

Zeolite. J. A. BERTSCH and A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,701,075, 5.2.29. Appl., 27.2.26).—Zeolites containing quadrivalent vanadium in non-exchangeable form are obtained by reducing quinquevalent vanadium compounds, converting the quadrivalent vanadium compounds into soluble vanadates, and treating the latter with soluble silicates in alkaline solution. Examples include vanadyl zeolites of copper and silver, potassium and chromium, etc. R. BRIGHTMAN.

Manufacture of fluorine compounds of low silicon content. VEREIN F. CHEM. U. MET. PROD. (B.P. 289,383, 24.4.28. Czechoslov., 25.4.27).—In the manufacture of hydrofluoric acid from fluorspar by heating with sulphuric acid, the greater part of the water in the issuing gases is removed by fractional condensation or by treatment with oleum and the residual gases are cooled below 20°, whereby an acid containing more than 85–90% HF is obtained. Silicon tetrafluoride and hydrofluosilicic acid, being practically insoluble in this concentrated liquor, are not condensed in this operation, but are recovered by absorption in water; treatment of the solution with alumina removes free hydrofluoric acid as aluminium fluoride, leaving a concentrated solution of hydrofluosilicic acid. A. R. POWELL.

Production of nitrogen and hydrogen. KALI-IND. A.-G., and C. T. THORSSELL (B.P. 288,154, 30.3.28. Ger., 1.4.27).—Air and then steam are passed over iron or ferrous oxide, and the ferric oxide is reduced by treatment with water-gas, blast-furnace gas, etc. The three reactions are carried out in the same chamber and under such conditions that an external supply of heat is unnecessary. L. A. COLES.

Process for obtaining organic iodine. P. GLOESS (U.S.P. 1,710,255, 23.4.29. Appl., 2.7.27).—Dry seaweed is continuously leached with water in counter-current and rapidly pressed after each leaching. The solution is precipitated with lead or cuprous salts, the precipitate treated with iron filings, and the ferrous salt is converted into the sodium salt of the organic iodine substances. Mucilaginous substance is separated from the residual seaweed. R. BRIGHTMAN.

Apparatus for purification of impure solutions of caustic soda etc. L. CERINI (U.S.P. 1,719,754, 2.7.29. Appl., 7.1.27. Italy, 29.1.26).—See B.P. 265,126; B., 1927, 329.

Treatment of sulphur-bearing minerals. M. F. COOLBAUGH and J. B. READ (U.S.P. 1,719,534, 2.7.29. Appl., 27.6.24).—See Can. P. 264,357; B., 1927, 877.

Exothermic reactions (B.P. 305,534). **Fire-extinguishing compound** (B.P. 315,123).—See I. **Regenerating washing liquids** (B.P. 314,842). **Ammonia** (U.S.P. 1,696,446).—See II.

VIII.—GLASS; CERAMICS.

Dehydration of kaolin in relation to the mullite question. E. KLEVER (Glastechn. Ber., 1929, 7, 85–90).—A special calorimeter is described by means of

which the heats of dissolution of silicates in concentrated hydrofluoric acid were determined. When Zettlitz kaolin was heated in a dry atmosphere for 3 hrs. at different temperatures, the curve showed that the heat of dissolution remained practically constant between 600° and 800°, proving that the loss of water below 600° was not accompanied by decomposition of the kaolin molecule into its components. Metakaolin is the stable compound in the temperature range 600–900°. Between 1050° and 1100° metakaolin decomposes into silica and alumina. At higher temperatures the solubility of the product was so reduced that the heat of dissolution could not be determined. The theory of the sillimanite (or mullite) formation is discussed, and it is concluded that it is not a question of a new 2:3 compound, but that sillimanite, when heated to a sufficiently high temperature, decomposes, giving a final product of approximately mullite composition.

F. SALT.

Lead content of red glazes. A. GRONOVER and E. WOHLNICH (Z. Unters. Lebensm., 1929, 57, 360–363).—Vessels such as cups are filled with boiling 4% acetic acid and heated on the boiling water-bath for 30 min. Larger vessels may be half or two thirds filled and the soluble lead calculated accordingly. The treatment must be carried out repeatedly on the same article and several articles of each consignment should be tested. The colorimetric method of Winkler (cf. Sudendorf and Penndorf, *ibid.*, 1923, 45, 361) and the volumetric chromate method of Beck, Lowe, and Stegmüller (Arb. Kaiserl. Gesundheitsamte, 1910, 33, 240) were found suitable. W. J. BOYD.

Source and prevention of stones in glass. II. INSLEY (Bur. Stand. J. Res., 1929, 2, 1077–1084).—See B., 1929, 355.

PATENTS.

Heat-resistant composition suitable for arc deflectors. W. R. STEELE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,696,614, 25.12.28. Appl., 20.5.26).—A mixture of 60–75% of zirconia, 5–6% of slaked lime, and 20–40% of asbestos or other inorganic fibrous material is steam-hardened, e.g., at 120–150 lb./in.² for 6–12 hrs. R. BRIGHTMAN.

Reclamation of abrasive materials. S. F. WALTON, Assr. to EXOLON Co. (U.S.P. 1,718,264, 25.6.29. Appl., 9.11.27).—Scrap composition containing abrasive or refractory material is heated and suddenly cooled. The binder, thus rendered relatively friable, can be separated after crushing. F. G. CLARKE.

IX.—BUILDING MATERIALS.

Hardening of Portland cement. D. AYDALIAN and E. N. GAPON (Giorn. Chim. Ind. Appl., 1929, 11, 203–206).—The velocity of setting of Portland cement is expressible by the equation $\log(w_0 - w_\infty)/(w_t - w_\infty) = kt$, where w_0 is the initial value of any of its properties, such as the change in the water absorbed or in the sp. gr., w_∞ the value after setting, and w_t the value after the lapse of time t . The value of the constant

k diminishes gradually during the process, owing to increase in the thickness of the colloidal membrane. The setting takes place in three phases: (1) hydration, which proceeds from the second to the third day, (2) diffusion, from the third to the fourteenth day, and (3) crystallisation. The diffusion phase is characterised by a similar expression in which t becomes \sqrt{t} . The constant of the hydration process is directly proportional to the resistance of the cement to compression after 28 days, so that the solidity may be deduced from the velocity of this process.

T. H. POPE.

Density of calcium hydroxide, and its rôle in the shrinking of Portland cement. P. JOYE and P. DEMONT (J. Chim. phys., 1929, 26, 317—318).—The density of pure powdered calcium hydroxide was found to be 2.239, in agreement with the earlier determination of Lamy. There is therefore a decrease in volume in the formation of the hydroxide from the oxide, and this partly accounts for the diminution in volume in the setting of Portland cement.

O. J. WALKER.

Basic principles of concrete making. F. R. McMILLAN (Eng. News-Rec., 1929, 102, 580—583, 625—632, 673—679, 705—709, 748—752).—The ultimate usefulness of concrete depends on the characteristics of the cement, the relative proportions of cement and water, and the degree of completeness of the hydration of the cement. Homogeneity and a proper degree of plasticity ensure proper placing of the mix without segregation. For ordinary size ranges the dimension of the aggregate has little effect on the strength, which varies only in relation to the absorptive coefficient of the type of aggregate. The conditions and duration of curing have much greater influence than other variables, and water-tightness is also influenced by the cement: water ratio. The combination of aggregates to form a satisfactory concrete is a matter for intelligent practice rather than calculation. In large mass construction conditions are usually favourable for the production of high-strength concrete due to slow evaporation and conservation of heat generated, but thin sections should be protected from rapid drying conditions.

C. A. KING.

PATENTS.

Manufacture of cement. J. E. VELZY and W. T. GRONER, Assrs. to SOUTHWESTERN PORTLAND CEMENT Co. (U.S.P. 1,714,060, 21.5.29. Appl., 20.4.28).—Lime slurry is treated with the requisite amount of water, mixed centrifugally with dry waste dust, and delivered to the cement mill.

F. R. ENNOS.

Colouring and stamping small articles of magnesia cement. A. N. P. JACOBS (B.P. 315,126, 14.8.28).—The articles, portions of which may be protected, are dipped successively in solutions containing an oxidising salt, e.g., potassium permanganate or ammonium dichromate, and a cobalt salt.

L. A. COLES.

Preservation of wood. A. OBERLE, Assr. to T. E. SCOFIELD (U.S.P. 1,717,888, 18.6.29. Appl., 19.10.25).—The wood is impregnated with a halogenated hydrocarbon having a sulphur content sufficient to give it toxic properties, and is then treated with a halogen gas under pressure.

A. B. MANNING.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of phosphorus on graphite formation [in cast iron]. O. VON KEIL and R. MITSCHE (Stahl u. Eisen, 1929, 49, 1041—1043).—Tests on a cast iron with 2.8—2.9% P cooled in various ways show that a high phosphorus content tends to restrain the separation of graphite rather than to favour it. Previous contradictory results of other workers are shown to be due to the effect of silicon in displacing the line CE in the ternary iron-carbon-phosphorus diagram towards the region of lower carbon content.

A. R. POWELL.

Comparison between behaviour at the Ac3 point of single-crystal iron and polycrystal iron, both in the strained and unstrained state. H. QUINNEY (Proc. Roy. Soc., 1929, A, 124, 591—603).—Experiments have been made to determine the effect of crystal size on the behaviour of pure iron in its passage through the Ac3 point. The energy per unit mass should be less in a single crystal than in a specimen consisting of a number of crystals owing to the absence of surface energy. An over-strained specimen would contain additional energy. Temperature-time charts taken for single crystals of iron show that there is no difference in the temperature of the Ac3 point, whether the iron, on being heated for the first time, passes from the single-crystal to the polycrystal state at this point or, on being heated again, passes from the β - to the γ -state in the polycrystal conditions. The portion of the curve beyond the critical point is, however, continuously lower in the case of the single crystal than when the specimen changes through the point as a polycrystalline aggregate. If the exact form of the temperature-time curve for no absorption of heat at the Ac3 point were known, the heat absorbed at this point could be determined by measuring the distance by which the observed curve drops below the hypothetical curve. Certain assumptions being made, it is calculated that the difference between the single-crystal and polycrystal heat absorption at the Ac3 point is about 4°. The heat absorption at the Ac3 point is also greatly reduced when a single crystal is heavily over-strained, and a comparison of the heating curves for strained and unstrained polycrystal iron shows that, in the case of the strained material, the ordinary absorption of heat at the Ac3 point is associated with an evolution of heat due to release of strain energy at this point, so that the net result may be an actual evolution of heat.

L. L. BIRCUMSHAW.

Making steel in a basic-lined converter from Indian pig iron at Mysore Iron and Steel Works. P. N. MATHUR (J. Indian Chem. Soc., 1929, 6, 353—355).—Indian pig iron is unsuitable for making steel in an acid converter owing to its high phosphorus content, and the usual practice is to import acid pig iron at high cost of transportation. The author has demonstrated at the Mysore Iron Works the possibility of making steel in a magnesite-lined converter from direct charcoal blast-furnace iron (0.12% P) or cupola metal (0.14—0.15% P), reducing the phosphorus to 0.026% in the best and 0.066% in the worst blows. The advantages of the process are enumerated.

F. L. USHER.

Comparative study of oxidation and deoxidation phenomena in the manufacture of [steel] rails by the Thomas and Martin processes. J. WAGNER (Rev. Mét., 1929, 26, 287—296).—In the Thomas converter, the oxidising agent which removes in turn the silicon, carbon, and phosphorus is not directly the oxygen in the blast, but ferrous oxide dissolved in the molten iron which is continuously regenerated by the blast. For the efficient removal of phosphorus there must be sufficient ferrous oxide continually formed to produce $\text{Fe}_3\text{P}_2\text{O}_8$, which is then decomposed by the lime in the lining of the converter; these reactions are so highly exothermic that a portion of the manganese in the slag is again reduced to metal and there is a diminution of the oxygen content of the molten iron and an enrichment in manganese. If the blast is stopped when the phosphorus content of the metal is 0.04%, the amount of dissolved iron and manganese oxide in the metal is less than that just prior to the oxidation of the phosphorus, namely, 0.07—0.08%, compared with 0.09—0.1%; after deoxidation the metal ready for casting contains about 0.014% O. In the open-hearth Martin process elimination of the phosphorus takes place simultaneously with removal of the other impurities owing to the high ferrous oxide content of the slag and the metal after deoxidation contains 0.010% O. These results indicate that Thomas steel is equally as good as open-hearth steel for the manufacture of rails.

A. R. POWELL.

Well-crystallised blast-furnace slag. F. ANGEL (Z. Krist., 1928, 68, 157—176; Chem. Zentr., 1929, i, 572).—The slag contained melilite (55%), olivine, pyroxene, and glass, the melilite ($a:c = 1:1.260$) consisting of sarkolite, $\text{Ca}_3\text{Al}_2\text{Si}_3\text{O}_{12}$ (70 mol.-%) and åkermanite, $\text{Ca}_2\text{MgSi}_2\text{O}_7$ (30 mol.-%), the pyroxene being closely related to schefferite, and the olivine consisting of Mg_2SiO_4 (65%), Mn_2SiO_4 (30%), and Fe_2SiO_4 (5%).

A. A. ELDRIDGE.

Resistance of nickel-chromium thermo-elements towards molten metals. R. HASE (Z. Metallk., 1929, 21, 200—203).—For measuring the casting temperature of red brass (87% Cu, 6% Sn, and 7% Zn) nickel-nickel-chromium thermocouples with wires of 5 mm. diameter will survive about 100 measurements before the nickel wire is dissolved away, whereas for 67:33 brass the nickel wire dissolves within a few minutes and a bare couple is therefore useless. In both cases the alloy wire is hardly attacked. A satisfactory couple for use with molten brass comprises a tube of nickel-chromium alloy (made by drilling a 10-mm. rod) inside which is a nickel wire insulated by means of a quartz sheath and welded at the lower end in such a way that only the alloy comes into contact with the molten metal.

A. R. POWELL.

Electrical conductivity, corrosion, and age-hardening of cadmium-zinc alloys. G. GRUBE and A. BURKHARDT (Z. Metallk., 1929, 21, 231—231; cf. Jenkins, B., 1926, 831).—From measurements of the electrical conductivity the solid solubility of cadmium in zinc is 3.7% Cd at 263°, 2.1% at 212°, and 0.92% at 142°, and that of zinc in cadmium is 4.3% at 263°, 2.97% at 180°, and 2.08% at 150°. The rate of

corrosion by 1—3*N*-sodium hydroxide solutions is very small in alloys containing 0—17.4% Zn; further addition of zinc causes a rapid increase in the rate of dissolution to a sharp maximum at 20% Zn, which is followed by a smooth fall to a flat minimum at about 50% Zn and then an equally smooth increase to a second sharp maximum at 82% Zn and a much steeper fall to 100% Zn. The alloy with 98% Zn and 2% Cd after annealing for 1 hr. at 300° and quenching in ice-cold water slowly age-hardens at the ordinary temperature, the tensile strength rising from 14.3 to 19.1 kg./mm.² in 9 days and the hardness from 57.2 to 65 in 3 days; the elongation also increases on ageing from 5.7 to 11.6% in 9 days. Ageing at 70° produces a maximum tensile strength of 18.9 kg./mm.², elongation of 13—14%, and hardness of 58 in 12 hrs.

A. R. POWELL.

Corrosion of metals by industrial benzene. S. FUJIO (Bull. Naval Fuel Storehouse Lab., 1928, 2, 65—85).—Samples of steel, copper, and aluminium were immersed in industrial benzene (containing 0.14% of carbon disulphide and 0.10% of " $\text{C}_6\text{H}_5\text{S}$ "). Corrosion, particularly with copper, is attributed to sulphur compounds and water.

CHEMICAL ABSTRACTS.

Trans-crystallisation of aluminium. E. SCHEIL (Z. Metallk., 1929, 21, 121—124).—The formation of large radial crystals in the cross-section of cast aluminium rods is favoured by a high casting temperature, a low mould temperature, and a thick-walled mould. With a casting temperature of 700° the radial crystals disappear with a mould temperature of 500° and the metal has a regularly oriented structure of large crystals with a mould temperature of 600°. When cast from 900° very large radial crystals extending to the core are obtained with mould temperatures below 300°. A theoretical explanation of these facts is put forward.

A. R. POWELL.

Protection of aluminium [against sea-water]. R. CAZAUD (Rev. Mét., 1929, 26, 309—310).—Immersion of aluminium in dilute solutions of sodium sulphite or of sodium carbonate with a chromate, permanganate, vanadate, or tungstate causes the formation of a film on the surface of the metal which protects it from corrosion by sea-water for at least 35 days. Solutions of alkali phosphates, borates, nitrates, silicates, or molybdates produce films which soon break down in sea-water.

A. R. POWELL.

Protection of light alloys by tars [coumarone and by-products of gasworks]. AUBERT and A. PICNOT (Rev. Mét., 1929, 26, 310—312).—Black varnishes with a basis of coal-tar pitch mixed with benzene and phenol, anthracene oil, or pyridine form an effective protection for aluminium alloys against sea-water corrosion, remaining homogeneous and elastic after several months' immersion, and being practically insoluble in petrol and lubricating oils. Similar varnishes made with condensation or polymerisation products of coumarone not only do not dry well, but soon crumble away in sea-water.

A. R. POWELL.

Protection of light metals and alloys against sea-water corrosion by means of electrolytic deposits. J. COURNOT and J. BARY (Rev. Mét., 1929,

26, 312—318).—Prior to electroplating aluminium and its alloys with copper the metal should be sand-blasted, pickled in nitric acid, and thoroughly washed in running water. Plating is effected in a 10% solution of sulphuric acid saturated with copper sulphate at 20° using 1 amp./dm.² at 2—3 volts, or in a solution containing 22.5 g. of copper cyanide, 30 g. of sodium cyanide, and 15 g. of sodium carbonate per litre using 0.6 amp./dm.² at 2—3 volts. Light metals can be satisfactorily plated with cobalt from a solution containing 175 g./litre of cobalt ammonium sulphate using a current density of 2 amp./dm.² provided that a thin coating of copper is first applied. Cadmium may be deposited on aluminium alloys from a bath containing 12 g. of cadmium carbonate and 35 g. of potassium cyanide/litre using 0.8—0.4 amp./dm.², the current density being gradually decreased during the plating. Chromium plating on aluminium alloys adheres strongly only when the alloy is first plated with copper; the best results were obtained with a bath prepared by dissolving chromium hydroxide, freshly precipitated from 200 g. of chrome alum, in a solution of 120 g. of chromic acid in 2 litres of water, using a current density of 10 amp./dm.² at 40—45°. The most resistant to sea-water of the above platings is chromium, and the least resistant cobalt. No satisfactory deposits were obtained by electroplating magnesium with copper.

A. R. POWELL.

Cementation of light and ultra-light alloys as a protection against sea-water corrosion. J. COURNOR and E. PEROR (Rev. Mét., 1929, 26, 318—325).—Annealing of copper-plated aluminium above 540° results in diffusion of the metals into one another at the zone of contact with the formation of CuAl₂ and eutectic; subsequent quenching from about 620° produces a hard surface layer which burnishes to an almost pure copper colour if the plating is not too thin. The resistance of this hard layer to corrosion by sea-water is no better than that of commercial aluminium.

A. R. POWELL.

Viscosity [of aluminium and its alloys]. J. COURNOR (Rev. Mét., 1929, 26, 326—328).—The dimensions of the test-piece affect the results obtained for the viscosity of aluminium and duralumin at high temperatures, the limit of viscosity increasing with the diameter and tending to rise asymptotically to a maximum. Temperature has little effect on this phenomenon, but the nature of the metal is of considerable importance, duralumin showing a much more rapid increase in the viscosity values with increasing diameter than aluminium.

A. R. POWELL.

Preparation of aluminium alloys for microscopic examination. H. CHOUANT (Z. Metallk., 1929, 21, 197—199).—Polishing of sections should be made on fine emery papers coated with paraffin wax, the final polishing being made on a soft fabric soaked in methylated spirits and a little 20% solution of ammonium alum. For the detection of CuAl₂ etching with a 5% alcoholic solution of ferric nitrate is recommended, and for FeAl₃ and FeSi a 20% solution of sulphuric acid at 70°. The nitrate solution etches CuAl₂ brown without affecting any of the other constituents (cf. Dix, B., 1923, 100 A).

A. R. POWELL.

Critical dispersion of laural. H. BOHNER (Z. Metallk., 1929, 21, 160—165; cf. Meissner, B., 1928, 196).—The variations in tensile strength and electrical conductivity during the ageing of laural have been determined in order to ascertain the critical ageing temperature, i.e., the maximum temperature at which, even after prolonged ageing, there is no decrease in the tensile strength. The electrical conductivity rises rapidly during ageing at temperatures above 140°, but the maximum tensile strength of 41 kg./mm.² is reached by ageing at 120° for 200 hrs.; at higher temperatures not only does the tensile strength not reach this maximum but a fall takes place after a certain time of heating owing to coagulation of the dispersed particles. The critical ageing temperature therefore seems to be about 120°, i.e., 40° below that found by Meissner by hardness tests.

A. R. POWELL.

Occurrence of beryllium. M. HOSENFELD (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 21—25).—A list of localities where beryllium minerals are found.

A. R. POWELL.

Decomposition of beryl and the preparation of beryllium salts suitable for electrolysis. K. ILLIG, M. HOSENFELD, and H. FISCHER (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 30—41).—For the large-scale preparation of beryllium compounds from beryl the most satisfactory method comprises heating an intimate mixture of the finely powdered mineral with an equal weight of sodium fluosilicate for several hrs. at 650° and leaching the product with cold water. Extraction of over 90% of the beryllia accompanied by a little iron and only traces of aluminium is thus readily effected. A higher temperature results in fusion of the mass and a lower extraction. Evaporation of the leach liquors gives a crop of large crystals of sodium fluoberyllate, Na₂BeF₄, which are purified by dissolving in water, bubbling air through the liquor to remove iron, and again crystallising. Practically the whole of the beryllium may be recovered by saturating the mother-liquors with sodium fluoride. Complete precipitation of beryllia from fluoberyllate solutions by ammonia is obtained only by using a ten-fold excess; addition of a slight excess of lime, however, effects complete precipitation of the hydroxide in a granular form. The washed precipitate is treated with hydrofluoric acid and the residual calcium fluoride removed by decantation. The solution is evaporated to dryness on the water-bath and the residue heated at 150°, whereby the oxyfluoride, 2BeO.BeF₂, is obtained. Extraction of the residue with an equal weight of hot water dissolves all the oxyfluoride, leaving sodium and calcium fluorides insoluble. Evaporation of the filtered solution affords the pure salt as a snow-white powder, *d* 2.3, m.p. 750°; mixed with barium fluoberyllate the salt forms a readily fusible electrolytic bath for the production of the metal. Methods are also described for the preparation of the double fluorides NaBeF₃, m.p. 520°, BaBeF₄, m.p. 800°, and BaBe₂F₆.

A. R. POWELL.

Preparation of beryllium by electrolysis. K. ILLIG, M. HOSENFELD, and H. FISCHER (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 42—58).—After an historical survey of the work of earlier investigators on the prepara-

tion of beryllium by electrolysis of fused salts an account is given of the Stock-Goldschmidt process (U.S.P. 1,427,919; B., 1922, 822A; cf. also Stock, Press, and Praetorius, B., 1925, 924) and its more recent modifications. The original method gave relatively poor yields of metal owing to the high volatility of the salts used at the working temperature of 1350°. Much better results are now obtained by the use of a bath consisting of a mixture of barium fluoride and beryllium oxyfluoride. The vapours evolved are drawn by suction through water, thence through a wash tower packed with Raschig rings. The solution in the tower after operating the process contains beryllium oxyfluoride whilst barium fluoberyllate remains in suspension; both salts are readily recoverable for further use. The electrolysis is carried out at 1400° in a graphite crucible which serves as anode, a water-cooled iron tube being used as cathode; fresh charges of beryllium oxyfluoride and barium fluoride are added from time to time to replenish the loss by volatilisation and electrolysis, so that the furnace can be operated for 24 hrs. at a time. The metal is obtained in the form of rough rods containing as impurities 0.31% Fe, 0.07% Al, and 0.04% C. The current yield is 75–80% of the theoretical and the recovery of metal about 90%. The metal may be freed from inclusions of salts by fusion under a flux consisting of a mixture of alkali and alkaline-earth chlorides. Attempts to prepare beryllium by electrolysis of the oxide dissolved in fused cryolite, borax, alkali hydroxides, or cyanides failed.

A. R. POWELL.

Influence of the composition and temperature of the electrolyte in the preparation of beryllium by the Stock-Goldschmidt and Siemens & Halske methods. H. FISCHER (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 59–73).—Beryllium oxyfluoride is not a conductor of electricity, but becomes a conductor on addition of barium fluoride, the current yield rising linearly to a maximum of 80% with 45% of barium fluoride in the mixture and falling again with more than 50% of the barium salt. The current yield is reduced rapidly by addition of sodium fluoride to the bath, alkalis and alkali chlorides increase the loss by volatilisation, and aluminium, silicon, and iron compounds contaminate the deposited metal. The electrolysis must be carried out in an oxidising atmosphere to prevent carbon from the anode entering the metal. The best results are obtained at 1400° with a current density of 100–400 amp./cm.² of cathode area.

A. R. POWELL.

Production of electrolytic deposits of beryllium from molten electrolytes. H. FISCHER (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 83–87).—Galvanic deposits of beryllium on copper, nickel, iron, aluminium, and light-metal alloys can be obtained from fused baths consisting of a mixture of equal parts of sodium fluoride and beryllium oxyfluoride. Copper is best plated at 750° with 10 amp./dm.², iron and nickel at 900°, and light metals at 550–580°. Subsequent annealing of the plated article results in the formation of a beryllium alloy layer which is usually very hard and fairly resistant to corrosion.

A. R. POWELL.

Formation of beryllium alloys by direct electrolysis. K. ILLIG and H. FISCHER (Wiss. Veröff. Siemens-

Konz., 1929, 8, [1], 88–93).—Alloys with 0.1–40% Al in beryllium may be obtained by addition of the requisite quantity of cryolite to the beryllium bath during the Siemens & Halske electrolytic process (cf. *supra*). Copper alloys with a low beryllium content may be prepared by using molten copper as the cathode in the above process and beryllium alloys with a small copper content by adding copper fluoride to the electrolyte. Nickel and copper alloys can also be obtained by adding copper or nickel to the bath so that it sinks to the bottom and behaves as the anode, being dissolved by the liberated fluorine and deposited on the cathode with the beryllium.

A. R. POWELL.

Alloys of beryllium with iron. W. KROLL (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 220–235).—The solid solubility of beryllium in iron falls from about 4% at 1100° to about 1% at the ordinary temperature. The maximum age-hardening effect is obtained with the alloy containing 3.9% Be, but the structure is very coarsely crystalline and the alloy has no technical value. Addition of nickel to beryllium-iron alloys decreases the solubility of beryllium but improves the structure considerably, although the hardness decreases with increasing nickel from 4 to 23%. Chromium, on the other hand, increases the hardness but does not refine the structure. A satisfactory structure with a high hardness value is obtained with steels of the V2A type containing 7% Ni, 20% Cr, and 1% Be which after ageing have a hardness of 500. If the chromium is reduced to 12% this alloy attains a hardness of 650 after ageing, but the impact strength and ductility are very small. The resistance of low-carbon (0.2%) beryllium steels containing nickel and chromium is about equal to that of the usual non-rusting steels, but increasing carbon content rapidly reduces their incorrodibility. The age-hardened alloy with 12% Cr, 5% Ni, and 1% Be is suitable for high-speed cutting tools, that with 20% Cr, 7% Ni, and 1% Be as a strong rustless steel, and that with 36% Ni and 1% Be as a substitute for invar, having higher tensile strength and resistance to atmospheric corrosion.

A. R. POWELL.

Technical properties and improvement of beryllium-copper alloys. G. MASING and O. DAHL (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 101–125).—Beryllium-copper alloys when quenched from above 800° undergo age-hardening at 250–400° if the beryllium exceeds 1% (cf. Masing and Dahl, B., 1928, 160). The maximum hardness of 510 is obtained in 1 hr. with the alloy containing 4% Be by ageing at 350°; higher or lower temperatures or more prolonged heating give lower hardness values, but once hardened the alloys remain unchanged at 250° for more than 100 days. The electrical conductivity of the 2.5% Be alloy increases in a smooth curve concave to the time axis over a period of 25 hrs. at 350°, whereas the hardness reaches a maximum in 1½ hrs. and then commences slowly to decrease; after 3 hrs. at 350° the alloy has a tensile strength of 135 kg./mm.², a yield point of 125 kg./mm.², and an elongation of 0.8%, the corresponding values for the quenched alloy being 48.9 kg./mm.², 15.7 kg./mm.², and 52%. Similar results may be obtained by the heat-treatment of sand- and chill-castings of alloys containing

2–3% Be. The corrosion-resisting power of beryllium-copper alloys is greater than that of 12–14% tin bronzes and about equal to that of 6–8% aluminium bronzes; after age-hardening the resistance to attack by hydrochloric acid is appreciably reduced. A. R. POWELL.

Ternary alloys containing beryllium and a copper basis. G. MASING and O. DAHL (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 202–210).—Addition of beryllium to α -solid solution alloys of copper with tin, zinc, or aluminium causes them to undergo age-hardening after quenching from 700–800°. The presence of a third metal reduces the solid solubility of beryllium in copper so that smaller amounts of beryllium are necessary to produce the maximum hardening than are required in the case of copper. With α -brasses with a high zinc content a small addition of beryllium increases the hardness of the quenched alloy considerably, and subsequent ageing causes only a relatively small increase. Addition of less than 1% Al to the 2–2.5% Be copper alloys accelerates the age-hardening without intensifying the action. A. R. POWELL.

Influence of small additions of phosphorus on the age-hardening of beryllium-copper alloys. G. MASING and O. DAHL (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 197–201).—Addition of phosphorus to beryllium-copper alloys accelerates the age-hardening process and causes it to take place at lower temperatures. With 0.1% P the alloy with 1.5% Be attains its maximum hardness of 250 at 250° in 24 hrs., whereas without phosphorus more than 50 hrs. at 350° are required to produce a hardness of 142. The maximum hardness obtainable with alloys containing more than 2% Be is slightly reduced by addition of phosphorus. A. R. POWELL.

Theory of the age-hardening process based on researches on beryllium-copper alloys. G. MASING (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 187–196).—All the phenomena associated with the age-hardening of beryllium-copper alloys can be explained on the assumption that a second phase is precipitated in a highly dispersed form by the decomposition of a super-saturated solid solution. A. R. POWELL.

Changes in the modulus of elasticity during ageing of beryllium-copper alloys. G. MASING and C. HAASE (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 142–148).—The torsion modulus of beryllium-copper alloys increases during ageing in a similar manner to the electrical conductivity. This is in accordance with the theory that the age-hardening of these alloys is due to precipitation of the γ -phase in a highly dispersed state. A. R. POWELL.

Changes in electrical conductivity and volume of beryllium-copper alloys during ageing. G. MASING and O. DAHL (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 126–141).—Ageing of quenched beryllium alloys results in considerable changes in the conductivity; with alloys containing less than 3% Be there is an initial fall followed by a more or less sharp rise. At 350° the conductivity is doubled by sufficiently prolonged tempering. Ageing also causes a progressive contraction in the volume amounting to a maximum of

0.6% in the case of the 2.5% Be alloy tempered at 250° for 81 hrs. A. R. POWELL.

Beryllium-nickel alloys. G. MASING and O. DAHL (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 211–219).—Beryllium dissolves in molten nickel with the evolution of much heat; the system contains one compound NiBe which forms a eutectic with nickel, m.p. 1155°, containing 5.2% Be. The saturated solid solution of beryllium in nickel contains 2.8% Be at the eutectic temperature and 1.4% at 800°. Alloys containing more than 1.5% Be, therefore, undergo age-hardening after quenching from 1100°, a maximum hardness of 550° being obtained with the 3% alloy in 1 hr. at 600°, but hardness values of over 600 can be obtained with the 2.5 and 3.0% Be alloys by prolonged annealing at 450°. These alloys after heat treatment have a tensile strength of 132 kg./mm.² with an elastic limit of 110 kg./mm.²; they are almost as resistant as pure nickel to corrosion by dilute nitric and hydrochloric acids and are quite unattacked by sea-water. A. R. POWELL.

Aluminium alloys containing beryllium. Silicon-beryllium alloys. G. MASING and O. DAHL (Wiss. Veröff. Siemens-Konz., 1929, 8, [1], 248–256).—The age-hardening properties of copper-aluminium alloys are not improved by addition of beryllium, and beryllium cannot be used as a substitute for magnesium in alloys of the type of aludur and aldreyl. Silicon and beryllium form a eutectic with 39% Be, m.p. 1090°, but no compounds or solid solutions. All the alloys are extremely hard and very brittle. A. R. POWELL.

Adherence of varnishes to light alloys. AUBERT and DIXMIER.—See XIII. Tin foil for packing cheese. ELTEN.—See XIX.

PATENTS.

Annealing furnace. W. M. HEPBURN and E. W. WEAVER, Assrs. to SURFACE COMBUSTION CO., INC. (U.S.P. 1,716,956, 11.6.29. Appl., 8.2.28).—A continuous annealing furnace is divided into three zones in the direction of travel of the goods, viz., (a) a preheating zone, (b) a main heating zone, (c) a final heating zone; b and c are connected by a restricted passage which will pass the goods but not much of the gases. The roof of b is raised at the end nearest a and a burner is situated in the raised part, sending the gases along the roof towards c and back along both b and a countercurrent to the goods, the final gas outlet being in a. Chamber c is heated separately. B. M. VENABLES.

Furnaces for annealing stampings. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 308,676, 10.11.28. Ger., 26.3.28).—The furnace comprises a closed rectangular annealing chamber with a liftable roof on the longitudinal sides of which are arranged a series of water-cooled tubes. The roof is covered by a hood which dips into sealing troughs, and the space between the hood and the roof is connected by pipes to vents in the hearth of the furnace. When the annealing is completed the roof is raised slightly inside the hood so that the hot gases rise into the hood, where they are cooled by the water-tubes and circulate back into the furnace through the vents in the hearth. Rapid cooling of the furnace is

thus effected without its contents coming into contact with oxidising gases. A. R. POWELL.

Determining the content of foreign substances in iron and steel. B. D. ENLUND, Assr. to H. ENLUND (U.S.P. 1,718,687, 25.6.29. Appl., 16.8.27).—The specific resistance of hardened and annealed test pieces of the material is measured and compared with known factors. H. ROYAL-DAWSON.

Manufacture of ferro-aluminium alloy. R. P. and H. A. DE FRIES, Assrs. to LUDLUM STEEL CO. (U.S.P. 1,718,685, 25.6.29. Appl., 25.4.27).—Aluminium in small quantities is added to molten iron to deoxidise it, followed by a further addition to bring the aluminium content up to 60% of the mixture.

H. ROYAL-DAWSON.

Ferrous molybdenum alloys. CLIMAX MOLYBDENUM CO., Assces. of A. KISSOCK (B.P. 314,032, 19.6.28. U.S., 1.11.27).—Molten pig iron containing molybdenum is tapped directly from the blast furnace into a charge of molten iron or steel in any type of steel furnace. Alternatively, the pig iron containing molybdenum may be melted together with the usual charge in a cupola to produce cast iron containing molybdenum.

A. R. POWELL.

Coating metals [e.g., iron, aluminium, zinc, etc. with manganese dioxide]. O. SPRENGER PATENTVERWERTUNG JIROTKA M.B.H., and B. JIROTKA (B.P. 314,769, 2.3.28).—The metal is pickled in a bath containing 5% of permanganate, 2% of hydrofluoric acid or acetic acid, and 0–0.5% of copper sulphate or ferric chloride. For aluminium or zinc the acid is replaced by 2% of hydrogen peroxide or 5% of bichromate. The coatings obtained are brown to black in colour and highly resistant to corrosion.

A. R. POWELL.

Production of non-corrosive and heat-resisting surfaces on iron. J. Y. JOHNSON. From I. G. FARRERIND. A.-G. (B.P. 314,314, 19.3.28).—The iron article is cleaned by sandblasting, embedded in a mass of powdered aluminium-chromium alloy containing 15% Al, and heated in an atmosphere of hydrogen at 1000°.

A. R. POWELL.

Pickling of iron and steel. J. H. GRAVELL (B.P. 287,911, 28.3.28. U.S., 29.3.27).—A mixture of a foaming agent (e.g., cellulose pulp waste-liquor) and a thioamide (e.g., thiocarbamide) is added to the ordinary pickling bath so as to produce on its surface a stable foam which prevents the formation of an acid spray above the bath.

A. R. POWELL.

Electrolytic recovery of metals [nickel]. S. C. SMITH (B.P. 314,579, 30.3.28).—Nickel may be recovered from sulphate solutions by electrolysis, using an aluminium cathode and lead anodes, provided that the p_H of the solution is kept between 3.5 and 6.5. This is effected by continuous agitation, accompanied by the gradual addition of ammonia, the temperature being kept above 50° to avoid crystallisation of nickel ammonium sulphate. The spent electrolyte is evaporated to recover the remaining nickel as the double salt, which is insoluble in saturated ammonium sulphate solution, and subsequently further evaporated to recover ammonium sulphate.

A. R. POWELL.

Refining of chromium ores. I. G. FARRERIND. A.-G. (B.P. 288,973, 3.4.28. Ger., 16.4.27).—Low-grade chromite ores are heated under pressure with solutions of, or are fused in a neutral atmosphere with, sodium hydroxide or carbonate and the washed product is treated with a dilute mineral acid. A. R. POWELL.

Production of zinc from oxidised zinc compounds. METALLGES. A.-G. (B.P. 298,636, 6.10.28. Ger., 13.10.27).—Oxidised zinc ores are mixed with powdered iron, formed by the gaseous reduction of ferrosferic oxide, and the mixture is heated at 850–1000° under reduced pressure whereby zinc is distilled off relatively free from lead and other impurities. By regulating the amount of iron used so that the residue in the retort consists of ferrosferic oxide, this oxide may be recovered by magnetic separation and used again in the process.

A. R. POWELL.

Purification of alkali or alkaline-earth metals and production of alloys. BRIT. THOMSON-HOUSTON CO., LTD., Assces. of J. J. CONLIN (B.P. 288,333, 5.4.28. U.S., 6.4.27).—Metals, e.g., calcium, magnesium, are purified by placing the metal in a container having a safety opening at the bottom, and immersing the container in a molten salt (e.g., calcium chloride) which does not react with and has a higher sp. gr. than the metals. The salt should not be volatile at a temperature which causes separation of the impurities in the metals.

C. A. KING.

Surface treatment of light metals. E. C. BURDICK, Assr. to DOW CHEM. CO. (U.S.P. 1,709,894, 23.4.29. Appl., 11.1.27).—Magnesium or magnesium alloy is treated with a boiling solution of sodium or magnesium dihydrogen phosphate.

R. BRIGHTMAN.

Melting finely-divided materials and agglomeration of blast-furnace dust. A. DAWANS (B.P. 297,813, 15.6.28. Belg., 30.9.27. Addn. to B.P. 294,639).—To the apparatus described in the original patent (cf. B., 1929, 648) is added a screen of refractory material which confines the very hot melting zone to the downstream extremity of the hearth, and directs the gases leaving that zone towards the unmelted material; the temperature in the preheating or roasting zone is thus reduced to such a point that mechanical stirrers may be used. A supplementary supply of air may be admitted above the screen.

B. M. VENABLES.

Froth flotation [agents]. R. J. LEMMON (B.P. 314,822, 3, and 12.3.28).—Oxidised ores, especially copper ores, are floated in a circuit to which salts or esters of isothiocyanic acid (thiocarbimide) or its derivatives are added. *iso*-Di- and *-per*-thiocyanic acids and their derivatives and the compounds obtained by the action of alkalis on cyanogen sulphides are specifically claimed.

A. R. POWELL.

Flotation agents. T. H. DONAHUE and F. F. FRICK (U.S.P. 1,711,087, 30.4.29. Appl., 3.5.26).—Aqueous xanthate solutions are treated with excess of chlorine to give white emulsions claimed as flotation agents.

R. BRIGHTMAN.

Increasing the output of mechanical roasting furnaces and the like. A. CARLSON (B.P. 314,585,

31.3.28).—The fuel stream is admitted to the furnace tangentially and is directed over the material in the furnace. Products of combustion leave the furnace centrally and pass through a drum through which the charge is admitted to the furnace. Mechanical means of producing intimate contact between the charge and the hot gases are provided. C. A. KING.

Treatment of ores with chloride solutions. N. C. CHRISTENSEN (U.S.P. 1,696,471, 25.12.28. Appl., 25.5.26).—In the treatment of ores with acid chloride solutions (cf. U.S.P. 1,539,711—3; B., 1925, 598), the dissolved metals are precipitated with a suitable calcium compound, e.g., calcium carbonate or hydroxide for aluminium, iron, zinc, or manganese, or calcium hydrosulphide (cf. U.S.P. 1,549,062 and 1,572,268; B., 1925, 812; 1926, 330) for iron, zinc, or manganese, the calcium chloride solution is treated with sulphuric acid, and the hydrochloric acid brine thus produced is used in the treatment of further ore, either with or without previous filtration from the calcium sulphate. R. BRIGHTMAN.

Manufacture of thin bodies made of alloys of metals or metalloids. C. MÜLLER (B.P. 313,891, 15.12.27).—Thin wire or foil of one metal is coated with a thin layer of another metal by electrodeposition, thermal dissociation of a compound of the second metal, or by any other suitable means, and the composite wire or foil is heated at such a temperature under non-oxidising conditions that complete diffusion of the constituents to form a homogeneous alloy takes place. The resulting alloy can be subsequently reduced in thickness by rolling or drawing. A. R. POWELL.

Fluxes for welding. K. GEISEL (B.P. 315,010, 8.3.28).—A mixture of powdered zinc chloride (90), ammonium bromide (8), and sodium fluoride (2 pts.), having m.p. about 190°, is employed for welding aluminium or duralumin to such metals as iron, brass, or copper. H. ROYAL-DAWSON.

Proofing metal, especially iron and steel, against rust. W. H. COLE (U.S.P. 1,719,463, 2.7.29. Appl., 28.2.27. U.K., 1.1.27).—See B.P. 289,906; B., 1928, 488.

Process of refining magnesium and its alloys. G. PISTOR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,720,436, 9.7.29. Appl., 8.11.27. Ger., 13.11.26).—See B.P. 280,530; B., 1928, 337.

Protection of surface of baths of easily-oxidisable metals such as magnesium. G. MICHEL (U.S.P. 1,720,286, 9.7.29. Appl., 2.8.26. Fr., 18.8.25).—See B.P. 257,221; B., 1927, 528.

Melting and refining non-ferrous metals. D. CUSHING, Assr. to BARRETT Co. (Reissue 17,347 of U.S.P. 1,556,591, 2.7.29. Appl., 12.10.27).—See B., 1925, 996.

Treating antimonial ores. H. GOLDMANN (U.S.P. 1,719,657, 2.7.29. Appl., 11.2.27. Ger., 29.3.26).—See B.P. 268,278; B., 1927, 448.

[Apparatus for] manufacture of cast [metal] articles. W. A. LOTH (B.P. 294,239, 4.7.28. Fr., 21.7.27).

Degreasing metal and like articles [by means of solvents]. IMPERIAL CHEM. INDUSTRIES, LTD., and B. P. CRAWSHAW (B.P. 314,912, 4.2.28).

Cleaning of metal castings [by water under pressure]. J. J. V. ARMSTRONG. From BADISCHEN MASCHINENFABR. U. EISENGIESSEREI (B.P. 315,446, 12.3.28).

Colloidal mixtures (B.P. 286,316).—See I. **Lead oxide** (B.P. 315,128).—See VII. **Anti-corrosive compositions** (B.P. 314,673).—See XIII.

XI.—ELECTROTECHNICS.

Burn-out of incandescence lamps. Phenomena influencing the life of gas-filled lamps. G. R. FONDA (Gen. Elec. Rev., 1929, 32, 206—212).—The tensile strength, crystalline changes, disintegration of the filament by chemical changes, and the influence of ionisation of the gas were studied. CHEMICAL ABSTRACTS.

Electrolytic production of beryllium. ILLIG and others; FISCHER; ILLIG and FISCHER. **Protecting light metals and alloys.** COURNOT and BARY.—See X.

PATENTS.

[Rotary] electric furnaces. BIRMINGHAM ELECTRIC FURNACES, LTD., and A. G. LOBLEY (B.P. 314,775, 28.3.28).—A rotary electric furnace comprises a furnace proper and a work-preheating conveyor substantially longer than the heating chamber and having an end part only contained within the chamber. The conveyor comprises two compartments separated by a heat-conducting partition or cylinder and having work-transfer communication with one another, and is provided at the end remote from the part contained in the chamber with charging and discharging openings, so that during rotation of the conveyor with the furnace in-going and out-going work travel in opposite directions. J. S. G. THOMAS.

Light-sensitive cell. C. RUZICKA (B.P. 314,838, 3.4.28).—A powdered mixture of at least two of the elements selenium, tellurium, phosphorus, uranium, thorium, boron, arsenic, antimony, chromium, silicon, cobalt, bismuth, manganese, and tantalum, together with, if desired, 0.5—1% of silver, copper, or other good electrically-conducting metal, is compressed, heated *in vacuo* or in an inert or reducing atmosphere containing nitrogen, argon, helium, krypton, or neon, and the coherent mass produced is mounted in a cell provided with electrical conductors. J. S. G. THOMAS.

[Gas-filled] electric discharge tube [for rectifying multiphase alternating current]. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 314,925, 2.4.28).—The gas-filling contains mercury vapour, and liquid mercury is contained in a separate reservoir. J. S. G. THOMAS.

X-Ray tube. A. BOUWERS and W. H. VAN DE SANDE BAKHUYZEN, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,718,849, 25.6.29. Appl., 9.2.24. Holl., 28.9.23).—The envelope is filled with gas containing at least one of the two elements of smallest atomic number, at a pressure greater than 0.0006 mm. J. S. G. THOMAS.

Improving the tensile strength of fabric materials [insulating tape]. P. B. COCHRAN, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. Co. (U.S.P. 1,699,779,

22.1.29. Appl., 15.7.27).—The tape is exposed to ozonised air at 110–130°. C. HOLLINS.

Illumination of light-sensitive cells. J. NEALE (B.P. 315,079, 19. and 26.6.28).

[Ultra-violet] irradiation of substances [liquids or gases]. QUARZLAMPEN-GES.M.B.H. (B.P. 295,023, 27.7.28. Ger., 4.8.27).

Liquid polymerisation products (B.P. 294,100).—See II. **Heat-resistant composition for arc deflectors** (U.S.P. 1,696,614).—See VIII. **Recovery of nickel** (B.P. 314,579).—See X.

XII.—FATS; OILS; WAXES.

Detection of rancidity of fat in whole seeds and fruits. A. NIETHAMMER (Z. Unters. Lebensm., 1929, 57, 358–360; cf. Pritzker and Jungkunz, B., 1926, 1020).—The fat of seeds alters during storage. That from fresh seeds and fruits gives no Kreis or Fellenberg reaction whereas these reactions are given by the fat from seeds stored for some years and incapable of germination. W. J. BOYD.

Saponifier-emulsifier for fats. I. S. KIZBER (J. Chem. Ind. Moscow, 1928, 5, 1176–1181).—The residue (100 g.) from the distillation of stearic acid is treated with a mixture of fuming sulphuric acid (50 g.) and sulphuric acid (50 g.), added in small portions during 20–30 min., the temperature being maintained at 38–40°, and the mixture is kept overnight. The emulsifier is not so efficient as an equal quantity of Petrov's catalyst. CHEMICAL ABSTRACTS.

Physico-chemical studies on the mechanism of the drying of linseed oil. I. Changes in density of films. G. L. CLARK and H. L. TSCHENTKE (Ind. Eng. Chem., 1929, 21, 621–627).—Linseed oil films produced under regulated conditions and submitted to a standard baking to permit solidification and preliminary drying were exposed to natural and artificial weathering, and the changes in density of the films (usually increases) were accurately measured at stated intervals (floatation method). The effects of added driers, inhibitors, metallic soaps, and superposed films are considered. Extreme sunning and weathering produced the greatest increases, extending over considerable periods, ranging from 2–4% after 32 hrs. to 25% after 226 days; under sunning, the greatest changes occur in the upper layer, whereas under ultra-violet ageing smaller changes occur uniformly through the film. Thin films showed greater increases in density than thick films; the density data indicate the mechanism of the failure of films as a result of unequal contractions, assisted by the non-homogeneity due to the crystallisation of metallic soaps. E. LEWKOWITSCH.

Saponification value of fats with special reference to barley fat. K. TÄUFEL and M. RUSCH (Z. Unters. Lebensm., 1929, 57, 345–348).—The saponification value of barley fat increases with increasing time of saponification and with increasing excess of alkali. Dilution of the alcoholic alkali with xylene raises the b.p. of the mixture, but lowers the saponification value owing to the decrease in concentration of alkali. The increase in saponification value is due partly to rosin-

like substances which enter into side reactions, an partly to conversion of unsaturated acids into acids of lower mol. wt. The Reichert-Meißl and Polenske values also increase with increasing time of saponification (cf. A., 1929, 961). W. J. BOYD.

"Liquid-phase" rule in emulsions with sulphonated oils. W. SCHINDLER (Kolloid-Z., 1929, 48, 254–270).—In the emulsification of a constant amount of a mineral oil by increasing amounts of sulphonated train oil there is an optimum volume relation between the two liquids. Small quantities of emulsifier are insufficient to disperse the mineral oil, whilst large quantities have a coagulating effect. A similar optimum volume relation holds between the mixture of oils and water, its value depending on the kind of train oil used and on its proportion in the mixture. Sulphonated castor oil is a poorer emulsifier than train oil, but is improved by the addition of oleic acid. E. S. HEDGES.

Bentonite. WOODMAN and TAYLOR.—See XVI. **Test for vitamin-A in fatty foods.** ANDERSEN and NIGHTINGALE. **Testing of seeds etc.** NIETHAMMER.—See XIX.

PATENTS.

Manufacture of bleached pure wool fat. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 314,587, 31.3.28).—Crude wool fat emulsions are bleached and purified in one operation by heating at above 100° (about 160°) under pressure in the presence of a reducing agent which exerts a bleaching action (e.g., sodium bisulphite, hyposulphite, sulphurous acid, etc.). E. LEWKOWITSCH.

Manufacture of refined products from wool fat. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 314,586, 31.3.28).—The crude wool oil (low-melting fraction) derived by solvent-extraction from wool fat is saponified, and the acids are separated from the unsaponifiable compounds (including wool oil alcohols). E. LEWKOWITSCH.

Refining of carnauba wax and candelilla wax. A. RIEBECK'SCHE MONTANWERKE A.-G. (B.P. 299,357, 5.6.28. Ger., 24.10.27).—The crude wax is dissolved in ethyl acetate, methyl alcohol, glycol, butanone, etc. and treated with an absorbing agent, especially decolorising carbon activated with zinc chloride. E. LEWKOWITSCH.

Decolorisation of vegetable oils. H. E. BIERCE (U.S.P. 1,696,338, 25.12.28. Appl., 29.6.25).—The oils are rendered slightly alkaline and treated with absorbent earth. R. BRIGHTMAN.

Production and use of stable sulpho-acid products of high mol. wt. ORANIENBURGER CHEM. FABR. A.-G., Assees. of CHEM. FABR. MILCH A.-G. (B.P. 308,280, 20.12.27. Ger., 1.4.27. Addn. to B.P. 288,126; B., 1929, 529).—The method of producing stable, emulsifiable sulpho-acids of high mol. wt. previously described (*loc. cit.*) is modified by the use of mixtures of sulphonating and dehydrating and/or condensing agents. E. LEWKOWITSCH.

Foam-producing composition. E. M. A. STHAMER (U.S.P. 1,696,507, 25.12.28. Appl., 29.3.28. Ger., 4.4.27).—Mixtures of, e.g., 83% of lactose and 17% of saponin have foam-producing properties superior to those of saponin itself. R. BRIGHTMAN.

Production of soap. A. WELTER (U.S.P., 1,719,349, 2.7.29. Appl., 28.6.26. Ger., 3.7.25).—See B.P. 254,755; B., 1927, 946.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Rapid determination of the permeability and adherence of varnishes used for the protection of light alloys. AUBERT and G. DIXMER (Rev. Mét., 1929, 26, 307—308).—The time taken for a current of 0.05 amp. to pass between a varnished and an unvarnished cylindrical electrode 1 cm. in diam. and 5 cm. apart, immersed in sea-water, when an *E.M.F.* of 2 volts is set up between them is a measure of the adherence and permeability of a varnish, and hence of its power to protect the metal from corrosion. Nitrocellulose varnishes soon break down in this test, whereas those with a basis of boiled oil or tar require 60—100 hrs.

A. R. POWELL.

Constitution of Bakelite-C. A. E. BLUMFELDT (Chem.-Ztg., 1929, 53, 493—494).—Evidence is adduced in support of the theory that Bakelite-C is formed by the condensation of two more or less long-polymerised *ortho*-substituted hydroxydiphenylmethanes with the elimination of water, and is therefore a highly polymerised aromatic hydrocarbon; Novolacks are, on the other hand, polymerisation products of the *para*-substituted compounds, and contain oxygen or hydroxyl groups.

A. R. POWELL.

1:4-Dioxan. REID and HOFMANN.—See III. Protection of light alloys. AUBERT and PIGNOT.—See X.

PATENTS.

Preparation of anti-corrosive compositions. M. H. LANG (B.P. 314,673, 29.6.28).—A paste consisting of a mixture of desulphated and aerated pulverised slag, a pigment and linseed oil, masticated rubber in a suitable solvent, copal gum varnish, nitrocellulose varnish, boiled linseed oil, and driers.

C. A. KING.

Water-paint. A. P. GOODELL and G. W. TARR (U.S.P. 1,700,404, 29.1.29. Appl., 11.3.26).—Opacity and covering power of water-paints are improved by addition of emulsified wax (paper-makers' wax) or Japan wax (1 oz. to each lb. of pigment).

C. HOLLINS.

[Marking] ink. BRIT. CELANESE, LTD. (B.P. 296,759, 5.9.28. U.S., 7.9.27).—A marking ink for fabrics, especially those containing cellulose derivatives, which will withstand dyeing, bleaching, scouring, etc., is made by grinding a pigment (lead chromate) and a drying or semi-drying oil in a solution of a cellulose ester (*e.g.*, nitrocellulose in ethyl acetate).

E. LEWKOWITSCH.

Manufacture of [titaniferous] lithopone. W. J. O'BRIEN (U.S.P. 1,709,999, 23.4.29. Appl., 9.5.28).—Sufficient zinc oxide is added to a solution of titanium sulphate in sulphuric acid to neutralise the sulphuric acid resulting from hydrolysis of the sulphate, the product is treated with barium sulphide to precipitate the zinc completely, and the mass pressed, dried, muffled, and quenched in water.

R. BRIGHTMAN.

Varnishes etc. [Artificial leather.] NOBEL INDUSTRIES, LTD., Assees. of E. I. DU PONT DE NEMOURS & Co. (B.P. 314,571, 29.3.28).—Carboxylic esters of aryloxy-

ethyl alcohols, particularly β -phenoxyethyl phthalate, β -*o*- or -*m*-tolylxyethyl phthalate or laurate, etc., are used in place of castor oil as softeners for cellulose ester varnishes, especially for cellulose nitrate solutions used in manufacture of leather-cloth.

C. HOLLINS.

Extraction of rosin and turpentine from wood. A. A. SHIMER, Assr. to HERCULES POWDER Co. (U.S.P. 1,696,696, 25.12.28. Appl., 30.12.26).—The waste wood chippings are passed through a crusher which converts them into flattened thin discs without tearing or destroying the fibre before feeding to the extractor for steam and gasoline treatment in the known manner. A 10—20% higher recovery of turpentine is claimed.

R. BRIGHTMAN.

Manufacture of products from resins. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 314,588, 31.3.28).—Resins (*e.g.*, colophony or rosin-phenol condensation products) are heated with a dehydrating agent (*e.g.*, 2% of zinc chloride) in the presence of an aralkyl halide (*e.g.*, benzyl or xylyl chloride). According to the temperature chosen (110—180°) the products can be more or less readily sulphonated to form water-soluble derivatives.

E. LEWKOWITSCH.

Manufacture of condensation products of urea and formaldehyde. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 314,908, 5.1.28).—Clear, hard condensation products are formed by fusing urea or thiourea, or both, and a solid polymeride of formaldehyde in the presence of small amounts of mild alkaline agents, such as sodium acetate, borax, etc., and/or organic substances, *e.g.*, formamide, and hardening the opaque product, after casting or disintegration, by hot-pressing. The product is suitable for cementing veneer or glass plates and celluloid etc.

E. LEWKOWITSCH.

Manufacture of artificial resins etc. BRIT. CYANIDES Co., LTD., and E. C. ROSSITER (B.P. 314,358, 23.12.27, 19.4. and 21.7.28).—Dicyanodiamide is condensed with aqueous formaldehyde to give a thermo-hardening resin, which may be incorporated with a resin from formaldehyde and carbamide and/or thiocarbamide. The proportion of formaldehyde may be varied, and further amounts of formaldehyde may be condensed with the products of the first condensation. The resins may be combined with acidic compounds such as formic acid (colourless product, swelling and dissolving in water), butyric acid (non-hardening, soluble resin), oleic, linoleic, ricinoleic acids (for plasticisers; miscible with rubber), oxalic acid (hard, soluble resin).

C. HOLLINS.

Manufacture of phenolic condensation products. O. A. CHERRY and F. KURATH, Assrs. to ECONOMY FUSE AND MANUF. Co. (U.S.P. 1,710,722, 30.4.29. Appl., 21.4.26).—Excess of phenol is condensed with formaldehyde in presence of furfuramide, the resin freed from water and hardened by heat.

R. BRIGHTMAN.

Resinous material and its manufacture. P. KNAPP (U.S.P. 1,696,490, 25.12.28. Appl., 18.5.21. Renewed 18.2.26).—Dilute solutions of furfuraldehyde, such as those obtained in the acid distillation of pentoses or pentosans, are treated at 60° with amines in presence of a condensing agent, *e.g.*, acetic or mineral acid, calcium, iron, or zinc chloride. The resin obtained, *e.g.*, the

product, m.p. 150°, from aniline and furfuraldehyde, may be further condensed or polymerised by heat, *e.g.*, at 180–220° under pressure. R. BRIGHTMAN.

Pyroxylin composition. R. CALVERT, ASSR. to VAN SCHAAK BROS. CHEM. WORKS, INC. (U.S.P. 1,702,151, 12.2.29. Appl., 26.1.28).— $\alpha\delta$ -Dimethylamyl acetate, b.p. 146°/748 mm., is used as pyroxylin solvent in lacquers for brushing and spraying. Examples of such compositions are given. R. BRIGHTMAN.

Esters of hexyl [$\alpha\delta$ -dimethylamyl] alcohol. H. D. YOUNG, ASSR. to VAN SCHAAK BROS. CHEM. WORKS, INC. (U.S.P. 1,702,188, 12.2.29. Appl., 9.1.28).— $\alpha\delta$ -Dimethylamyl esters of aromatic dibasic acids are claimed as pyroxylin solvents. The *phthalate*, b.p. 170°/4 mm., is obtained by esterification in benzene in presence of sulphuric acid. R. BRIGHTMAN.

Organic moulding composition. S. M. HULL, ASSR. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,711,025, 30.4.29. Appl., 29.1.26).—A dry protein (*e.g.*, casein) is condensed with furfuraldehyde after mixing with a filler and a waterproofing agent. R. BRIGHTMAN.

Manufacture of press masses from phenol-formaldehyde resins. F. SEEBACH, ASSR. to BAKELITE GES.M.B.H. (U.S.P. 1,720,062, 9.7.29. Appl., 6.2.26. Ger., 17.2.25).—See G.P. 431,514; B., 1926, 869.

Ethers of polyhydric alcohols (B.P. 313,892). Condensation products of urea etc. (B.P. 290,192).—See III. Lead oxide (B.P. 315,128).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Viscosity of rubber latex. C. M. BLOW (Trans. Faraday Soc., 1929, 25, 458–462).—The viscosities of different rubber latices at various concentrations have been determined, using Ostwald viscosimeters, the original latices being diluted with the sera obtained by filtration from the respective latices. Einstein's equation is found to hold for low concentrations. The changes in viscosity of a rubber latex when diluted with water have also been measured. The viscosity of latex which has been diluted with serum is very much dependent on the viscosity of the latter.

H. T. S. BRITTON.

PATENTS.

Manufacture of rubber goods. P. KLEIN and A. SZEGVARI, ASSRS. to ANODE RUBBER CO., LTD. (U.S.P. 1,719,984, 9.7.29. Appl., 5.6.25. U.K., 31.7.24).—See B.P. 245,177; B., 1926, 204.

Vulcanising rubber to leather. L. B. CONANT, ASSR. to STANDARD PATENT PROCESS CORP. (U.S.P. 1,719,101, 2.7.29. Appl., 2.7.27. Can., 29.7.26).—See B.P. 275,194; B., 1928, 494.

XV.—LEATHER; GLUE.

Preservation of hides with brine and salt. (Miss) M. KAYE (J.S.C.I., 1929, 48, 141–148 r).—The production of a perfectly preserved skin or hide necessitates a raw material in good condition, an efficient preservative, properly applied, and the prevention of subsequent conditions which would adversely affect the product. The efficiency of sodium chloride alone, either as brine or as crystals, as a preserving agent, and the effect of

the addition of certain "denaturants" are discussed. Microscopical observations on the histological changes which occur when hides are treated with salt solutions of different concentrations, and at different periods of time after death, show that it is essential to use either solid salt or a brine of at least 23% concentration, and that salting must be commenced within a few hours after death or permanent damage to the skin tissues will result. If the temperature be above 22°, the concentration of the brine should not fall below 27%. Brining previous to salting appears to be advantageous in that it ensures rapid penetration and prevents undue shrinkage. Of the colourless denaturants examined, sodium bifluoride and sodium carbonate improve the preserving action of the brine on the skin tissues; sodium fluoride gives approximately the same result as the saturated brine alone; sodium bisulphate produces some shrinkage of the tissues, and magnesium chloride appears to loosen the hide fibres too much. Of the coloured denaturants which are said to produce stains, potassium dichromate does not produce stains on hides, whereas copper sulphate produces stains which are only temporary if the hides are fresh, but become permanent if the hides are salted in a stale condition, or become dried after salting. Some common defects of salted hides (marbled grain, "pipe," loose grain, greasiness, and veininess) are attributed to different degrees of autolytic and bacterial action. Marbled grain appears to be the result of surface bacterial attack, whereas veininess in many cases is the result of partial destruction of the walls of blood-vessels.

Sulphuric acid in vegetable-tanned leather.

R. F. INNES (J.S.C.I., 1929, 48, 149–152 r).—Partly-tanned hides which have been plumped by immersion in dilute sulphuric acid lose the whole of the acid absorbed as soon as they are "struck through" by subsequent suspender liquors. The use of sulphuric acid in the early stages of tanning should not be harmful to the finished leather and cannot be the cause of a positive sulphuric acid test by the Proctor-Searle method. When sulphuric acid is found in finished leather it must have been applied to the tanned leather in the later stages of manufacture. Sulphuric acid combined with pelt (collagen sulphate) is markedly resistant to the action of both time and temperature. The rotting of leather is often associated with a high content of sulphuric acid, but a high content of sulphuric acid does not always lead to ready rotting, and attention is now being directed to the effect on rotting of the nature of the tannin used.

Determination of the p_H value of tan liquors and lime liquors. (Miss) W. B. PLEASS (J.S.C.I., 1929, 48, 152–156 r).—The glass electrode, hydrogen electrode, quinhydrone electrode, and colorimetric methods of determination of p_H values are discussed. Accurate and trustworthy results have been obtained by the glass electrode method for all kinds of liquors. The hydrogen electrode method gives accurate and trustworthy determinations when the liquors are free from substances which produce an oxidation-reduction potential. The quinhydrone electrode is untrustworthy for all trade tan liquors. When a capillator

compensating cell is used to compensate for the colour of the liquors, the capillator method gives results which are accurate to 0.2 p_H unit for all types of tan liquors, but is not applicable to lime liquors.

Effect of pretreatment on hydrolysis of hide powder by saturated calcium hydroxide solutions. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1929, 21, 701–702).—Samples of hide powder were exposed in atmospheres of 0, 50%, and 100% humidity respectively at 4°, room temperature, and 37.5°, until the protein had reached equilibrium with the moisture in the atmosphere, and the rate of hydrolysis by lime water was determined on them. It is shown that previous thorough desiccation of hide powder causes markedly greater hydrolysis in saturation solutions of calcium hydroxide. In similar tests the powder after pretreatment with sodium sulphate decomposed more readily in the calcium hydroxide than after pretreatment with sodium chloride. D. WOODROFFE.

Influence of acids on the fixation of wattle tannin by hide powder. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1929, 21, 697–698).—Portions of hide powder were treated with solutions of wattle bark tannin brought to different p_H values by means of various acids for 6 or 24 hrs., filtered in Wilson-Kern extractors, and the fixed tannin was determined. It is shown that the order of diminishing influence of the acid used for p_H 2.5–3.0 was acetic, lactic, formic, tartaric, oxalic, hydrochloric for a 6-hr. period of treatment, and acetic, lactic, succinic, formic, citric, tartaric, oxalic, chloroacetic, hydrochloric for a 24-hr. period. The latter list is almost inversely proportional to the dissociation constants of the acids. D. WOODROFFE.

PATENTS.

Treatment of leather. M. SEGORIA (U.S.P. 1,696,867, 25.12.28. Appl., 30.11.27).—Leather is rendered smoother and more durable and pliable by treatment with a mixture of 25 pts. of white pine oil, 5.8–6.0 pts. of linseed oil, and 6 pts. of beeswax. R. BRIGHTMAN.

Artificial leather (B.P. 314,571).—See XIII. **Hardened gelatin layers (B.P. 288,146).**—See XXI.

XVI.—AGRICULTURE.

Ultimate natural structure of soils. G. J. BOUYOUCOS (Soil Sci., 1929, 28, 27–37).—The ultimate natural structure of soil granules may be examined by allowing a dried natural soil to slake and disintegrate in a large quantity of water with gentle agitation. The gradation in the size of granules so obtained is stable, and considerable external force must be applied to reduce the original sizes of the granules. Mechanical analyses of such slaked soils may be made by the hydrometer method. The ultimate natural structure of soils, rather than the distribution of the ultimate particles, should form the basis of investigation of many of the physical properties of soils. The ordinary structure of field soils is not the ultimate natural structure, but a temporary structure resulting from the immediate treatment received. A. G. POLLARD.

Soil profile studies. I. Soil as an independent body and soil morphology. J. S. JOFFE (Soil Sci., 1929, 28, 39–54).—The formation of soil is discussed with special reference to the recent work of Russian investigators. The introduction of a special section of soil science for the study of buried soils is suggested. A. G. POLLARD.

Exchange reactions of insoluble alkaline-earth phosphates in permutits and clays. E. UNGERER (Kolloid-Z., 1929, 48, 237–241).—Experiments have been carried out on the interaction of potassium and ammonium permutits and of clay with magnesium phosphate, magnesium ammonium phosphate, and calcium phosphate. Exchange of bases takes place. The reactions are of importance in the decomposition of difficultly-soluble phosphates in soils. E. S. HEDGES.

Chemical composition of Florida Everglades peat soils, with special reference to their inorganic constituents. H. E. HAMMAR (Soil Sci., 1929, 28, 1–13).—Analyses of a number of soils are recorded and the formation of these soils is discussed. A. G. POLLARD.

Character, properties, and possible uses of bentonite, a sodium clay. R. M. WOODMAN and E. McK. TAYLOR (J.S.C.I., 1929, 48, 121–125 r).—Bentonite, a colloidal clay, has been proved to consist mainly of a sodium clay, exhibiting the usual characteristics of a sodium clay such as liberation of sodium hydroxide by hydrolysis in water, impermeability to water, etc. When suspended in water the sample examined yielded a solution of p_H 10.5. A determination of the replaceable bases showed 69.9 mg.-equiv. of replaceable sodium and 23.9 mg.-equiv. of replaceable calcium per 100 g. of dry material. It has been proved a good emulsifier for certain oils, usually forming the oil-in-water type of emulsion suitable for the spraying of plants. Evidence is given showing the formation of dual types with cresylic acid. In addition, bentonite has been proved to be a water-softener acting by base exchange, thus being useful in cases of permanent hardness. The fault of bentonite as a practicable emulsifier and water-softener is its impermeability to water.

Base exchange in orthoclase. J. F. BREAZEALE and O. C. MAGISTAD (Ariz. Agric. Exp. Sta. Tech. Bull., 1928, No. 24, 609–629).—The solubility in water of the potassium in orthoclase varies with the origin of the sample, the fineness of subdivision, and the ratio of solid to liquid. Treatment with alkaline salt solutions increased, whilst treatment with acid solutions decreased, the base-exchange capacity of finely-ground orthoclase. Digestion of ignited orthoclase with alkaline solutions may increase the base-exchanging capacity beyond that of non-ignited material. When solid orthoclase occurs in the soil in presence of calcium, magnesium, or sodium zeolite, an equilibrium between the potassium in solution and the zeolites will be established. The plant probably feeds largely on hydrolysed zeolitic potassium. The zeolites of calcium, magnesium, and sodium may act as stabilisers by fixing much of the soluble potassium during periods of little plant growth, and slowly liberating it as required by plants. CHEMICAL ABSTRACTS.

Hydrolysis of sodium and potassium zeolites with particular reference to potassium in the soil solution. O. C. MAGISTAD (Ariz. Agric. Exp. Sta. Tech. Bull., 1928, No. 22, 521—547).—The amount of alumina present as sodium aluminate on hydrolysis of sodium zeolite is a logarithmic function of the p_H of the solution. At equal dilutions the hydrolysis of potassium zeolite is less than that of sodium zeolite; in mixtures the hydrolysis is mutually depressed. The depression of the hydrolysis of potassium zeolite by calcium zeolite is diminished in solutions containing carbon dioxide. If the amount of replaceable potassium in 100 g. of soil is known, that in the soil solution at any dilution can be calculated. The amount and rate of hydrolysis of replaceable potassium are discussed in relation to plant feeding. CHEMICAL ABSTRACTS.

Soil zeolites and plant growth. J. F. BREAZEALE (Ariz. Agric. Exp. Sta. Tech. Bull., 1928, No. 21, 499—520).—The concentration of potassium in soil solutions is always low, and cannot exceed the sum of the solubilities of the potassium in the zeolite and in the orthoclase. Hence a heavy application of potassium salt does not proportionately increase the soil-extract potassium; hence, also, potassium is not readily leached away. The presence of other salts with replaceable bases, particularly nitrates, stimulates the absorption of potassium from solutions containing potassium zeolites. The zeolite residue of alumina and silica, after absorption of potassium by plants, exists either in the colloidal state or in some form of combination; zeolite may be regenerated by addition of a salt containing a replaceable base. Ignited and acid-treated soils which have lost their property of base-replacement are partly restored by treatment with salt solutions having a neutral or alkaline reaction.

CHEMICAL ABSTRACTS.

Availability of potassium in some Scottish soils. R. STEWART (J. Agric. Sci., 1929, 19, 524—532).—The total potash content of 34 samples from 13 soils typical of large areas in the East of Scotland averaged 2.04%. No relation was found between total potash and potash soluble in hot concentrated hydrochloric acid; the average figure for the latter was 0.50%. In the profile samples there was generally an increase in solubility with increase in depth. The average amount of citric-soluble potash was 0.0074%, and with one notable exception citric solubility decreased with depth. The citric-soluble potash varied from 26 to 79% of the exchangeable potash. The Neubauer method of analysis applied to 10 of the samples indicated a deficiency of available potash in most cases, and comparison with the figures for exchangeable potash indicated that the content of exchangeable potash determines the amount of potash which can be absorbed from a soil by the seedlings. E. HOLMES.

Rapid electrometric determination of chloride content of soils. R. J. BEST (J. Agric. Sci., 1929, 19, 533—540).—An apparatus consisting essentially of a quinhydrone electrode and an $Ag/AgCl$ electrode connected internally by means of a 3% agar-saturated potassium nitrate salt bridge, in series with a galvanometer of such a sensitivity that it gives a deflection of

one division per micro-amp., has been devised. The $Ag/AgCl$ electrode and the agar tube are dipped into a suspension of 4 g. of soil in 50 c.c. of distilled water, and titration with silver nitrate follows the usual course. Using a buffer solution of p_H 3.0—3.3, if titration is carried out between 25° and 16° with an $N/35.46$ silver nitrate solution the current reverses its direction at the end-point of the titration. E. HOLMES.

Cobaltinitrite (volumetric) method of determining potassium in soil extracts. G. MILNE (J. Agric. Sci., 1929, 19, 541—552).—A volumetric cobaltinitrite method, which is described in minute detail, enables known amounts of potassium to be determined, independently of the presence of alkaline-earth sulphates, or phosphates, provided the amount of potassium is not varied over too great a range. The factor 0.000830 g. K_2O per c.c. of 0.1N-permanganate suits the prescribed procedure over a range of 3—50 mg. of K_2O . The method is applicable to plant-ash analyses, to more accurate small-quantity work on soils than the perchlorate method, and also to citric acid extracts. Analyses of ammonium chloride extracts have not proved satisfactory. E. HOLMES.

Alterations of soil structure by natural and artificial manures. G. HAGER (Mitt. deut. landw. Ges., 1929, 1, 143; Bied. Zentr., 1929, 58, 293—294).—Humus and basic calcium compounds are indispensable for the maintenance of good soil structure. Calcium chloride and nitrate and gypsum are less effective since they are not intimately associated with the soil particles and cannot remove soil acids. Basic magnesium compounds are similarly favourable to soil structure but to a smaller extent than the corresponding calcium salts. Owing to the formation of ammonium carbonate from the urea contained in liquid manure the latter tends to incrust and "puddle" heavy soils. Superphosphate has no effect on soil structure, and no unfavourable effects result from the use of ammonium sulphate or combined fertilisers containing ammonium salts. The unbalanced use of potash salts and of sodium nitrate is injurious to soil structure, but the ill-effects may be avoided by the judicious combination with other fertilisers. A. G. POLLARD.

Comparative effect of various nitrogen fertilisers on bearing apple trees of low vigour. A. L. SCHRAEDER and E. C. AUCHTER (Proc. Amer. Soc. Hort. Sci., 1927, 229—234).—Sodium nitrate causes a greater terminal spur and trunk circumference growth and higher yields than ammonium sulphate. The total and soluble nitrogen of spurs was greater for sodium nitrate; spur starch in June was favoured by ammonium sulphate. CHEMICAL ABSTRACTS.

Nitrogenous manuring of grassland. H. W. GARDNER, J. H. SMITH, J. W. REID, and H. R. WILLIAMS (J. Agric. Sci., 1929, 19, 500—523).—The results of two years' treatment of grass by periodic dressings with nitrogenous manures, following autumn complete dressings, coupled with systematic grazing, indicate that the addition of 4 cwt. of ammonium sulphate in fractional amounts raised the stock-carrying capacity of the treated plots by about 50% in 1927 and 20% (with 3 cwt.) in

1928. The percentage of clovers on the nitrogen-treated plots was only one quarter that on the controls, whilst the dry matter of the herbage averaged 17.7% and 15.5% respectively of crude protein. The tips of the young grass contained 17.72% of dry matter, of which 27.2% was crude protein, and the rest of the herbage 14.41% of dry matter (19.5% protein).

E. HOLMES.

Yield in cereals. VI. A. Influence of nitrogenous top-dressing on wheat. B. Influence of disease ("take-all") on the yield of wheat. L. R. DOUGHTY, F. L. ENGLENDOW, and T. K. SANSOM (J. Agric. Sci., 1929, 19, 472—490).—An elaborate analysis of results of tillering counts, yield measurements, and other examinations of a series of 1-ft. row samples of nitrogenously manured winter wheat, grown on an imperfectly drained gault clay in the Eastern Counties, leads to the conclusions that increased survival of tillers is not induced by a February nitrogenous dressing. Owing to the disease "take-all" yields were low, but the dressed strips yielded at the rate of 26.4 bushels per acre and the untreated at 19.1 bushels per acre. The common practice of February or March top-dressing augments ear size, but cannot raise yield per acre in any other way. For increased ear-formation December application might be necessary, but practical considerations suggest a half dressing at sowing and the rest at the beginning of February.

E. HOLMES.

Effect of anions of potassium salts on potato heads and the effect of root activity on the soil. W. VON BREHMER (Ernähr. Pflanze, 1928, 24, 393—397; Chem. Zentr., 1929, i, 691—692).—The p_H of the soil, adjusted to 4.0 by addition of potassium hydrogen sulphate after fertilisation, was increased by plant growth to 7.3. In the "chloride and sulphate series" the p_H curve was nearly parallel with the head-height.

A. A. ELDRIDGE.

Analysis of tomato plants. I. O. OWEN (J. Agric. Sci., 1929, 19, 413—432).—At the Cheshunt Research Station phosphorus does not appear to play such an important rôle in tomato culture as American work suggests. Unmanured plants give fruit of inferior quality, but the weight ratio of fruit to foliage and stems is about 2.6 as compared with 1.6 on the manured plants. The removal of plant food per plant averages 9.3, 2.0, and 18.2 g. of N, P_2O_5 , and K_2O respectively on manured plants, and only 4.9, 1.0, and 2.8 g. respectively on the unmanured plants. Analyses of trimmings and leaves at different periods of the year suggest that the actively growing parts are richest in plant nutrients. Fresh fruits contain the following percentages of N, P_2O_5 , and K_2O : 0.16, 0.05, and 0.32; and 0.18, 0.035, and 0.055 in the manured and unmanured plants respectively. Tomato plants yielding good crops show a relatively high ash content. The third and fourth trusses on a plant are richest in plant nutrients, but the relative amounts of potash in the aerial parts of the plant fall as the season progresses.

E. HOLMES.

Active lime of dephosphoration slags and of so-called disaggregated phosphates. C. BRIOUX and E. JOURIS (Compt. rend., 1929, 189, 117—119).—

In addition to free lime, the slags contain calcium silicates and silicophosphates which are more or less rapidly decomposed by water in the presence of carbon dioxide, with the formation of calcium carbonate. Their activity may be measured from the gradual change in p_H value when 1 g. is well mixed with 500 g. of an acid soil of known lime requirement and p_H value, in the presence of 20% of water. In the cases recorded and with a soil of p_H 5.49, decomposition was complete after 8 days, and the active lime content (including alkalinity due to soda and potash in the case of the disaggregated phosphate) exceeded 30%. With more alkaline soils, however, it is less.

J. GRANT.

Fungicidal properties of certain spray-fluids. V. W. GOODWIN, H. MARTIN, and E. S. SALMON (J. Agric. Sci., 1929, 19, 405—412; cf. B., 1926, 506).—In experiments with the fungus *Sphaerotheca humuli* (DC.), Burr., on a hop known to be susceptible to mildew, it was shown that a solution of dicalcium hydrogen arsenate containing 0.0125% As_2O_5 was fungicidal, whilst the solution containing 0.006% was not. In a similar manner calcium thioarsenate solution equivalent to 0.006% of As_2O_5 was fungicidal, whilst that equivalent to 0.003% was not. "Lime casein" containing calcium hydroxide reduced the fungicidal properties of calcium arsenate sprays. It is suggested that the increased fungicidal properties of the mixed lime-sulphur-lead arsenate spray are due to the presence of calcium thioarsenates.

E. HOLMES.

Pyrethrin I and II. Their determination in pyrethrum (*Chrysanthemum cinerariæfolium*). II. F. TATTERSFIELD and R. P. HOBSON (J. Agric. Sci., 1929, 19, 433—437; cf. B., 1929, 488).—Although different compositions have been ascribed to the active insecticidal principles of Japanese and Swiss pyrethrum, it is now shown that the acid method of determining pyrethrin I and II is equally suitable for testing flowers from Japanese and from Swiss seed, indicating that the poisons are identical. A quick method of acid analysis of the more important pyrethrin I has been devised, in which 10 g. of the ground pyrethrum is extracted with light petroleum (40—50°), the extract is added to 4—5 c.c. of *N*-caustic soda in methyl alcohol, and the mixture refluxed for 1½—2 hrs., when it is acidified with *N*-sulphuric acid and steam-distilled. The first and second 50 c.c. of aqueous petroleum distillate are transferred successively to a separating funnel, the aqueous layer is removed, and the residue added to 20 c.c. of water and titrated with 0.02*N*-caustic soda, using phenolphthalein as indicator; 1 c.c. of 0.02*N*-alkali = 3.36 mg. of monocarboxylic acid = 6.6 mg. of pyrethrin I.

E. HOLMES.

Boron in the soils and irrigation waters of Southern California and its relation to citrus and walnut culture. W. P. KELLEY and S. M. BROWN (Hilgardia, 1928, 3, 445—458).

Sulphur-spray residues. CULPEPPER.—See XIX.

PATENTS.

Selection of cottonseed. V. U. CLOER (U.S.P. 1,718,332, 25.6.29. Appl., 24.12.26).—After treatment under pressure with a gaseous hydrogen halide, the dry

seed is heated to 82–99°, and then agitated to remove the lint. F. G. CLARKE.

Production of fertiliser mixtures. F. L. SCHMIDT (B.P. 296,022, 1.8.28. Ger., 23.8.27).—The fertilisers are obtained by roasting mineral phosphates etc. with potassium carbonate produced by treating potassium chloride with magnesium carbonate and carbon dioxide to yield potassium magnesium bicarbonate and subsequently decomposing this into potassium and magnesium carbonates. The carbon dioxide evolved during the roasting and the magnesium carbonate are used again in the process. L. A. COLES.

Production of fertiliser. A. MACLACHLAN, Assr. to AMER. BY-PRODUCT MACHINERY CO. (U.S.P. 1,718,297, 25.6.29. Appl., 12.5.27).—Material high in casein, *e.g.*, poultry manure, is acidified and evaporated to dryness. H. ROYAL-DAWSON.

Insecticidal plant food or fertiliser. F. G. DOKKENWADEL, Assr. to T. W. MILLNER, V. R. WALLEY, B. M. STEVENS, E. C. FISHEL, and J. E. GREEN (U.S.P. 1,696,709, 25.12.28. Appl., 19.11.26).—Cottonseed hulls are incorporated or impregnated with active fertilisers, with or without the addition of phenol or other insecticide or insect-repelling material. R. BRIGHTMAN.

Improving plant materials used for floral decoration etc. [by coating with varnish etc.]. E. ELÖD and W. BLÜCHEL (B.P. 295,052, 4.8.28. Ger., 6.8.27).

Pest-destroying agents (B.P. 313,934).—See III.

XVII.—SUGARS; STARCHES; GUMS.

Desiccation and briquetting of [beet] pulp. L. TERNYNCK (Bull. Assoc. Chim. Sucr., 1929, 46, 200–218).—The drying of exhausted beet pulp is strongly advocated on grounds of economy of transport, and for other reasons. Ensilage of the wet pulp involves losses of nutritive matter which may amount to 30% in three months, and the product is not suitable for feeding cattle which supply milk for infants. Preparatory to drying, modern pulp presses will raise the dry-substance content of fresh pulp to 16–18%, or even to 19–20%. A process and plant are described for automatically mixing a proportion, *e.g.*, 25%, of molasses with dried pulp, and compressing the mixture to cakes of standardised weight which do not crumble and yet disintegrate completely in water within $\frac{1}{2}$ hr. The compression is effected in two stages, first under 8 kg. and then under 120 kg./cm.² (114 and 170 lb./sq. in.). The apparatus will produce 15 cakes of 5 kg. each per min., with a steam consumption of 300 kg. per hr., the waste steam serving for evaporation. This process avoids bagging and simplifies transport of the dried pulp, and the molasses adds to the feeding value of the product. J. H. LANE.

Control of polarisation losses in [beet] sugar manufacture. A. DUMOULIN (Bull. Assoc. Chim. Sucr., 1929, 46, 218–225).—Methods are described for calculating sugar losses in exhausted slices, diffusion waste waters, and carbonation scums, and those occurring during evaporation, so as to arrive at a correct balance of sugar entering and leaving the factory. The loss in exhausted slices is best calculated to original beets on the basis of the relative amounts of marc in both. The

volume of diffusion juice obtained, as measured directly, needs correction for temperature, if this differs much from 20°, and also for air bubbles; the latter correction was found to be 3.5% of the measured volume of the juice. For the determination of the total sugar lost in the carbonation scums the ammonium nitrate method is recommended. Observed decreases of the purity quotient of the juice during evaporation, amounting to 0.26–0.5%, cannot be accounted for by the influence of evolution of ammonia and carbon dioxide on the density, but are attributed to actual destruction of sugar. J. H. LANE.

Physico-chemical analysis of massecuites. D. SIDERSKY (Bull. Assoc. Chim. Sucr., 1929, 46, 272–277).—In studying the progressive exhaustion of massecuites in the crystallisers, the accurate determination of the amount of sugar still in solution at any stage is rendered difficult by the presence of microscopic sugar crystals which cannot be separated from the molasses by centrifuging. These crystals, however, do not affect the refractive index of the molasses as determined by means of reflected light, a method of observation which is possible with several forms of sugar refractometer now obtainable. J. H. LANE.

Determination of starch in sound and frozen potatoes. SCHWONKE (Z. Spiritusind., 1929, 52, 198).—A criticism of the suggestions of Lampe and Kilp (B., 1929, 575). The accurate sampling of damaged tubers is so difficult that the results of chemical analysis would still be of doubtful value. If the sample is divided into sound and unsound portions and the starch determined on the former, the result may be applied to the whole sample for technical valuation, since though the unsound potatoes usually contain more starch owing to loss of water, this is counterbalanced by increased losses in their use. F. E. DAY.

Determination of starch in sound and frozen potatoes. B. LAMPE and W. KILP (Z. Spiritusind., 1929, 52, 198–199).—A reply to Schwonke (preceding). F. E. DAY.

Starch factory control. II. Determination of removable starch in pulp. III. Determination of fixed starch in pulp. O. WOLFF (Z. Spiritusind., 1929, 52, 222–223, 231–232; cf. Sprockhoff, B., 1929, 223).—For well-mixed material 100–200 g. of sample are sufficient. Excessive water may be removed by squeezing through a thick dry cloth, but material containing over 10% of dry matter is difficult to mix. The removable starch is determined by washing the pulp with a brisk jet of water through an apparatus consisting essentially of a series of graded fine sieves to retain fibre. The starch is finally collected in a centrifuge tube, and its volume (in c.c.) $\times 0.6 =$ g. of dry starch. Moisture must be determined by drying a portion at 120°, and tables are given to facilitate the calculation and to illustrate the effect of varying total starch content on the results when calculated to the original material. The total starch should thus always be determined, preferably by the author's interferometric method (B., 1924, 567), modified by omitting the grinding of the control test. Experience has shown this method to give results which are readily duplicated,

and are lower than those obtained by acid hydrolysis and higher than those by the Maerck-Delbrück diastase method, in which some starch remains unattacked. The fixed starch is determined by difference. F. E. DAY.

PATENTS.

Refining of sugar. H. WIESE (B.P. 314,750, 24.11.28).—The solution of raw sugar is heated to above 70°, mixed with 3–5% of lime as cream and carbonated till neutral, then heated to above 80° by injection of steam, and filtered. E. B. HUGHES.

Production of gum from locust beans. W. WINDGASSEN (B.P. 315,173, 13.11.28).—Alkylated naphthalenesulphonic acids are added to the ground or unground albumin of the beans or to the water used for lixiviating them. L. A. COLES.

Activated charcoal (U.S.P. 1,704,765).—See II.

XVIII.—FERMENTATION INDUSTRIES.

Preparation of an active dried pitching-yeast. F. WINDISCH (Woch. Brau., 1929, 46, 288–291).—Bottom-fermentation yeasts (races U and D) were pressed, crumbled, and dried at 2°, 20–22°, and 33°. After 11 days they were seeded into wort. Though all the dried yeasts stained completely with methylene-blue, those dried at 2° gave fermentations and cell increases but little lower than those of the control fresh yeast. Those dried at 33° were much inferior, but better than those dried at 20–22°, probably because more rapid drying lessened autolysis. F. E. DAY.

Extraction and utilisation of pectase. PAUL and GRANDSEIGNE (Bull. Assoc. Chim. Sucr., 1929, 46, 233–237, 245–246).—Pectase or pectinase occurs in certain plants either in an insoluble form, in association with other substances, or in a pseudo-soluble form precipitable by alcohol, the precipitate becoming almost completely insoluble on drying. Its characteristic action is the transformation of pectin into gelatinous compounds, pectic acid and pectates, in presence of alkaline-earth salts or (less readily) magnesium salts. The optimum temperature for this action is 52–55°. The enzyme is destroyed very slowly at 70° and rapidly at 80°. Acids have a retarding influence, which is perceptible in 0.06% hydrochloric acid and very strong in 1% acid. A 1% solution of pectin is transformed into a very firm jelly by pectase, and only minute amounts of the enzyme are required. In absence of pectase pectin yields gels in liquids containing free alkalis or alkaline-earths or in presence of free acids and sugar; as a purifying or clarifying agent it can be used alone only in alkaline media. In conjunction with pectase, however, it can be used in neutral or very faintly acid liquids, if alkaline-earth salts are present. Milk can be transformed into a jelly by pectin and pectase without heating and without loss of vitamins. The same means can be used for manufacturing jellies free from sugar, for diabetic patients, and other gelatinous pharmaceutical products. Solid pectase preparations which retain their activity indefinitely and readily dissolve or disperse in water are made from suitable plant juices, after a preliminary purification of the latter, by adding a colloid which readily forms sols in water, and then precipitating

the colloid with acetone. The precipitate contains the pectase of the original juice, and is dried at 50° and maintained dry until required for use. J. H. LANE.

Concentration of lichenase and cellulase. V. ENZYMES of barley malt. G. OTTO (Biochem. Z., 1929, 209, 276–289).—Attempts were made to concentrate the enzymes from extract of barley malt by adsorption and elution. Almost quantitative adsorption was obtained by shaking with aluminium β -hydroxide and glass beads in hydrochloric acid-citrate buffer at p_H 4. Various methods of elution were tried, but a loss of not less than 50% of the enzyme always occurred. The maximum concentration effected using 0.5% ammonia solution and glycerol for elution was 3.4 times. With silica gel the results were less favourable. By evaporation of the malt extract in a stream of air and *in vacuo*, concentration by six to eight times was obtained. The preparation after concentration two to three times readily yielded dried samples by precipitating with acetone (cf. A., 1928, 671).

P. W. CLUTTERBUCK.

Volumetric determination of carbon dioxide in beer. J. DE CLERCK (Bull. Assoc. anc. Elèves Louvain, 1928, 29, 35; Woch. Brau., 1929, 52, 262–263).—The titration is performed in a Walpole comparator. To 15 c.c. of 0.1N-caustic soda 10 c.c. of well-cooled beer are added in each of one pair of vessels. To one 2.5 c.c. of 0.5% phenolphthalein are added, to the other 2.5 c.c. of water. The other pair of vessels contain, respectively, water and 25 c.c. of Clark and Lubs' borate buffer, p_H 8.4, with 2.5 c.c. of phenolphthalein solution. An approximate result is obtained by adding 0.1N-hydrochloric acid to the glass containing beer and indicator until the two sides match in tint. In a second accurate titration an amount of hydrochloric acid equal to that used is added to the beer and water, and the volume of the buffer solution is increased by the same amount. This adjusts the p_H of the beer and concentration of the indicator to about that which will finally be present in the test. A similar titration is made on the same beer after removal of the carbon dioxide. By subtracting the value of this "fixed acidity" from the former result the volume of 0.1N-soda necessary to convert the carbon dioxide into bicarbonate is obtained. F. E. DAY.

Potato flakes as distillery raw material. E. LÜHDER (Z. Spiritusind., 1929, 52, 221–222).—The difficulties attending the sole use of potato flakes may be overcome by replacing about half of the fresh potatoes by the equivalent quantity of flakes. The flakes may be soaked and liquefied in hot water while the potatoes are undergoing digestion under pressure. The liquefied mass of flakes is cooled below saccharifying temperature, which is reached and maintained by addition of the digested potatoes. Green malt of good saccharifying and liquefying power is used at the usual rate of 8–10% of the equivalent raw material, and the flakes yield 38–40 litres of alcohol per 100 kg. Another method, which avoids preheating the flakes to 75–78°, is to mash the flakes with a small quantity of green malt in water at 52° from the dephlegmator, with continual stirring. By this process 600 kg. of flakes (including

malt) yielded 254 litres of alcohol. The yield is better and the procedure simpler than with maize, but the residue has a lower value as fodder, though an equal value to that from fresh potatoes. The procedure may be applied to flaked maize. F. E. DAY.

Balance of effects of different components on the b.p. of wine. O. HÖGL (Z. Unters. Lebensm., 1929, 57, 288—312).—The effects of various sugars, salts, acids, etc. on the b.p. of wine are found to be quite additive even when the depressing effects of salts and the elevating effects of acids are combined. The b.p. of a wine calculated from its analysis and that found by the ebullioscopic method agree closely. In sound wine not too poor in alcohol the effects of the various components neutralise one another almost exactly, whereas weak sour wine tends to have a higher and fortified neutralised wine a lower b.p. than that expected from the alcohol content. W. J. BOYD.

Chemical changes in wine due to moulds. H. SCHELLENBERG and G. KUHN (Z. Unters. Lebensm., 1929, 57, 313—325).—The density of wine attacked by moulds rises owing to removal of alcohol and subsequently to production of acetic acid. The quantity of glycerol and non-volatile reducing substances decreases in low concentrations of alcohol. Total acids diminish first and then increase. Non-volatile acids steadily decrease. Hydrogen-ion concentration is constant at first, subsequently rising as acetic acid is formed. Of the organic acids tartaric is the most stable and succinic the most readily attacked. Lactic, malic, and tannic acids are destroyed. Ash content decreases owing to removal of potash, phosphoric and sulphuric acids. Alkalinity of the ash increases owing to increase in carbonate as these inorganic acids are removed. Ammonia is used up more rapidly than other forms of nitrogen. A portion of the organically combined nitrogen is not utilised by the mould. Wines from marc deficient in nitrogen and phosphoric acid are poor nutrient media for moulds. W. J. BOYD.

Luminescence of wood extracts, wine distillates, brandies, and vinegars in ultra-violet light. G. REIF (Z. Unters. Lebensm., 1929, 57, 269—276).—Alcoholic and acetic acid wood extracts, wine distillates the colour of which is due to substances extracted from the wood of the casks, and brandies made from grape marc, from fruit, and from grain show almost complete correspondence between their content of tannin substances and the intensity of their luminescence. The luminescence of certain wine distillates, brandies, arrack, and rum is also influenced by other substances, and that of fermentation vinegar chiefly by substances of a protein nature. W. J. BOYD.

Werder's method for the detection of fruit wines in grape wines. C. VON DER HEIDE and K. HENNIG (Z. anal. Chem., 1929, 77, 441—450).—The method (B., 1929, 619) has been investigated and found to be satisfactory. Admixture of 5% of fruit wine with grape wine is readily detected. Grape wines, however, often contain mannitol, which also forms a benzylidene derivative (m.p. 213—218°), and hence the benzylidene derivative obtained should be confirmed by determination of m.p. and by microscopical examination. J. S. CARTER.

Rapid determination of the dry extract in vinegar. G. DONÀ (Giorn. Chim. Ind. Appl., 1929, 11, 264—265).—The vinegar is evaporated to one third of its volume and the residual liquid made up to the original volume with water and titrated to determine its acidity. An equal volume of an acetic acid solution of equal titre is also prepared. A special highly sensitive densimeter, previously standardised with various liquids with known contents of extract, is immersed in the acetic acid solution and its movable scale adjusted to coincide with the meniscus of the liquid. It is then immersed in the liquid under test, the reading on the scale, divided by the constant of the apparatus, giving the weight of dry extract in the original vinegar. T. H. POPE.

Distillation columns for the production of alcohol. J. PÉRARD (Bull. Assoc. Chim. Sucr., 1929, 46, 247—252).—Practical notes are given on the working and control of columns in agricultural distilleries producing alcohol of 90—91% from wines of 5% or lower alcohol content. J. H. LANE.

XIX.—FOODS.

Effect of heat on milk: (A) on the coagulability by rennet; (B) on the nitrogen, phosphorus, and calcium content. E. C. V. MATTICK and H. S. IALLET (J. Agric. Sci., 1929, 19, 452—462).—Milk which has been heated for $\frac{1}{2}$ hr. at 41.2—43.4° coagulates with rennet in about 3 min. after keeping for about 5 hrs.; that heated at 90.6—92.3° only coagulates if the rennet is added within $\frac{1}{2}$ hr. of heating, and that heated to 96.2—98.4° cannot be coagulated at any time after heating. No change takes place in the diffusibility of the nitrogenous substances in milk after heating through the same range of temperatures, but the diffusibility of the phosphorus content appears to be reduced after heating at 79.5° for $\frac{1}{2}$ hr., and that of the calcium content is markedly diminished after heating at 51.7° only. E. HOLMES.

Dispersoid-chemical methods for the examination of milk. A. SCHNECK (Milchwirt. Forsch., 1928, 7, 1—34; Chem. Zentr., 1929, i, 704—705).—The transparency of diluted milk is determined with a potassium-argon cell. At 1 in 250 Beer's law is approximately obeyed by both full-cream and skimmed milk. The extinction of skimmed milk at this dilution is practically independent of the origin and only a few % of that of full cream milk. In clotting, the extinction coefficient of the fat is unchanged. The clotting time is purely arbitrary. A. A. ELDRIDGE.

Physical examination of milk. W. GRIMMER and H. BENDUSKI (Milchwirt. Forsch., 1928, 7, 76—79; Chem. Zentr., 1929, i, 704).—In souring milk the hydrogen-ion concentration at first rises nearly parallel with the titratable acidity, and later much more rapidly; possibly the relation is exponential. With increase of titratable acidity the refraction of the calcium chloride serum is not parallel to either acidity; three types of relation are distinguished. A. A. ELDRIDGE.

Action of viscogen (calcium saccharate) on milk and cream. G. T. PYNE (J. Agric. Sci., 1929, 19, 463—471).—The increase in the viscosities of milk and cream brought about by the addition of viscogen is

due primarily to the formation of a precipitate of tricalcium phosphate. Considerable quantities of casein are carried down, and this is probably the factor which most influences viscosity. Casein is not directly precipitated by viscogen, but the viscosity of solutions is slightly increased as a result of the increase in alkalinity induced. E. HOLMES.

Toxicity of vegetable milk for infants. K. STOCKERT and E. GRÜNSTEIDL (Z. Unters. Lebensm., 1929, 57, 326—338).—In view of the possibility of bitter almonds being present among the sweet almonds used for preparing vegetable milk (a 7.5% emulsion of almonds in milk-serum) the effect of the process of preparation and the various components on the scission of amygdalin by emulsin was studied, as well as the effect of the digestive juices of the stomach and intestine on amygdalin. The action of emulsin is highest in pure water and decreases with successive additions of individual milk components. It is further lowered by addition of boiled vegetable extract along with the milk-serum, being less than half that in pure water. Pasteurisation of the product at 60° increases the scission of amygdalin by emulsin, but heating at 80° for 1 min. destroys the enzyme. It is concluded that there is no risk of injurious effects through scission of amygdalin during preparation of the product. The enzymes of the stomach and the intestine hydrolyse amygdalin to a small extent. Admixture of 10% of bitter almonds with sweet almonds is readily detected by the odour in preparation of the emulsion, and it is shown that no serious effects can result from the inclusion of several times this proportion.

W. J. BOYR.

Significance of choline in summer and winter milk. W. VAUBEL (Deut. med. Woch., 1928, 54, 1971; Chem. Zentr., 1929, i, 587).—Summer milk or butter contains a considerably higher proportion of lecithin or choline than the winter product. A. A. ELDRIDGE.

Test for vitamin-A in margarine, butter, and other fatty foods. A. ANDERSEN and E. NIGHTINGALE (J.S.C.I., 1929, 48, 139—140).—The authors describe a modified application to fats and fatty foods of Carr and Price's coloration test with antimonious chloride which yields a blue colour when added to oils rich in vitamin-A. As vitamin-A is associated with the unsaponifiable fraction in fats this fraction is isolated by saponifying the fat and extracting the unsaponifiable matter with ether; the ethereal solution is decolorised with charcoal to remove natural or artificial colouring substances and dried with anhydrous sodium sulphate. After filtration the ether is evaporated and the unsaponifiable residue dissolved in a volume of chloroform equal to the original quantity of fatty food under examination. A "dilution test" is now applied by diluting the chloroform solution or an aliquot part until it gives a just visible blue colour when to 1 c.c. of the diluted solution is added 1 c.c. of the antimonious chloride. The volume to which the chloroform solution can be diluted to give this just visible colour expresses numerically the potency in vitamin-A of the product. The method is particularly applicable to butters and vitaminised margarines, but is also being extended to milk and other fatty foods.

Tin foil as a material for packing crustless cheese. ELTEN (Chem.-Ztg., 1929, 53, 586).—The corrosion of the tin-foil wrapping of crustless cheese is accompanied by the formation of a black film of antimony on the inside of the foil and by the development of a grey or brown colour in parts of the cheese adjacent to the corroded metal. Tests on some samples of cheese showed the presence of over 2% Sn in the affected parts, which also had the high acid value of 2.3—2.4 compared with a normal of about 1.5. The trouble therefore appears to be due to inefficient mixing of the acid (generally citric acid) used with the powdered cheese prior to melting for filling the containers.

A. R. POWELL.

Storage of rice. M. KONDO and T. OKAMURA (Ber. Ohara Inst. landw. Forsch., 1929, 4, 1—18).—Data are given illustrating the superiority of storing hulled rice in airtight vessels or in carbon dioxide over the normal method used by the Japanese farmer of storage in straw bags.

E. A. LUNT.

Sulphur-spray residues and the swelling of tin cans packed with peaches. C. W. CULPEPPER (J. Agric. Res., 1929, 39, 31—40).—The swelling of tins of peaches is traced to the generation of hydrogen and hydrogen sulphide from the residues of sulphur sprays. Washing of fruit previous to tinning with hot or cold water does not always remove the sulphur residue. Dipping the fruit in hot dilute soda lye for a few seconds and washing with jets of water proved more satisfactory. All sulphur compounds examined caused swelling and in addition attacked the metal, producing catalytic substances accelerating the normal action of fruit juices on the tin. The presence of organic acids is necessary for swelling to occur, but corrosion may take place in the absence of acids if the sulphur exists in a form capable of combining with the metal. The organic acids liberate hydrogen from iron but not from tin. The corrosion may be explained electrochemically.

A. G. POLLARD.

Effect of exhaustion on the formation of hydrogen swells in canned-ready-to-serve prunes. E. M. MRAK and P. H. RICHERT (Fruit Prod. J., 1928, No. 6, 17—19).—Exhaustion is preferably effected for 10 min. at 99°.

CHEMICAL ABSTRACTS.

Banana pectin. H. VON LOESECKE (Fruit Prod. J., 1929, 8, No. 6, 14—16).—Pectin extracted from banana peel has a low jellying power and poor keeping quality.

CHEMICAL ABSTRACTS.

Respiration factor in the deterioration of fresh vegetables at room temperature. M. J. BENOY (J. Agric. Res., 1929, 39, 75—80).—Respiration changes occurring in a number of green vegetables during storage for 24 hrs. are recorded. The carbon dioxide evolved corresponded to the oxidation of dextrose ranging from 1 to 14% of the dry matter of the materials examined.

A. G. POLLARD.

Detection and determination of carbon disulphide in fumigated almonds. G. MARSH and W. V. CRUESS (Fruit Prod. J., 1929, 8, No. 6, 31, 34).—Huff's method is modified.

CHEMICAL ABSTRACTS.

Testing of seeds and other products by means of the quartz lamp. A. NIETHAMMER (Z. Unters.

Lebensm., 1929, 57, 354—358).—Sections of various seeds and nuts and samples of various oils were examined in ultra-violet light. In many cases it was possible to distinguish fresh from old samples of these products thereby. Various pure fatty acids were examined; most of these exhibit a lilac-violet luminescence with minor differences of shade. It was not possible to distinguish coffee substitutes from coffee by means of the quartz lamp. W. J. BOYD.

Rancidity of fat in seeds etc. NIETHAMMER.—See XII. **Beet pulp.** TERNYNCK. **Starch in potatoes.** SCHWONKE: LAMPE and KILP.—See XVII. **Pectase.** PAUL and GRANDSEIGNE.—See XVIII.

PATENTS.

Improving the milling quality of grain. K. DIENST (B.P. 302,992, 1.8.28. Ger., 24.12.27).—Grain previously cleaned, washed, and dehusked by a wet method is repeatedly moistened, passed through drying columns, and then heated to about 70° and cooled to about 20°, thus producing particles of uniform hardness requiring only low-power milling and giving high yields of white flour of good baking quality. E. B. HUGHES.

Preservation of [egg]-albumin. A. J. BELLAMY, and EGG PATENTS, LTD. (B.P. 314,887, 2.4.28).—Egg white is neutralised by acid (e.g., phosphoric acid) and then dried; 0.1—5% of glycerin may be added before or after neutralisation. Suitable apparatus is claimed. E. B. HUGHES.

Freezing of food substances. GEN. FOODS CO., and C. BIRDSEYE (B.P. 292,457, 7.2.28. U.S., 18.6.27).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Detection of isopropyl alcohol in cosmetics by means of piperonal. G. REIF (Z. Unters. Lebensm., 1929, 57, 277—288).—The alcoholic distillate from 10 c.c. of the sample is cooled in ice-water, 1 c.c. is added to 3 c.c. of an aqueous solution of hydroxylamine hydrochloride (0.05 g. of hydroxylamine hydrochloride for mouth washes and perfumes and 0.1 g. for hair washes), the mixture is shaken, and after 3 min. again shaken with 0.4 g. of animal charcoal and filtered; 5 c.c. of a 0.5% solution of piperonal in alcohol, and then 20 c.c. of sulphuric acid are added slowly to avoid boiling. The method is continued as for the detection of isopropyl alcohol in brandy (B., 1928, 686).

W. J. BOYD.

Chemical composition of tobacco. A. SCHMUK and V. BALALUCHA (U.S.S.R. State Inst. Tobacco Inv., Bull. 49, 1929, 5—62).—Analytical data are given for tobaccos from Kouban, Crimea, Black Sea coast, and Abkhazia with reference to protein, total nitrogen, nicotine, free alkali, ammonia, ash, carbohydrate, and polyphenol contents. Decrease in the last two of these constituents and increase in the others are indicative of good tobacco. E. A. LUNT.

Ethereal oil extract of German garden pink. W. TREFF and H. WITTRISCH (J. pr. Chem., 1929, [ii], 122, 332—339; cf. B., 1926, 850).—The blossoms of the garden pink (*Dianthus caryophyllus*, L.) yield 0.0432% of essential oil, d_{25}^{20} 1.0375, the composition of which

has been determined. After removal of small amounts of aldehydes and acids by shaking with sodium hydrogen sulphite and hydrogen carbonate, phenolic constituents are isolated with cold 3% and 10% solutions of sodium hydroxide. The extract contains a little methyl salicylate, determined by hydrolysis with 25% sodium hydroxide solution, whilst distillation of the remaining phenolic fraction in a vacuum yields mainly eugenol. The primary alcohols present in the ethereal extract of the alkaline solution (above), after neutralisation, were separated by esterification with phthalic anhydride. Hydrolysis of the phthalates yields mainly phenylethyl alcohol together with a trace of an unsaturated (probably terpene) alcohol. The ester fraction which does not react with phthalic anhydride is freed from traces of ketones by the action of semicarbazide acetate, and on hydrolysis yields a mixture of benzoic and salicylic acids (8:1) and benzyl alcohol, together with a trace of an unsaturated terpene alcohol. The extract has the composition: eugenol 30%; phenylethyl alcohol, 7%; benzyl benzoate, 40%; benzyl salicylate, 5%; methyl salicylate, 1%; unidentified material, 17%.

J. W. BAKER.

PATENTS.

Production of sterols. SOC. CHEM. IND. IN BASLE (B.P. 292,133, 14.6.28. Switz., 14.6.27).—A high yield of very pure sterols, particularly ergosterol, is obtained by heating yeast (or other fungi) under pressure (at about 120°) with an aqueous solution of an alkali, e.g., 2 pts. of potassium hydroxide to 1 pt. of water, and separating the sterols by skimming and extraction with a suitable solvent (e.g., ether) from which they crystallise on cooling. E. LEWKOWITSCH.

Manufacture of 3-nitro-2-aminopyridine-5-sulphonic acid. DEUTS. GOLD- & SILBER-SCHNEIDANST. VORM. ROESSLER (Austr. P. 107,595, 20.1.26).—2-Aminopyridine-5-sulphonic acid is nitrated with mixed acid in the cold to give 3-nitro-2-aminopyridine-5-sulphonic acid, decomp. 260°, which has therapeutic value. C. HOLLINS.

Manufacture of camphor from borneols. H. GAMMAY (U.S.P. 1,710,573, 23.4.29. Appl., 24.12.27. Ger., 30.12.26).—Borneols are heated in presence of graphite and an alkali or alkaline-earth compound, e.g., calcium hydroxide. R. BRIGHTMAN.

Extraction of nicotine from tobacco and tobacco waste. C. KINDSTRÖM (B.P. 315,157, 13.10.28).—Tobacco or tobacco waste is pulverised, mixed in the dry state with unslaked lime, and warmed; superheated steam is applied to slake the lime and to carry off the vapours, from which the nicotine is recovered.

E. LEWKOWITSCH.

Manufacture of tubercle waxes free from any tubercle bacilli and from tuberculin. I. G. FARFENIND. A.-G. (B.P. 287,941, 30.3.28. Ger., 30.3.27).—Crude tubercle wax is digested on the water-bath for a prolonged time with a dilute acid (e.g., 5% hydrochloric acid) to remove the greater part of the tubercle bacilli and fatty matter, the wax is separated, digested with warm water, dissolved in ether, and precipitated by acetone or alcohol. E. LEWKOWITSCH.

Manufacture of hydrogenated [cinchona] alkaloïds. J. W. BLAGDEN, Assr. to HOWARDS & SONS, LTD. (U.S.P. 1,720,462, 9.7.29. Appl., 20.3.26. U.K., 24.3.25).—See B.P. 250,380; B., 1926, 565.

Removing nicotine from tobacco. H. FEDERMANN (U.S.P. 1,719,291, 2.7.29. Appl., 23.2.28. Ger., 10.7.26).—See B.P. 302,560; B., 1929, 149.

Hydroxypyridine compounds (B.P. 288,628). **N-Aminoalkylaminobenzaldehydes** (B.P. 288,972).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Does fixation reduce the density of the silver deposit? L. P. CLERC (Bull. Soc. franç. Phot., 1929, 16, 142—143).—Normal fixation in either a neutral or an acid fixing bath is shown to have no effect on the density of the silver deposit. J. W. GLASSETT.

PATENTS.

Use of cellulose films in photography and kinematography. L. W. OLIVER and W. T. L. BECKER (B.P. 312,656, 17.2.28).—The buckling arising in the preparation of composite colour prints due to shrinkage on drying of the cellophane or like materials is avoided by arranging the component photographs on either side of a thin sheet of non-permeable celluloid, contact being secured by a dilute gelatin solution.

J. W. GLASSETT.

Hardened gelatin layers, more especially for imbibition printing. TECHNICOLOUR MOTION PICTURE CORP., Assees. of J. F. KIENNINGER (B.P. 288,146, 26.3.28. U.S., 1.4.27).—The hardening action produced in gelatin sols by the addition of an alkali dichromate together with an acid activating agent, such as an alkali sulphite or bisulphite, is controlled by the addition of a suitable quantity of a volatile base, such as ammonia, which is gradually expelled during the heating and drying operations, thus permitting the hardening agents to become effective.

J. W. GLASSETT.

Manufacture of photographic anti-halation coatings. I. G. FARBENIND. A.-G. (B.P. 294,104, 29.6.28. Ger., 16.7.27).—One or both sides of the photographic support are coated with a solution of a colloid containing a mixture of dyes so selected that the spectral absorption gap of one corresponds with the spectral absorption maximum of one or more of the others, thus giving total absorption. Examples are malachite-green with acid-magenta, or manganese dioxide with washing-blue. The dyes are bleached during the production of the negative, but if a coloured negative is required a suitable stable dye is admixed. J. W. GLASSETT.

Manufacture of positive light-sensitive paper. ZAIDAN HOJIN RIKAGAKU KENKYUJO (B.P. 290,565, 27.4.28. Jap., 14.5.27).—Paper is coated with an aqueous solution of a complex zinc *p*-diazodiphenylamine salt and a mineral or organic acid, with or without the addition of a coupling component (H-acid). A blue positive is obtainable by direct printing (or on development).

C. HOLLINS.

Photographic desensitisers. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 314,144, 3.4.28).—Sulphonated or carboxylated anthraquinone or phenanthraquinone derivatives are employed as desensitisers enabling panchromatic films to be developed in red light. Examples are sodium 1-nitroanthraquinone-2-carboxylate (0.25%), alizarin-direct-blue (0.1%), anthraquinone-violet (0.1%), and sodium anthraquinone-2-sulphonate (0.03%).

C. HOLLINS.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Ammonium nitrate explosive. J. A. WYLER, Assr. to TROJAN POWDER CO. (U.S.P. 1,720,459, 9.7.29. Appl., 23.3.26).—An explosive comprising ammonium nitrate and hexamethylenetetramine is claimed.

Non-corrosive priming mixture. J. D. McNUTT, Assr. to WINCHESTER REPEATING ARMS CO. (U.S.P. 1,718,370, 25.6.39. Appl., 13.7.27).—The mixture contains a metallic dioxide, a metallic silicide, and a metallic fulminate.

S. BINNING.

Treatment of mercury fulminate. J. D. McNUTT, Assr. to WINCHESTER REPEATING ARMS CO. (U.S.P. 1,718,371, 25.6.29. Appl., 13.7.28).—Mercury fulminate is freed from metallic mercury by heating.

S. BINNING.

Explosive projectile for infantry rifles and quick-firing small arms. O. MATTER (B.P. 287,906, 26.3.28. Ger., 29.3.27).—The projectile has in its nose an initiator of mercury fulminate or heavy metal azide and behind the initiator an explosive charge of a detonating substance such as trinitrotoluene, tetranitromethylaniline, pentaerythritol tetranitrate, or mixtures of these. The detonating charge may be compressed.

S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Composition and decomposition of fresh sewage solids collected from different parts of a settling tank. H. HEUKELEKIAN (Sewage Works J., 1928, 1, 19—30).—Determinations of fat, nitrogen, and gas production indicated that the total yield of gas is not correlated with the fat content of the sludge.

CHEMICAL ABSTRACTS.

Pyrethrin I and II. TATTERSFIELD and HOBSON.—See XVI.

PATENTS.

Germicidal compound. J. J. GOODWIN, Assr. to GOODWIN LABS., INC. (U.S.P. 1,696,762, 25.12.28. Appl., 22.11.27).—An aqueous solution of, e.g., 40% of coconut oil soap and 60% of trisodium phosphate, or other sodium salt hydrolysed in water, is used as germicide. Acid-fuchsin, for instance, may be added as indicator.

R. BRIGHTMAN.

Respiratory apparatus. DEUTS. GASLÜHLICHT-AUER-GES.M.B.H., and HANSEATISCHE APPARATEBAU GES. (B.P. 297,808, 4.4.28. Ger., 29.9.27).

Respiration devices. E. BOLDT (B.P. 315,091, 4.7.28. Addn. to B.P. 262,300).

Pest-destroying agents (B.P. 313,934)—See III.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 20, 1929.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Furnaces. J. F. O. STRATTON (U.S.P. 1,713,566, 21.5.29. Appl., 28.8.25).—In a furnace in which comminuted fuel slips down the imperforate sloping walls of a combustion chamber which converge to an oscillating air-inlet grate at the bottom or throat, the forced draught to the grate is first caused to pass over the outside of the walls of the throat to cool it. The upward air currents are adjusted to keep the lighter particles of coal (mostly coked) in suspension while the heavier fused particles of ash drop through the grate.

B. M. VENABLES.

Furnaces and furnace grates. T. O. WILTON, and CHEM. ENG. & WILTON'S PATENT FURNACE CO., LTD. (B.P. 313,380, 19.9.28).—An inclined grate is provided through which air may be forced in at the back of the grate of a Lancashire boiler or the like, so as to produce a swirling motion of the partly-burnt gases in the neighbourhood of the white-hot firebridge. A method of construction is described by which standard parts may be used for several different sizes of furnace.

B. M. VENABLES.

Soaking-pit furnaces. F. W. MANKER, ASSR. to SURFACE COMBUSTION CO., INC. (U.S.P. 1,711,273—4, 30.4.29. Appl., [A] 15.7.25, [B] 17.11.27).—(A) A furnace of the pit type has tapering combustion chambers in opposite walls, with gas burners situated in the smaller parts; the products of combustion are exhausted through passages situated below each combustion chamber. (B) A number of soaking pits are arranged side by side, and each has independent firing and exhausting means, but passages are provided between adjacent pits, so that products of combustion can be passed from one pit to another.

B. M. VENABLES.

Heat exchangers. SUPERHEATER CO., LTD. FROM COMP. DES SURCHAUFFEURS SOC. ANON. (B.P. 312,470, 4.5.28).—This invention may be applied to the heating of feed water by live steam. The feed water (or other fluid to be heated) is contained in a drum which also contains one or more heating units comprising superposed inlet and outlet headers connected by U-shaped heating tubes which fall all the way, and the headers are also connected directly by a smaller drainage hole or passage; the result is that condensate is cleared so rapidly that the heating agent may be live steam taken from a boiler and circulated back to the same boiler.

B. M. VENABLES.

Heat-exchange apparatus. G. D. MANTLE, ASSR. to MANTLE ENGINEERING CO. (U.S.P. 1,712,892, 14.5.29. Appl., 9.4.26).—The fluid to be heated flows in passages transverse to the flow of heating gases in a zig-zag

manner, the resultant general direction being counter-current. The fluid to be heated is, at entry, divided into two streams each controlled by a valve and flowing in separate parallel passages to the exit, where they recombine. At the hot end one of the streams makes additional transverse passes, and this stream only is subjected to the highest temperature.

B. M. VENABLES.

Heat-exchange apparatus comprising [brass] tubes having an electrolytic [chromium] deposit on the interior surface. F. G. MARTIN and W. RAMSAY (B.P. 299,100, 22.4.27).—The corrosion of brass condenser tubes is prevented by coating them internally and externally with electrolytically deposited chromium.

A. R. POWELL.

Heat-exchanging devices. C. MCNEIL (B.P. 313,780, 11.7.28).—The inner fluid passes through a bundle of tubes which is rotated in a fixed drum containing the outer fluid. The headers for the tubes are radial and are connected to hollow hubs and trunnions forming the inlet and outlet. The outlet hub is divided into compartments, one for each spoke or radial header, to prevent undue choking by the outlet fluid, which may be condensed steam.

B. M. VENABLES.

Heat-transferring devices, such as air preheaters. W. A. WHITE (B.P. 313,998, 21.3.28).—The preheater described is built up of unit cells formed from corrugated plates, the heating and heated gases passing at right angles through the cells.

B. M. VENABLES.

Heat-treatment apparatus. A. VUILLEUMIER, ASSR. to NEW DEPARTURE MANUF. CO. (U.S.P. 1,715,317, 28.5.29. Appl., 7.5.27).—The chamber or mixer for holding the articles to be treated is shaped as a wide-mouthed beaker flask and is rotated about a normally horizontal axis, but is preferably tiltable together with the surrounding heating chamber to discharge the articles. The heating chamber is preferably closed and electrically heated.

B. M. VENABLES.

Dryer. D. E. WASHINGTON, ASSR. to W. B. CONNELL (U.S.P. 1,715,729, 4.6.29. Appl., 25.7.27).—A revolving drum for the material is provided with an interior heating barrel which is revolved independently.

B. M. VENABLES.

Material dryer. M. MADSEN, ASSR. to MADSEN IRON WORKS (U.S.P. 1,715,852, 4.6.29. Appl., 11.10.26).—A rotating-cylinder dryer is provided with a hopper and stack at the feed end, with a stationary cowl surrounding the drum at the other end, and a stationary firebox (within the drum) in the form of a brick-lined cylinder with open ends. Fluid fuel is supplied through the closed end of the cowl to the interior of the firebox, and the air for combustion is preheated in the space

between the cowl and the drum and firebox. The material discharges through the space between the firebox and the drum, to which is connected a shoot extending through the cowl. B. M. VENABLES.

Desiccating apparatus. B. BOUDA and G. G. SKELTON, Assrs. to COLLIS CO. (U.S.P. 1,717,491, 18.6.29. Appl., 23.11.27).—A rotating heated drum is provided with a scraper bar bearing heavily on it and reciprocated in a direction parallel to the axis of the drum.

B. M. VENABLES.

Drying system for use with annular kilns. O. BUSCH (U.S.P. 1,718,190, 18.6.29. Appl., 14.5.27. Ger., 21.9.26).—A number of parallel drying chambers are arranged at opposite sides of the kiln, and are provided with upper and lower series of flues, so connected that combustion gases and hot air from the kiln may be used for direct and indirect heating of the chambers, and to permit moist air to be returned to the flues for admixture with fresh air. B. M. VENABLES.

Extraction of moisture from air or other gases. A. E. SHERMAN and I. MAYNARD (B.P. 314,592, 15.10.28).—The drying medium, *e.g.*, calcium chloride, is used in two stages, the incoming gas passing first in contact with partially spent calcium chloride liquor (which is used till it is completely spent) and finally in contact with solid calcium chloride, the liquor dripping from which is used in the first stage. A suitable apparatus is described, including light-weight flap valves to close automatically the final drying chamber when not in use.

B. M. VENABLES.

Pulveriser. A. F. THOMPSON (U.S.P. 1,715,724, 4.6.29. Appl., 13.6.27).—In a stamp mill constructed so as to be light and easily dismantled, and not to need much water, there are no dies, and the square shoes of the stamps are close together and operate directly on a floor plate that slopes downwards from the feed end. A cover plate is provided for the mortar box; the stamps are spring-loaded and a special form of lifting cam is described. B. M. VENABLES.

Apparatus for pulverising materials. W. T. DOYLE, Assr. to STURTEVANT MILL CO. (U.S.P. 1,715,123, 28.5.29. Appl., 1.10.28).—In a closed-circuit grinding system the net feed is caused to vary inversely as the circulating feed to compensate for variation in the friability of the material. B. M. VENABLES.

Grinding plate for attrition mills. W. H. MECHLIN, Assr. to BAUER BROS. CO. (U.S.P. 1,715,772, 4.6.29. Appl., 26.9.27).—The segmental grinding plates are held in the end of the mill by bolts the heads of which are in pockets in the back of the plates, with openings to the periphery for insertion of the bolts. B. M. VENABLES.

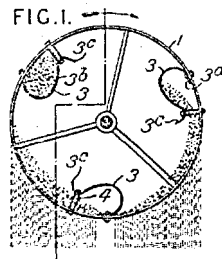
Mixing apparatus. R. W. SHAFOR, Assr. to GILCHRIST & CO. (U.S.P. 1,701,164, 5.2.29. Appl., 13.2.25).—The liquids or substances to be mixed are circulated in a closed system, *e.g.*, by propeller stirring, centrifugal pump, etc., through a series of baffle plates arranged to divide the stream of liquid into a series of channels, the separate streams being further divided and intermingled, and finally united in a common stream. The process may be continuous or discontinuous and the direction of flow

changed repeatedly in the operations. Several such mixing tanks may be arranged in series. R. BRIGHTMAN.

Mixing apparatus. J. M. BRINKLEY (U.S.P. 1,708,493, 9.4.29. Appl., 29.5.26).—A rotatable shaft projects from the lid of the apparatus, and a head bar, adjustable horizontally and vertically in relation to the shaft, carries a mixing frame having a pair of diamond-shaped side blades connected by a triangular bottom blade. W. G. CAREY.

Mixing machine. W. B. RANNEY, Assr. to W. WRIGLEY, JUN., Co. (U.S.P. 1,713,554, 21.5.29. Appl., 2.8.26).—An elongated open receptacle is formed from a number of parallel transverse troughs in each of which is rotated a mixing blade of distorted Z-shape; the mixers run at equal speeds, but in opposite directions in alternate compartments. The feed is at one end and outlet at the other, but no other means is provided for creating longitudinal flow, so that the motion is rapidly undulating, with very slow longitudinal travel when used continuously. B. M. VENABLES.

Screening or sifting devices. A. S. D. CRONE (B.P. 302,829, 2.2.28).—The interior of a rotary drum screen or trommel is provided with a number of scoops,



so that the descending lower quarter of the circumference is effective for screening, as well as the ascending quarter. B. M. VENABLES.

Screening of materials. WOODALL-DUCKHAM (1920), LTD., and S. SYMINGTON (B.P. 312,743, 26.3.28).—The screening surface comprises alternate sections of fixed spaced bars and rotating fingers; the latter lift the material from the set of fixed bars above and deposit on the set of fixed bars below. B. M. VENABLES.

Centrifugal machine. C. V. BOUILLON (U.S.P. 1,717,753, 18.6.29. Appl., 14.7.28. Fr., 6.6.28).—A centrifugal separator which is provided with continuous decanting means for muddy liquid, comprising a number of tapered chambers with an orifice in the apex of each, has a revolving eccentric blade inside each orifice, the blades being pressed towards the orifice by centrifugal force and rotated by gearing while the separator is spinning. B. M. VENABLES.

Reduction of froth formation at centrifugal liquid separators etc. G. W. RUDA, Assr. to AKTIEBOLAGET BALTIC (U.S.P. 1,718,081, 18.6.29. Appl., 24.9.27. Swed., 25.8.27).—The separated liquid flows over a weir in the neck of the bowl into a circumferential groove which is provided with a dam to cause the out-flowing liquid to flow the opposite way round to the rotation, towards the final outlet. The weir extends

round the whole circumference except at the final outlet.

B. M. VENABLES.

Separation of comminuted matter from the liquid in which it is immersed. [Thickener.] C. H. NORDELL (U.S.P. 1,718,871, 25.6.29. Appl., 19.8.26).—The feed pulp is caused to flow, with as little disturbance as possible, inwardly through a number of superposed shallow circular cells, clear liquid being exhausted from the centre and the settled sludge moved outwards mechanically.

B. M. VENABLES.

Gas-washing apparatus. H. A. BRASSERT, C. E. DOUGAN, and E. VON MALTITZ, Assrs. to H. A. BRASSERT & Co. (U.S.P. 1,707,548, 2.4.29. Appl., 9.3.27).—The apparatus comprises a series of washing chambers supplied with sprays for injecting washing liquid and arranged in a casing between two horizontal diaphragms one above the other. The diaphragms extend inwards about halfway across the diameter of the casing, and are midway between two circular discs supported by a central shaft and extending outwards from the shaft to a point about midway between the walls and the inner ends of the diaphragms. The space between discs and diaphragms is closed by annular rows of interacting disintegrator elements which break up the gas stream between the various compartments.

A. R. POWELL.

Washing flue gases. J. T. BARON and J. B. CLARKE (B.P. 314,110, 22.3.28).—A chimney for the washing and discharge of the whole of the flue gases flowing without mechanically induced draught is constructed so that the flue gases enter sideways part way up; above that point are situated a number of stories of mist-producing sprinklers, each story offset to the next, the grit and water (or other fluid) being carried straight down past the inlet to the lower part of the chimney which is adapted to collect the mud produced.

B. M. VENABLES.

Air filter. L. L. DOLLINGER (U.S.P. 1,714,854, 28.5.29. Appl., 21.3.27).—A number of filtering units are suspended below holes formed in a plate within the upper part of a casing which converges above the plate to form a single outlet. The filter units consist of perforated plates or wire mesh pleated into cylinders the walls of which are in the form of radial pockets. The mesh work is covered by filter medium and the flow of air is inwards, the lower ends of the cylinders being closed by plates which also secure the filters to the plate above by means of V-brackets and screwed rods within.

B. M. VENABLES.

Preparation and consumption of compressed gases. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 287,486, 20.3.28. Ger., 21.3.27).—A vehicle for the transport of liquefied gases is provided with a compressor, an expansion machine, a liquefier, and a vaporiser. One use of the apparatus is that when the material, e.g., oxygen, is to be delivered in a gaseous form additional liquid oxygen may be made from the atmosphere.

B. M. VENABLES.

Rotary chemical furnace. L. TOCCO and M. LANDI, Assrs. to FOUR CHIMIQUE ROTATIF SOC. ANON. (U.S.P. 1,708,526, 9.4.29. Appl., 22.6.26. Italy, 26.6.25).—See B.P. 278,774; B., 1927, 927.

II.—FUEL; GAS; TAR; MINERAL OILS.

Material and morphological behaviour of lignin-rich conifer tissues in the formation of forest humus and brown coal. W. GROSSKOPF (Brennstoff-Chem., 1929, 10, 161—167, 213—217; cf. B., 1926, 939).—The chemical and morphological changes accompanying the transformation of pine needles and pine wood into peat, lignite, and brown coal have been studied. All conifer needles possess a high lignin content, usually greater than 35% of the cell-wall material. In the transformation of pine needles into peat a close relationship exists between the rate of disappearance of the lignin and the rate of appearance of the humus. The cellulose:lignin ratio in the plant tissue gives a measure of its ease of decomposition. Morphological examination of decomposing pine needles shows that the parenchyma rich in cellulose is more rapidly destroyed than are the more highly lignified tissues. In the transformation of conifer wood into peat and brown coal the autumn wood presents a greater resistance to decomposition than the spring wood; this, however, is due, not to its greater lignin content, but probably to the different distribution of lignin within the tissue. Chemical examination of samples of pine-wood peat and lignite shows that, compared with the unchanged wood, the changes in composition are greater than is apparent from the morphological examination. Thus the pinewood peat contains only half the cellulose (24%) of the undecomposed wood and 20% of humus, whilst the Senftenberg lignite contains only 5% of cellulose and 70% of humus. The excess of the decrease in the cellulose over the increase in the humus is proof of the complete disappearance of cellulose in these decomposition processes. Attention is directed to the probable importance of conifer needles as the starting material from which brown coals have been formed.

A. B. MANNING.

Reactivity of coke. D. J. W. KREULEN (Brennstoff-Chem., 1929, 10, 128—131, 148—153, 168).—The reactivity is determined by passing air at a rate of 20 litres/hr. over the powdered coke contained in a platinum boat within an electrically heated furnace, and measuring the rate of formation of carbon dioxide. After placing the boat containing the sample in the furnace the air current is passed until temperature equilibrium is attained (2 min.) and the exit gases are then passed for a definite time (1—5 min.) through a weighed absorption tower charged with potassium hydroxide. The reactivity is expressed as mg. of carbon dioxide per sq. cm. of coke surface per 5 min. Taking the same total volume of material each time, and using sugar charcoal, the reactivity was found to be independent of particle size. Experiments carried out with four different cokes and with different lengths of coke surface in the boat showed that the alteration in the surface during the determination did not appreciably affect the relative reactivities. A length of 0.5 cm. was taken as standard. At 600° the reactivities of a number of cokes varied from 17 for a foundry coke and 22 for a blast-furnace coke to 125 for a coke made from a Polish flaming gas coal. At 700° these differences became smaller, and above 800° all the cokes gave the same value for the reactivity. Graphite and diamond

possessed relatively low reactivities. A study of the influence of catalysts on the reactivity and the form of the reactivity-temperature curve confirms the assumption that the true reactivity of the material is being determined, independently of its relative surface area. The reactivity as determined by the passage of the air current through a column of the coke involves the surface area as well as the actual reactivity; it is suggested, therefore, that a comparison of the results of the two methods should give a means of determining the relative surface areas of different cokes. By determining the reactivities at temperatures below 500° and by extrapolating to zero reactivity the ignition temperatures of the cokes may be determined. The slope of the curve, however, often renders accurate extrapolation difficult, and it is suggested that the relative ignition temperatures be taken as those at which the reactivity possesses a small definite value, *e.g.*, 10 mg./cm.²/5 min. These values varied from 338° for a brown-coal coke to 730° for graphite. A. B. MANNING.

Reactivity of coke. F. G. HOFFMANN (Brennstoff-Chem., 1929, 10, 287—288; cf. Kreulen, preceding abstract).—Kreulen's assumption that the rate of reaction measured in his experiments is independent of the particle size of the coke is criticised. A. B. MANNING.

Reactivity of coke. D. J. W. KREULEN (Brennstoff-Chem., 1929, 10, 288—289; cf. preceding abstract).—Further experiments are quoted to show that the influence of particle size on the rate of reaction, if appreciable at all, is very small. In comparing reactivities, however, it is agreed that coke of a definitely limited range of size should be chosen. A. B. MANNING.

Reducing power of brown-coal coke. F. SEIDENSCHNUR and A. JÄPPELT (Brennstoff-Chem., 1929, 10, 195—198).—A current of carbon dioxide at 5 litres/hr. was passed through a 5-cm. layer of the coke (2—3 mm. size) in an electrically heated porcelain tube and the exit gases were analysed. The results are shown as curves in which the volume of carbon monoxide produced from 100 vols. of carbon dioxide is plotted against the temperature, which ranged from 400° to 1000°. Low-temperature cokes from brown coal possessed a greater reducing power than either wood charcoal or active charcoal, which in turn were more reactive than cokes from a bituminous coal. The reducing power of the brown-coal cokes fell off as the temperature of carbonisation was raised. Treatment with methane at 800° or 1030° reduced the reactivity of the brown-coal cokes, as did also extraction of the ash with hydrochloric acid. A. B. MANNING.

Practical apparatus for Fischer's coking test. O. KELLER (Chem.-Ztg., 1929, 53, 517).—The apparatus described by Schaefer for the determination of water in coals (B., 1928, 557) may be used for the determination of gas, water, tar, and coke in bituminous coals by providing it with a side tube terminating in a flask with side tube for removing the gas. The tar collects in the side tube of the heating apparatus and the water in the flask; the sum of the two is obtained by noting the increase in weight of the tube and flask, and the water is then determined by the xylene method of Schaefer. A. R. POWELL.

Retardation of carbonisation in coke-oven flues. C. A. BAGLIN (Gas World, 1929, 90, Coking Sect., 58—59).—The combustion of coal gas with a normal excess of air gives a short flame which delivers more heat to the lower part of the charge. The speed of combustion in the flues may be reduced by diluting the combustion air with products of combustion, thereby obtaining a long flame; various methods of diluting the combustion air are discussed and criticised, and experiments are described whereby products of combustion are introduced into reversible hairpin flues in which combustion takes place in two stages. C. B. MARSON.

Manufacture of domestic coke in the coke oven. ANON. (Gas World, 1929, 90, Coking Sect., 54—57).—Factors which influence the reactivity of coke are discussed. Highly reactive cokes are obtained by the low-temperature carbonisation of any coal carbonised below, say, 700° and by the high-temperature carbonisation of non-coking or poorly coking coals; fuels possessing the property of high reactivity do not become plastic during carbonisation. It is impossible to carbonise coals in the coke oven which do not become plastic because the resulting coke cannot be discharged, but coking and non-coking coals can be blended so that a coke is produced which is sufficiently strong to be discharged and robust enough to withstand transport conditions. Similar results may be obtained by (a) pre-oxidation of a portion of the coal before coking, (b) the coking of low-temperature coke with coking coal, and (c) the addition of mineral substances such as iron oxide and lime to the coal before carbonisation. The combustibility of coke is greatly reduced if the coke is heated to too high a temperature or if it is exposed unduly to a moderate temperature. It is suggested that the best conditions for the manufacture of a coke for domestic use are (i) treatment of the coal before carbonisation by one of the methods described above, (ii) manufacture of the coke in an oven in which the carbonisation is completed simultaneously at the top and bottom of the charge, and (iii) pushing of the charge with the centre slightly "green," so that overheating is avoided and a little volatile matter is retained in the coke. C. B. MARSON.

Activation of carbon by means of steam. P. P. KOSAKEVITSCH and N. A. ISMAILOV (Kolloid-Z., 1929, 48, 241—246).—A method is described for activating carbon for adsorption purposes by passing superheated steam over the carbon, which is heated in a porcelain tube in an electrical resistance furnace. Examination of the influence of varying the conditions of activation shows that between 700° and 800° the activity of the carbon increases with the duration of activation and falls when the velocity of the current of steam is reduced. At 850° the activity reaches a maximum after activation for 15 min., and subsequently falls off rapidly; the appearance of the maximum is delayed by reducing the rate of the current of steam; the maximum is even more strongly marked at 900°. As a measure of activity, the adsorption of phenol from a 1% aqueous solution was taken; similar results were obtained when a basic substance (ethylamine) was used for adsorption. Some experiments were also carried out on the influence

of various common constituents of the ash of charcoal on the activity. This was effected by impregnating the charcoal with salts of iron, aluminium, or calcium, precipitating the hydroxides by means of ammonia, and heating the products before activation by means of steam. Calcium oxide was found to increase the activity of the carbon, ferric oxide diminished the activity, whilst the effect of aluminium oxide was irregular.

E. S. HEDGES.

Products of combustion from typical gas appliances. IV. 21ST REP. OF JOINT RES. COMM., INST. GAS ENG. AND UNIV. LEEDS (Gas J., 1929, 186, 740—744; cf. B., 1928, 557).—A number of tests were made with a 2½-gal. geyser, a surface-combustion grill, and a grill and ring-burner of a typical domestic cooker. It is shown that (a) the permissible gas rate increases as the flue length of a geyser is increased, and it is therefore recommended that the baffle should be placed as near as practicable to the ceiling; (b) causes tending towards a high carbon monoxide production with the geyser examined are: (1) smothering of the outer ring of flames if too short a flue pipe is used, and (2) the flames becoming ragged and blowing out intermittently (due to excessive gas pressure and rate on individual burners, together with excessive velocity of air and products through the combustion chambers); (c) production of carbon monoxide from a surface-combustion grill is remarkably low; (d) a typical domestic grill, having a single central burner and fixed iron frets, produces larger quantities of carbon monoxide, but the quantity produced can be materially lessened by raising the frets; carbon monoxide production is greatly increased by immersing a solid substance in a flame; (e) a drilled burner, when employed for heating a pan of water, and if not used above its normal gas rate, does not produce carbon monoxide in any harmful amount; this is probably because the considerable distance between the pan and the burner head allows adequate admixture with secondary air, while only the tips of the flames are chilled by the pans; (f) disc burners, under similar conditions, give relatively high carbon monoxide production (but the quantity is not influenced so greatly by the gas rate as with the drilled burners); this may be due to the greater chilling of the flame by the vessel or to the less favourable conditions for secondary aeration offered by a thin film of flame exposed to the air on one side only. It is concluded that the carbon monoxide production of a disc burner may be kept reasonably low by paying careful attention to details of design, without interfering with its higher thermal efficiency (compared with a drilled burner).

C. B. MARSON.

Purification [of coal gas from hydrogen sulphide]. G. C. PEARSON (Gas J., 1929, 186, 797—807).—An historical survey is made of the development of the process for the removal of hydrogen sulphide from crude gas by means of iron oxide in box purifiers, and the design and layout of a typical plant are described in full detail. The use of various liquid purifying agents is discussed, and the results of a number of experiments are given.

C. B. MARSON.

Higher alcohols from petroleum hydrocarbons.

I. S. PILAT and J. WINKLER. II. S. PILAT, W. J. PIOTROWSKI, and J. WINKLER (Przemysł Chem., 1929, 13, 185—195, 208—220).—I. The highest yields of olefines, as well as of benzine, are obtained from the cracking of gasoline at 600—670°. The olefine gases are converted into a mixture of higher secondary alcohols, from which isopropyl, *sec.*-butyl, and *sec.*-amyl alcohols are isolated.

II. The gaseous products of the cracking of gasoline, using the Cross system, contain 10—12 vol.-% of olefines (propylene and homologues). In order to convert these olefines into alcohols, the gas must first be freed from water, sulphur, and gasoline. The first two objects are best achieved by passing the gas through two towers, the first of which is packed with a mixture of calcium oxide, sodium hydroxide, and wood shavings, whilst the second contains granular ferric hydroxide in place of the lime in the first tower. Gasoline is best removed by adsorption on active charcoal, which can, provided that the gas has previously been properly desulphuretted, be used repeatedly. Absorption of olefines increases with concentration of sulphuric acid, from 32% in 75% acid to 83.7% in 96% acid, but the yield of alcohol diminishes with increasing concentration of acid, from 84.2% of the theoretical yield using 75% acid to 36.3% using 96% acid, various oily highly polymerised products being obtained in the latter case. The best yields of alcohol are obtained using 80—85% sulphuric acid, which at 15—20° and 2 atm. absorbs about 70% of the olefines present. Absorption is more complete if for each volume of acid in the absorption towers 2 vols. of solar oil are present. The highest yields of alcohols are obtained by adding 1.5—2 vols. of water to the absorption acid, and then distilling until the residual acid is about 50%. This residual acid may be further concentrated to 80%, when it may, after being freed from sulphur dioxide, be again used for absorption. The distillate contains 35—40% of alcohols, and, after neutralisation with lime, gives on rectification 86—88% alcohol. Practically anhydrous alcohol is obtained from this distillate by saturation with sodium chloride, and this product may be further purified by acidifying with sulphuric acid and adding 1% of potassium permanganate, agitating, and alkalisng with sodium silicate, when the silica gel formed adsorbs most of the empyreumatic impurities present. The gasoline adsorbed on the charcoal during purification of the olefine gas amounts to about 200 g. per m.³ of gas, and consists of 56% of paraffin, 6% of aromatic, 2% of naphthene, and 35% of unsaturated hydrocarbons. The last-named fraction consists chiefly of amylenes, hexylenes, and heptylenes, and may be converted by the action of sulphuric acid into a mixture of isoamyl and higher alcohols. The residue may by appropriate rectification be converted into benzine containing only aliphatic hydrocarbons.

R. TRUSZKOWSKI.

Treatment of bitumens with "sulphosil." V. TOKMANOV (Nef. Choz., 1928, 15, 73—74).—Silica gel is used for the determination of neutral tars and soft asphalts, and is followed by treatment with "sulphosil" (activated silica gel), whereby the unsaturated compounds and aromatic substances are extracted, leaving paraffins and naphthenes.

CHEMICAL ABSTRACTS.

Bunsen flames of unusual structure. F. A. SMITH and S. F. PICKERING (Bur. Stand. J. Res., 1929, 3, 65—74).—See B., 1928, 880.

PATENTS.

Apparatus for dehydrating crude [petroleum] oils. W. L. PALMER, Assr. to REGAN FORGE & ENG. Co. (U.S.P. 1,696,859, 25.12.28. Appl., 31.1.25).—The oil, after removal of natural gas, is heated and sprayed into a horizontal, rotating, elongated cylinder. The water and other foreign substances are discharged by valve-controlled peripheral outlets, and the oils remaining in the central portion of the cylinder are sent to storage.

R. BRIGHTMAN.

Treating hydrocarbon oil. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,710,063, 23.4.29. Appl., 31.8.22. Renewed 13.9.26).—Cracked petroleum distillate is treated successively with sulphuric acid, sodium hydroxide solution, d 1.045—1.091, and sodium plumbite solution, and after distillation is mixed with 50—60% of refined bottoms or high-boiling residues and again distilled.

R. BRIGHTMAN.

Purification of hydrocarbons. J. C. BLACK and W. H. LOW, Assrs. to PAN AMER. PETROLEUM Co. (U.S.P. 1,696,377, 25.12.28. Appl., 17.3.27).—Hydrocarbons are purified (desulphurised) by counterflow treatment with cadmium hydroxide in presence of sodium hydroxide.

R. BRIGHTMAN.

Purifying petroleum oils. J. C. BLACK, W. D. RIAL, and R. T. HOWES, Assrs. to PAN AMER. PETROLEUM Co. (U.S.P. 1,709,203, 16.4.29. Appl., 26.4.26).—The oil is treated with dilute sodium hydroxide under pressure and agitation with steam, then with an alkaline-earth hydroxide and an adsorbent, agitated, and heated to 150°.

R. BRIGHTMAN.

Process of purifying hydrocarbons. J. C. BLACK and M. L. CHAPPELL, Assrs. to PAN AMER. PETROLEUM Co. (U.S.P. 1,710,205, 23.4.29. Appl., 28.5.27).—Petroleum distillate, *e.g.*, gasoline stock, preferably after treatment with sodium hydroxide or hydrochlorite, is cooled and treated with about 25% of liquid sulphur dioxide at 8°, then passed in countercurrent with fuming sulphuric acid (3—15% SO_3), and the remainder of the sulphur dioxide removed by heat, and cooled and compressed for re-use. Suitable heat-exchanging cycles are claimed.

R. BRIGHTMAN.

Removing sulphur compounds from petroleum oils. W. N. DAVIS and W. H. HAMPTON, Assrs. to STANDARD OIL Co. (U.S.P. 1,705,809, 19.3.29. Appl., 15.10.24. Renewed 2.6.28).—The oil is cooled by heat exchange with treated oil and treated at -10° to 5° in countercurrent with 15% fuming sulphuric acid.

R. BRIGHTMAN.

Treatment of residual [petroleum] oils. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,705,199, 12.3.29. Appl., 22.10.23).—Residual oil from cracked petroleum oil is withdrawn hot from the expansion chamber, agitated with sulphuric acid, and settled. The treated oil may be returned to the cracking still, the precipitated matter being used, *e.g.*, for asphalt.

R. BRIGHTMAN.

Cracking of [petroleum] oils. J. R. MARDICK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,706,629, 26.3.29. Appl., 5.5.23).—Petroleum oil, *e.g.*, gas oil, b.p. about 250°, is treated at about 300° with 0.25—1% of dry chlorine previously activated by electrical means and supplied under, *e.g.*, 5 lb./in.² Alternatively, petroleum oil may be refined with 0.1% of the activated chlorine at 60—75°.

R. BRIGHTMAN.

Treatment [cracking] of hydrocarbons. J. C. BLACK, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,706,396, 26.3.29. Appl., 12.8.25).—Light cracking oil, *e.g.*, gas oil, is passed through a series of heating coils after successive heat exchange with residuum from the evaporator and hot oil passing from the coils to the evaporator. The gas oil is raised to cracking temperature (400—480°) in the first two coils and is mixed with heavy cracking oil, also previously heated (320—400°), in primary and secondary coils, and passed into a final coil maintained at the mean temperature or above, the heat supplied in the secondary heaters balancing losses by radiation and absorption in the endothermic cracking reaction. From the third coil the oil passes through the heat exchanger to an evaporator the vapours from which pass up a fractionating tower, and the residuum is run into storage. Condensate from the fractionating tower passes through a steam still where heavy naphtha distils off, the residue being used to spray the fractionating tower or is returned to the heavy oil supply. Light naphtha vapours from the fractionating tower are condensed and collected in the usual way.

R. BRIGHTMAN.

Treatment of compounds preferably of a hydrocarbon nature. W. J. KNOX, Assr. to PETROLEUM CONVERSION CORP. (U.S.P. 1,715,239, 28.5.29. Appl., 9.9.22. Can., 24.8.22. Renewed 16.4.29).—After a partial fractional distillation, hydrocarbons are cracked by treatment with a highly heated gas containing hydrogen or a lower hydrocarbon, *e.g.*, methane. The hot cracked gases are used to effect the partial distillation by passing them in countercurrent to the incoming oils. The vapours thus distilled are passed in part to the cracking chamber, and in part to the heater, *e.g.*, a hot-blast stove, which supplies the hot gas for effecting the cracking process.

F. G. CLARKE.

Conversion [cracking] of petroleum oils. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,707,348, 2.4.29. Appl., 16.8.22. Renewed 2.6.27).—The oil is fed into the dephlegmator and passes out with the phlegms in countercurrent with the entrant vapours to a vaporising chamber containing a partition which prevents the reflux from mixing with the heated vaporising oil, and directs its flow through a header into the heating tubes, from which it returns through a second header to the other end of the vaporiser. The second header extends sufficiently far into the vaporiser to prevent carbon draining back into the heating zone.

R. BRIGHTMAN.

Separation of liquids [hydrocarbon oils] having different b.p. B. TORREY, JUN., and G. R. SANFORD, Assrs. to SEMET-SOLVAY Co. (U.S.P. 1,701,988, 12.2.29. Appl., 13.11.22).—Crude light oil is washed and distilled, preferably continuously with direct steam. The water-

white condensate, containing oils of lower b.p. than xylenes, is passed continuously through a series of dephlegmating columns such that in each column only the liquid of lowest b.p. is completely distilled off. The residual phlegm from each column, free from that product of lower b.p., is fed into the succeeding column. First runnings (benzene, toluene, and xylenes) are drawn off pure from successive condensates. Phlegms from the final (xylene) column are returned to the reservoir for high-boiling residues from the preliminary distillation.

R. BRIGHTMAN.

Fractional distillation [of hydrocarbon oils].

J. PRIMROSE, Assr. to FOSTER WHEELER CORP. (U.S.P. 1,707,369, 2.4.29. Appl., 30.1.24).—The oil is fed into a steam-heated storage tank and is heated progressively by circulation through a tubular heater, the oil and vapour returning to a separating tower, whence the vapours escape and are condensed in the usual manner, while the oil returns to the storage tank. Alternatively, the oil and vapours may be returned direct to the storage tank, the vapours escaping with evaporation vapours through a vapour column. R. BRIGHTMAN.

Distillation of mineral oil. A. E. PEW, JUN., Assr. to SUN OIL Co. (U.S.P. 1,707,448, 2.4.29. Appl., 22.12.26).—Water is de-aerated by evaporation and condensation in a high vacuum while flowing in shallow streams over the evaporator shelves, converted into steam in a boiler below atmospheric pressure, and injected into the oil, also under vacuum. The oil vapours and steam from the still pass into air-jacketed towers, the lubricating oil and lighter oil fractions being successively and separately condensed, while the uncondensed steam passes out to a barometric injection condenser and is condensed by injected water, water vapour and non-condensable still gases being ejected by a wet or dry vacuum pump. R. BRIGHTMAN.

Treatment of [mineral] oils for distillation. J. M. McCLAVE, Assr. to CONSERVO Co. (U.S.P. 1,703,158, 26.2.29. Appl., 8.12.25).—The crude oil is treated with manganese oxide (pyrolusite) and hydrochloric acid prior to and at the time of distillation.

R. BRIGHTMAN.

Refining of mineral oils. A. LACHMAN, Assr. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,709,315, 16.4.29. Appl., 8.12.26).—Vapours from the cracking still are passed upwards through a tower down which a stream of 85% phosphoric acid is directed by a spreader over brickwork or resistant material, the concentration of the acid being maintained constant by the injection of steam at the base of the tower. The gasoline vapour passes to a condenser, and after settling from entrained water is agitated for 5–10 min. with a solution containing 18% of sodium hydroxide and 9% of sodium hypochlorite. R. BRIGHTMAN.

Refining used [lubricating] oils. R. V. AYCOCK and W. D. HARRIS, Assrs. to REFINOIL MANUF. CORP. (U.S.P. 1,707,671, 2.4.29. Appl., 27.7.28).—Used mineral or lubricating oil is settled from water, agitated at 50° with about 2% of sulphuric acid, *d* 1.84, for 15–30 min., again settled, and drawn off from the resultant sludge to a still where it is heated to about 230° in a relatively high vacuum to vaporise diluents.

Fuller's earth (15–18%) is added to the heated oil so that the oil passes upwards through the reagent, which settles into a bed, through which the oil is finally filtered.

R. BRIGHTMAN.

Clarifying, decolorising, and neutralising oils.

D. R. MERRILL, Assr. to UNION OIL Co. OF CALIFORNIA (U.S.P. 1,695,198, 11.12.28. Appl., 1.2.26).—The oil, *e.g.*, petroleum lubricating oil, is agitated with a suspension of 4–20% of raw clay or mineral earth in about half its weight of water containing a strong mineral or organic acid in amount equal to 0.25% of the weight of oil treated, *e.g.*, at 160°, then, after separating the earth, with 4–16% of fresh clay and about 2–8% of a dilute alkali solution containing not more than about 0.5% of alkali on the oil treated, together with a salt of an acid affording an insoluble calcium salt, *e.g.*, oxalate or fluoride. R. BRIGHTMAN.

Process for treating emulsions. C. FISCHER, JUN., and W. J. REDDISH, Assrs. to KONTOL Co. (U.S.P. 1,710,159, 23.4.29. Appl., 2.8.26).—The emulsion is heated with "sludge layer mineral oil aluminium sulphionate" and sodium hydroxide. R. BRIGHTMAN.

Submerged-combustion process. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,716,433, 11.6.29. Appl., 17.3.25).—A pool of hydrocarbon oil is distilled by heat generated from the combustion of a hydrocarbon fuel within the pool. H. ROYAL-DAWSON.

Apparatus for recovering gasoline. H. B. BERNARD, Assr. to SINCLAIR OIL & GAS Co. (U.S.P. 1,713,323, 14.5.29. Appl., 14.11.24).—Vapours from a preliminary still are introduced under reduced pressure into the lower part of an exhausting tower, while liquid from the still is introduced into the upper part. The gases and vapours pass to a dephlegmator, where they are treated with a liquid absorbing medium.

H. S. GARLICK.

Manufacture of motor fuels and similar products. D. J. YOUNG (U.S.P. 1,706,686, 26.3.29. Appl., 13.4.27).

—Water-gas plant consisting of a fuel heating-chamber containing a bed of solid fuel, and a primary and secondary heat exchanger consisting of at least one shell containing checker brick is used. The fuel chamber is air-blasted, and the resulting heat stored by combustion in the primary heat exchanger and superheater to establish material temperature differences in the heat exchangers. The heated fuel bed is steam-blasted intermittently and reversely, and liquid hydrocarbons are admitted selectively at the top of the fuel chamber or at the top or bottom of the primary or secondary heat exchangers, the combustible gas being withdrawn through the fuel bed and passed through a scrubber or condenser. R. BRIGHTMAN.

Motor fuel and its manufacture. L. KIRSCH-BRAUN and C. B. BELKNAP (U.S.P. 1,707,019, 26.3.29. Appl., 13.1.20. Renewed 17.8.28).—A mixture of gasoline, kerosene, and gas oil, obtained, *e.g.*, by distilling crude petroleum until the gas oil has vaporised, is emulsified mechanically with, *e.g.*, 3–5% of water, in presence of a saturated cyclic hydrocarbon or crude naphthenic acid derivative as emulsifier, the hydrocarbon being in excess of 70% by vol. in the final product.

R. BRIGHTMAN.

Lubricating composition. G. W. GRAY, Assr. to TEXAS CO. (U.S.P. 1,716,310, 4.6.29. Appl., 8.6.27).—The rails of the curved sections of railway tracks are lubricated with a mixture which comprises approx. 60% of cylinder stock residuum and 40% of a residual oil from an asphalt-base crude petroleum.

H. S. GARLICK.

Pressure cracking treatment of liquid hydrocarbons. E. S. L. BEALE, G. H. COXON, and A. E. DUNSTAN, Assrs. to ANGLO-PERSIAN OIL CO., LTD. (U.S.P. 1,708,782, 9.4.29. Appl., 9.5.28. U.K., 15.3.27).—See B.P. 293,889; B., 1928, 701.

Apparatus for pulverising coal or other fuel. J. MULLIN (B.P. 313,245, 2.4.28).

Arrangement for admitting steam through the bottom cover of vertical-chamber ovens for gas manufacture. CHAMBER OVENS, LTD., Asses. of PINTSCH & DR. OTTO GES.M.B.H. (B.P. 289,058, 18.4.28. Ger., 21.4.27).

Apparatus for handling and quenching coke. R. DEMPSTER & SONS, LTD., and W. H. HANDLEY (B.P. 313,020, 3.2.28).

Installations for dry-cooling of incandescent coke. SULZER FRÈRES SOC. ANON. (B.P. 312,033, 17.9.28. Switz., 18.5.28).

Pulverised fuel burners. H. E. YARROW (B.P. 313,368, 24.8.28).

Burners for pulverised fuel. H. A. MARSTON (B.P. 312,972, 3.3.28. Addn. to B.P. 285,314).

Powdered fuel burners. H. E. HAZLEHURST and O. MARGETSON (B.P. 313,100, 7.12.27).

Liquid fuel burners. B. POWELL-BRETT (B.P. 313,262, 19.4.28).

Burner for liquid fuels. J. C. NILSEN (B.P. 313,830, 16.10.28).

Burners for powdered or other fuel. R. V. WHEELER and J. BRASS (B.P. 312,588, 23.2. and 5.4.28).

Liquid seals with special reference to gas producers or generators. HUMPHREYS & GLASGOW, LTD., and J. C. STELFOX (B.P. 313,754, 22.6.28).

Removal [by suction] of solid combustion residues from internal-combustion engines operated with pulverulent fuel. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,781, 13.7.28).

Production of acetylene and apparatus therefor. H. GEBHARDT (B.P. 313,669, 26.3.28).

III.—ORGANIC INTERMEDIATES.

Alcohols from petroleum hydrocarbons. PILAT and others.—See II.

PATENTS.

Continuous manufacture of vinyl esters. G. O. MORRISON, Assr. to CANADIAN ELECTRO PRODUCTS CO., LTD. (U.S.P. 1,710,181, 23.4.29. Appl., 8.11.26).—Acetylene is passed into an aliphatic carboxylic acid, e.g., acetic acid, in presence of a mercury orthophosphate. When the reaction slackens fresh catalyst is similarly prepared from mercuric oxide in, e.g., acetic

acid and molecular equivalents of orthophosphoric acid and acetylene are passed into it at, e.g., 78° until formation of vinyl acetate commences. The active catalyst mixture is then added to the reaction vessel and spent catalyst or sludge removed. R. BRIGHTMAN.

Manufacture of coloured polymerised styrene and its homologues. I. OSTROMISLENSKY, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,705,264, 12.3.29. Appl., 31.3.27).—Polymerised styrene is coloured with heavy metals in colloidal solution by forming a solution of the metallic salt (e.g., silver nitrate) in unpolymerised styrene containing 1–3% of polymerised styrene as protective colloid and polymerising at 140–180°.

R. BRIGHTMAN.

Reduction of aromatic nitro-compounds. I. G. FARBENIND. A.-G., and A. CARPMAEL (B.P. 314,573, 29.3.28. Addn. to B.P. 263,376; B., 1927, 156).—In the process of the prior patent a finely-divided oxide or hydroxide of a ter- or quadri-valent metal (aluminium, cerium, iron) is suspended in the reaction mixture.

C. HOLLINS.

Manufacture of phenol from chlorobenzene. I. G. FARBENIND. A.-G. (B.P. 288,308, 4.4.28. Ger., 6.4.27).—Chlorobenzene is boiled with aqueous alkali and the vapours of chlorobenzene and water are passed over active silica gel at 350°, condensed, and returned to the boiler until all chlorobenzene has been converted.

C. HOLLINS.

Manufacture of 4-amino-1-oxybenzene [*p*-aminophenol] and *N*-derivatives thereof. I. G. FARBENIND. A.-G. (B.P. 293,792, 23.6.28. Ger., 12.7.27).—Alkyl ethers of *p*-aminophenol are dealkylated by heating with 60–75% sulphuric acid. *N*-β-Hydroxyethyl-*p*-phenetidine at 160° gives *N*-β-hydroxyethyl-*p*-aminophenol, m.p. 96–97°; *diN*-(β-hydroxyethyl)-*p*-aminophenol, m.p. 140°, *p*-aminophenol, and *N*-methyl-*p*-aminophenol are similarly obtained from their methyl or ethyl ethers.

C. HOLLINS.

Production of salts of carboxylic acids. G. H. BUCHANAN, G. BARSKY, and K. D. ASHLEY, Assrs. to AMER. CYANAMID CO. (U.S.P. 1,717,353, 18.6.29. Appl., 18.12.23).—A solution containing mainly calcium cyanamide, obtained by treating a mixture of calcium cyanamide and cyanide with water, is digested under pressure at above 60°. The liberated ammonia is removed and the aqueous formate separated from the precipitated oxalates.

F. G. CLARKE.

Manufacture of oxygenated organic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,388, 24.2.28).—Liquid non-aromatic hydrocarbons, b.p. below 180°, with or without addition of a catalyst (manganese acetylacetonate), are passed in counter-current with a stream of air through a packed tube at about 145° and 20–50 atm. Hexane or benzene, b.p. 70°, gives fatty acids, C₂–C₈, and their esters.

C. HOLLINS.

Manufacture of condensation products from alicyclic ring-ketones. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 313,421, 7.2.28).—Arylamines having free *para*-position are condensed with cyclohexanones in presence of acid at 140–220°. The diarylcyclohexane so formed is converted into a hydro-

genated diphenyl derivative by heating alone or with an acid agent (sulphuric acid, zinc chloride). *cyclohexanone* and aniline yield (a) 1:1-*di-(p-aminophenyl)-cyclohexane*, m.p. 111°, b.p. 275—276°/12 mm., convertible into (b) 1-*p-aminophenylcyclohexane*, b.p. 170—171°/12 mm. Analogous compounds are obtained from *cyclohexanone* and *o-chloroaniline* (a, m.p. 126—128°; b, m.p. 32°, b.p. 196—198°/16 mm.), *o-toluidine* (a, m.p. 166°; b, b.p. 183—185°/15 mm.; *acetyl* derivative of b, m.p. 153°), *o-anisidine* (a, b.p. 289°/12 mm.; b, m.p. 50°, b.p. 185—187°/12 mm.), *ethyl-o-toluidine* (a, m.p. 118—120°; b, b.p. 180—185°/12 mm.), or *dimethylaniline* (a, m.p. 158—160°; b, b.p. 152—153°/3 mm.). 4-Methylcyclohexanone and *o-toluidine* yield 1:1-*di-(4-amino-3-methylphenyl)-4-methylcyclohexane*, m.p. 145°, and 1-(4-amino-3-methylphenyl)-4-methylcyclohexane, b.p. 194—196°/13 mm. (*acetyl* derivative, m.p. 163°).

C. HOLLINS.

Manufacture of condensation products from hydroxybenzenes [phenols] and hydroaromatic ring ketones [cyclohexanones]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 310,825, 28.1.28).—Phenol or *o-cresol* is condensed with *cyclohexanone* or an alkylcyclohexanone in presence of hydrogen halides in water or preferably in glacial acetic acid. 1":1"-*Di-(p-hydroxyphenyl)cyclohexane*, m.p. 186° (cf. Schmidlin and Lang, A., 1910, i, 836), and its 3:3'-*dimethyl* and 4"-*methyl* derivatives, m.p. 186° and 179°, respectively, are described.

C. HOLLINS.

Manufacture of hydrogenated hydroxy-derivatives of the diphenyl series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 310,832, 28.1.28. Cf. B.P. 310,825; preceding).—1":1"-*Di-(p-hydroxyphenyl)-cyclohexane* when heated, e.g., in a vacuum or with hydrogen chloride or zinc chloride at 200°, loses 1 mol. of phenol and yields 4'-hydroxy-2:3:4:5-tetrahydrodiphenyl [1-*p-hydroxyphenylcyclohexane*], m.p. about 124°, together with hydroxyhexahydrodiphenyl [*p-hydroxyphenylcyclohexane*], m.p. 128—129°, b.p. 180—210°, the latter being the main product when a catalyst is used. The condensation product from *o-cresol* and technical methylcyclohexanone behaves similarly.

C. HOLLINS.

Manufacture of aniline-2:5-disulphonic acid. I. G. FARBENIND. A.-G. (B.P. 285,488, 15.2.28. Ger., 18.2.27).—Metanilic acid is sulphonated with oleum at 160° and the resulting mixture of di- and tri-sulphonic acids is boiled with dilute sulphuric acid to yield pure aniline-2:5-disulphonic acid.

C. HOLLINS.

Manufacture of solid stable diazo compounds. KALLE & Co. A.-G. (B.P. 294,248, 16.7.28. Ger., 21.7.27).—Stable double salts are precipitated by addition of cadmium chloride, with or without sodium chloride, to diazo solutions, e.g., diazotised *p*-aminodimethylaniline or its *o*-carboxylic acid.

C. HOLLINS.

Manufacture of organic [quinoline] bases [from arylamines and acetylene]. I. G. FARBENIND. A.-G. (B.P. 296,423, 9.3.28. Ger., 2.9.27. Addn. to B.P. 283,163; B., 1929, 275).—Quinaldines are obtained by passing an arylamine vapour with acetylene at 300—350° over the catalysts of the prior patent, indoles being formed (in small amount) only when secondary

amines are used. Aniline gives quinaldine; *o*-toluidine, 2:8-dimethylquinoline; ethylaniline, 1-ethylquinaldine (*picrate*, m.p. 152°), quinaldine, and 1-ethylindole; and *o*-chloroaniline, 8-chloroquinaldine. C. HOLLINS.

Manufacture of condensation products of the benzodiazine [quinazoline] series. I. G. FARBENIND. A.-G. (B.P. 287,179, 16.3.28. Ger., 18.3.27).—2- or 4-Halogeno- or 2:4-dihalogeno-quinazolines are condensed with phenols or aromatic hydrocarbons in presence of aluminium chloride, or with halogenated hydrocarbons by means of sodium, or with a Grignard reagent. 2:4-*Di-(4'-hydroxy- α -naphthyl)quinazoline*, decomp. 240—250° without melting, and 4-(4'-hydroxy- α -naphthyl)quinazoline, m.p. 230—232°, are obtained from 2:4-dichloro- and 4-chloro-quinazoline, respectively, with α -naphthol and aluminium chloride. 4-Hydroxy-2-phenylquinazoline, m.p. 235°, from 2-chloro-4-hydroxyquinazoline, and 2-phenylquinazoline, m.p. 100—101°, b.p. 305°, are similarly prepared. 2-Chloro-6-nitro-4-hydroxyquinazoline, naphthalene, and aluminium chloride yield 6-nitro-4-hydroxy-2- α -naphthylquinazoline, reducible to an amine which when diazotised and coupled with G-acid gives a bluish-red dye. From 2-chloro-4-phenylquinazoline, chlorobenzene, and sodium, 2:4-diphenylquinazoline, m.p. 120—121°, is prepared; this is also obtainable from 2:4-dichloroquinazoline and magnesium phenyl bromide. 2-Phenyl-4-methylquinazoline results from the successive action of magnesium methyl and phenyl halides on 2:4-dichloroquinazoline. C. HOLLINS.

Manufacture of cyclic compounds containing aldehydic groups. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 311,208, 6.2.28).—Cyclic compounds having suitably reactive $\cdot\text{CH}\cdot$ groups, e.g., *m*-xylene, anthracene, ethylcarbazole, pyridine, quinoline, etc. are condensed at 15—80° in presence of phosphorus chlorides, sulphuryl chloride, thionyl chloride, or aluminium chloride, with formamide or its *N*-alkyl or -aryl derivatives. In the case of keto-enolic compounds when the formyl derivative of a secondary arylamine is used the enolic hydroxyl is at the same time replaced by chlorine. Examples are: anisaldehyde from anisole and *N*-methylformanilide; 2-ethoxy- α -naphthaldehyde; 4-methoxy- α -tetrahydro- α -naphthaldehyde, m.p. 59—60° (*aldazine*, m.p. 210—211°); the 2-methoxy-compound, m.p. 52—53° (*aldazine*, m.p. 237—238°), and 2-ethoxy-compound, m.p. 62—63° (*aldazine*, m.p. 215°); 9-*aldehydoanthracene*, m.p. 104°; 2:4-dimethylbenzaldehyde; 1(or 4)-chloro-9-*aldehydoanthracene*, m.p. 97—104°; 2-hydroxy- α -naphthaldehyde, m.p. 81—82° (oxime, m.p. 157°); the 4-hydroxy-compound; 2:7- and 4:8-*dihydroxy- α -naphthaldehydes*; 2-hydroxy-1-*aldehydo-3-naphthoic acid* and its *anilide* (*aldazines*, m.p. above 300°); 4-hydroxy-2:5-dimethylbenzaldehyde; β -resoreylaldehyde; vanillin; 6-chloro-2-*aldehydo-4-methylthioindoxyl* (*aldazine*, m.p. 277—278°); 6-ethoxy-2-*aldehydothioindoxyl* (*aldazine*, m.p. 248°) from 6-ethoxythioindoxyl and formamide, or the 3-chloro-compound, m.p. 166°, when methylformanilide is used; 2(?)*-aldehydo-N-ethylcarbazole*, m.p. 87°; 6-*aldehydonaphthastyril*, m.p. 223°; 3:6-dichloro-2-*aldehydo-4-methylthioindoxyl*; 9-chloro-10-*aldehydoanthracene*, m.p. 216°, from anthrone; the 2:9-dichloro- (m.p. 174°) and 1:5:9-trichloro- (m.p. 197°)

derivatives; 9-chloro-3:4-dimethoxy-10-aldehydoanthracene, m.p. 172°; 9-chloro-2:6-dimethoxy-10-aldehydoanthracene, m.p. 233°. C. HOLLINS.

Manufacture of 2:3-aminonaphthoic acid. I. G. FARBENIND. A.-G. (B.P. 284,998, 7.2.28. Ger., 7.2.27).—2:3-Hydroxynaphthoic acid (1 pt.) is added to molten zinc chloride-ammonia (2 pts.), or zinc chloride-ammonia is added to the molten acid, and the mixture is heated at 240–250° for 10–12 hrs. (Cf. Fierz and Tobler, A., 1922, i, 869.) C. HOLLINS.

Manufacture of compounds of the anthracene series [nitrotetrahydroanthraquinones]. I. G. FARBENIND. A.-G. (B.P. 295,943, 20.8.28. Ger., 18.8.27).—Tetrahydroanthraquinones are nitrated, chiefly in the *ar*- α -position, by nitric and sulphuric acids at 40–50°. The preparation of 5-nitro-1:2:3:4-tetrahydroanthraquinone, m.p. 192°, the 6-isomeride, m.p. 133–134°, 5-nitro-6-methyl-1:2:3:4-tetrahydroanthraquinone, m.p. 130°, and 5-nitro-8-acetamido-1:2:3:4-tetrahydroanthraquinone, decomp. 185°, is described. C. HOLLINS.

Manufacture of basic derivatives of anthraquinone [amination and alkylamination of hydroxylated anthraquinones]. BRIT. CELANESE, LTD., J. HALL, H. C. OLPIN, G. REEVES, and E. W. KIRK (B.P. 310,784, 31.1.28).—Reduced hydroxylated anthraquinones, i.e., the leuco-compounds or the anthrones, are treated with ammonia or alkylamines in presence of caustic alkali. The products are re-oxidised, e.g., with alkali and perborate. C. HOLLINS.

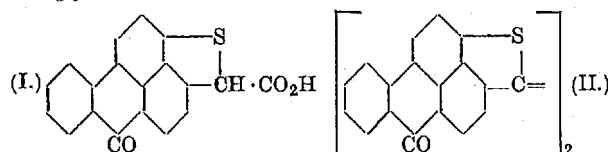
Preparation of *oo'*-dicarboxydiphenyldiaminoanthraquinones. W. L. RINTELMANN and R. J. GOODRICH (B.P. 312,678, 31.1.28).—A dihalogenoanthraquinone (especially 1:5) is heated with potassium anthranilate and basic copper acetate in methyl or ethyl alcohol at 135–140° and under 150 lb. Decarboxylation is thus avoided. C. HOLLINS.

Manufacture of benzanthrone derivatives. I. G. FARBENIND. A.-G. (B.P. 286,685, 4.2.28. Ger., 9.3.27. Addn. to B.P. 268,830; B., 1928, 666).—In the process of examples 13, 15, and 16 of the prior patent an unsaturated carboxylic acid (or ester) of the type $\text{CHR}:\text{CHR}'\cdot\text{CO}_2\text{H}$, in which R = hydrogen, alkyl, or aryl, and R' = hydrogen, aryl, or carboxylic ester group, may be employed. The intermediate additive compound, $\text{X}\cdot\text{CHR}\cdot\text{CHR}'\cdot\text{CO}_2\text{H}$ (where X = *ms*-anthronyl), is cyclised by acid reagents to give the desired benzanthrone; if R = Ph, oleum or chlorosulphonic acid, diluted with glacial acetic acid to avoid sulphonation, must be used. The resulting 3-hydroxybenzanthrone is converted by aqueous ammonia and zinc chloride into 3-aminobenzanthrone, from which the amino-group may be eliminated in the usual way by diazotisation and boiling with alcohol. The following compounds are described: β -*ms*-anthronylpropionic acid, m.p. 181°, and 3-hydroxybenzanthrone, from anthrone and acrylic or β -chloropropionic acid; β -*ms*-anthronyl-*n*-butyric acid, m.p. 160°, and 3-hydroxyl-1-methylbenzanthrone, m.p. 287°, from anthrone and crotonic acid; β -phenyl- β -*ms*-anthronylpropionic acid, m.p. 197°, and 3-hydroxy-1-phenylbenzanthrone, m.p. 320°, from anthrone and cinnamic acid or from methyl (phenyl-*ms*-anthronylmethyl)malonate. C. HOLLINS.

from anthrone and cinnamic acid or from methyl (phenyl-*ms*-anthronylmethyl)malonate. C. HOLLINS.

Manufacture of benzanthrone-*peri*-[3:4]-dicarboxylic acid or its derivatives. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 288,985, 16.4.28).—4-Benzoyl-1:8-naphthalic anhydride is heated with aluminium chloride at 190–210° to give benzanthrone-3:4-dicarboxylic acid (anhydride, m.p. above 300°). 4-*p*-Chlorobenzoyl-1:8-naphthalic anhydride and the derivatives obtained by condensing these compounds with ammonia, amines, or *o*-diamines, behave similarly. C. HOLLINS.

Manufacture of condensation products [thiazoles] of the benzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,047, 16.3.28).—3-Benzanthrone-1-thioglycolic acid fused with alkali gives the compound (I), which is readily decarboxylated and oxidised to a bluish-green vat dye (II). In place of the thioglycolic acid its ester, amide, or nitrile may be



used. Similar products are obtained from substituted derivatives, e.g., from the 9-chloro-3-benzanthrone-1-thioglycolic acid, m.p. 212–214°, obtained from 9-chloro-3-bromobenzanthrone, m.p. 258–259°. The alkaline condensing agent may be potassium hydroxide (alone or with sodium hydroxide) with or without water, alcohol, or pyridine. C. HOLLINS.

Manufacture of chemical compounds [di(halogenoacylated)diphenyl ethers]. O. VON SCHICKEL, ASST. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,717,424, 1.26.29. Appl., 28.2.28. Ger., 9.3.27).—See B.P. 286,688; B., 1929, 673.

IV.—DYESTUFFS.

PATENTS.

Manufacture of mordant [azo] dyes. I. G. FARBENIND. A.-G. (B.P. 285,097, 11.2.28. Ger., 11.2.27).—Chrome-printing brown azo dyes are obtained by coupling a resorcinol with 2 mols. of a diazotised aminosalicylic acid. Examples are: 5-aminosalicylic acid (2 mols.) \rightarrow resorcinol, α - or β -resorecylic acid; 5-aminosalicylic acid \rightarrow cresidine or α -naphthylamine \rightarrow resorcinol \leftarrow 5-aminosalicylic acid; [5-aminosalicylic acid \rightarrow cresidine] $_2 \rightarrow$ resorcinol. C. HOLLINS.

Manufacture of new basic azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 313,293, 23.5.28).—Azo dyes of the type dehydrothio-*p*-toluidine or -*p*-xyldine \rightarrow unsulphonated coupling component are alkylated with a toluene-*p*-sulphonic ester or an alkyl sulphate; alternatively, alkylation may precede coupling, the amino-group of the thiazole being first protected by acylation and the acyl group removed after alkylation. As coupling components the examples mention β -naphthol (yellowish-red on mordanted cotton), phenylmethylpyrazolone (yellowish-red), resor-

cinol (orange-brown), 2:7-dihydroxynaphthalene (yellowish-scarlet), α -naphthylamine (reddish-violet), ethyl- β -naphthylamine (violet), cresidine (claret-violet; \rightarrow resorcinol or *m*-tolylenediamine, brown), acetoacetanilide (greenish-yellow). 6-Amino-2-methylbenzthiazole is coupled with phenylmethylpyrazolone and methylated (reddish-yellow). C. HOLLINS.

Manufacture of [direct dis]azo dyes [for cotton and viscose silk]. I. G. FARBERIND. A.-G. (B.P. 285,812 and 286,226, [A] 15.2.28, [B] 27.2.28. Ger., [A] 21.2.27, [B] 28.2.27).—(A) Dyes possessing good solubility and good cotton affinity are obtained by coupling an *oo'*-dicarboxytetrazo compound with 2 mols. of an acetoacetic sulphonylamine; alternatively, a *p*-nitro-*o*-carboxydiazo compound is coupled with the sulphonated arylamide, the nitro-group reduced, and the product treated with carbonyl chloride, thiocarbonyl chloride, or carbon disulphide. Examples are: 4:4'-diaminodiphenylcarbamide-3:3'-dicarboxylic acid with acetoacetic *o*-chlorosulphonylamine (yellow on viscose; greenish-yellow on cotton, yellow when coppered), or with disulphonated acetoacetic α -naphthylamide (orange-yellow on viscose; yellow on cotton, brownish-yellow when coppered); 5-nitroanthranilic acid with acetoacetic sulphonylamine, reduced and phosgenated (golden-yellow on viscose or cotton, pure yellow when coppered). (B) A tetrazotised *pp'*-diaminodiphenylcarbamide is coupled with a sulphonated acetoacetic arylamide, or with an acetoacetic arylamide and subsequently sulphonated; alternatively, the corresponding sulphonated monoazo dye is phosgenated. Examples are: 4:4'-diaminodiphenylcarbamide-3:3'-disulphonic acid \rightarrow 2 mols. of acetoacetic sulphonylamine (yellow on cotton, golden-yellow on viscose; deep yellow lake on alumina); *p*-nitroaniline-*o*-sulphonic acid \rightarrow acetoacetic *o*-chloroanilide, sulphonated, reduced, and phosgenated (yellow on cotton or viscose or cuprammonium silk); 5-nitro-*o*-anisidine-4-sulphonic acid \rightarrow acetoacetic sulphonylamine, reduced and phosgenated (yellow). C. HOLLINS.

Thiazoles of benzanthrone series (B.P. 311,047).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Oxidation of alkali-cellulose by ageing and its importance in the manufacture of artificial silk. G. KITA and I. SAKURADA [with, in part, Y. NAKAMURA, K. SAKURADA, J. ONAHARA, and R. TOMIHISA] (Cellulose-chem., 1929, 10, 113—120).—Different effects are produced by ageing alkali-cellulose in the squeezed-out and steeped conditions. Oxidation through the action of the air occurs in both cases, but in the former the oxidation products remain in the mass, and are used in the subsequent preparation of viscose, whilst in the latter they are removed by dissolution in the sodium hydroxide liquor. Comparison is made between the properties of steeped and of pressed alkali-cellulose; the effect on the copper number of ageing for different times, the effects of ageing in a closed atmosphere of air and in an atmosphere of hydrogen, and of time of steeping in the production of the alkali-cellulose on the spinning properties of the fibres are discussed, and

differences in spinning properties, viscosity, and surface tension of viscoses obtained from alkali-celluloses prepared in different ways are described. An increase of copper number with increasing time of ageing results not only for material aged in closed or open atmospheres of air, but also for that aged in an atmosphere of hydrogen. There is no great difference between the properties of viscoses prepared from pressed and steeped alkali-celluloses. The longer is the time, or the higher the temperature of ageing in a closed atmosphere of air, the less viscous is the viscose, the thicker are the fibres, and the longer is the time of ripening necessary to give a spinnable viscose. Ageing in an atmosphere of hydrogen is a slower process than that in air; the viscose obtained is more viscous, more easily spun, and gives stronger fibres. Under certain conditions long steeping followed by a short period of ageing gives fibres as strong as those obtained by ageing in an atmosphere of hydrogen, whilst good fibres can also be obtained by ageing the alkali-cellulose for a short time only in a closed atmosphere of air.

B. P. RIDGE.

Manufacture and properties of regenerated cellulose films. W. L. HYDEN (Ind. Eng. Chem., 1929, 21, 405—410).—Transparent cellulose sheets and films, *e.g.*, Cellophane, are made by the viscose process, a final treatment with glycerin being given to impart softness and pliability. Plain transparent Cellophane permits the rapid penetration of water vapour and of water-soluble gases, *e.g.*, ammonia, carbon dioxide, but those sparingly soluble in water, such as hydrogen, diffuse through it slowly. A moisture-proof material has recently been developed, and is particularly useful as a wrapper for food products. Of the other properties of Cellophane described, the most interesting is its relatively high transparency to ultra-violet light, which is not decreased by long exposure. F. R. ENNOS.

Influence of atmospheric humidity on paper.

L. E. WALTER (Papier-Fabr., 1929, 27, 369—371).—Absorption and desorption of moisture take place more rapidly in the case of strips of paper cut transversely than in that of lengthwise strips, although the same final values are reached in both cases. At low R.H. the rate of absorption of water by paper is slower than, at average values approximately proportional to, and at high humidities more rapid than, that of increase of humidity. The extension produced is approximately proportional to the quantity of water absorbed. Absorption and desorption at ordinary room temperatures are completely reversible processes; extension is irreversible.

B. P. RIDGE.

Influence of drying on the strength of paper.

E. RIETH (Papier-Fabr., 1929, 27, 385—387).—Samples of a uniform paper from unbleached sulphite-cellulose were dried under different conditions and their moisture content, tearing length, extension, etc. were measured. The same material varies in tearing length between wide limits according to the conditions of drying. Not only is the manner of drying (*e.g.*, in the air, on the cylinder, etc.) important, but the time of contact with the cylinder has a considerable effect; the longer is this time the greater is the tearing length of the paper

and the lower are its moisture regain, extensibility, and bursting test number. Strength is dependent on the degree of swelling of the fibres, and this swelling is influenced both by the duration of drying and the nature of the grinding process.

B. P. RIDGE.

PATENTS.

Manufacture of [chloroform-soluble] acetylcellulose. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 291,001, 7.3.28. Ger., 23.5.27).—Chloroform-soluble cellulose acetates of satisfactory mechanical properties are obtained with zinc chloride as catalyst and a reaction time of 3–6 hrs. if the esterification is conducted in the early stages at 55° and is then completed at about 40°.

D. J. NORMAN.

Manufacture of cellulose esters. II. DREYFUS (B.P. 312,095—6, 17.12.27).—(A) Cellulose that has been treated with organic acids, *e.g.*, formic or acetic acid, is esterified by the action of a fatty acid anhydride in the presence of a ferric halide, particularly ferric chloride, preferably 10–30% on the weight of cellulose. The reaction is best conducted below or not substantially above the ordinary temperature. (B) A stannic halide, in the presence or absence of hydrogen halide, and optionally in mixtures with halides of iron, manganese, copper, nickel, or cobalt, is used as the condensing agent in the esterification of cellulose. Suitable quantities are 7–25% of stannic chloride or 5–15% each of stannic chloride and hydrochloric acid on the weight of the cellulose, and the parent material may, if desired, be pretreated with the lower fatty acids to increase its reactivity.

D. J. NORMAN.

Production of cellulose esters and cellulose ester products therefrom. H. DREYFUS (B.P. 312,098, 14.1. and 7.6.28).—Cellulosic material is more easily esterified if it is pretreated with 2–50%, preferably 5–10%, of hydrogen halide, alone or in conjunction with the lower fatty acids and/or ferric, stannic, manganese, cobalt, nickel, or copper halides. The pretreating agent may be used in gaseous form or in aqueous solution, and the reaction, which preferably takes place at the ordinary temperature, may proceed for 1–24 hrs. depending on the nature of the cellulosic material, the concentration of the pretreating agent, the temperature, and the degree of reactivity desired in the cellulose.

D. J. NORMAN.

Manufacture of mixed esters of cellulose and of conversion products thereof. I. G. FARBENIND. A.-G. (B.P. 283,595, 14.1.28. Ger., 15.1.27).—Mixed nitric acid-fatty acid esters of cellulose are made (1) by esterifying cellulose by means of an organic acid anhydride and adding nitric acid, preferably mixed with the fatty acid during the process when esterification has reached a more or less advanced stage; (2) by pretreating cellulose in a suitable diluent, *e.g.*, an organic acid, with a mixture of nitric acid and a catalyst (sulphuric acid) and finally esterifying by the addition of the acid anhydride.

F. R. ENNOS.

Treatment of artificial silk. Soc. D. O. (B.P. 305,622, 25.9.28. Fr., 8.2.28).—In order to increase its tenacity and elasticity, artificial silk of any type is immersed in an aqueous bath heated at 40–50° and containing

ordinary white soap, pure olive oil, sulphuric acid (*d* 1.83), and caustic soda (*d* 1.33).

F. R. ENNOS.

Celluloid compound. R. ROLAND, ASSR. to ROLAND FIREPROOF CELLULOID CORP. (U.S.P. 1,713,482, 14.5.29. Appl., 2.3.28).—A mixture of 25 pts. of acetylcellulose, 25 pts. of alcohol, 10 pts. of chlorobenzene, 10 pts. of tetrachloroethane, 10 pts. of benzylbenzoate, 10 pts. of triacetin, and 10 pts. of dichlorohydrin is heated at 75° until the acetylcellulose dissolves and the mixture becomes viscous. On evaporation a product capable of forming flexible, transparent, and non-inflammable sheets or films is obtained.

F. R. ENNOS.

Treatment of paper pulp. R. G. SMITH (U.S.P. 1,715,310, 28.5.29. Appl., 11.4.25).—Sodium sulphate and sulphuric acid are added to the pulp after digestion and before it is beaten. The solvent action is increased by the addition of sulphurous acid, and advantageously the three reagents are formed *in situ* by adding sodium sulphate and treating with excess sulphuric acid.

F. G. CLARKE.

Preparation of coconut fibres for spinning and weaving. F. L. C. BARBOUR. From B. G. H. VAN DER JAGT (B.P. 313,016, 5.12.27).

Drying of textile fibres or material. H. HAAS (B.P. 301,057, 22.8.28. Ger., 25.11.27).

Seaming compound fabrics containing rubber. DEUTS. GASLÜHLICHT-AUER-GES.M.B.H. (B.P. 292,170, 16.6.28. Ger., 16.6.27).

Spinning cans or boxes for artificial silk. N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 297,468, 20.9.28. Holl., 22.9.27).

Production of artificial silk by the stretch-spinning process. J. P. BENBERG A.-G. (B.P. 300,953, 31.8.28. Ger., 21.11.27).

Winding of artificial silk. S. DUNLOP. From SOIE DE CHATILLON (B.P. 313,425, 5.3.28).

Apparatus for drying cellulose films. FELD-MÜHLE, PAPIER- U. ZELLSTOFFWERKE A.-G. (B.P. 290,296, 11.5.28. Ger., 12.5.27).

Apparatus for continuous drying of shrinking films, particularly for drying of viscose films. H. HAMPEL (B.P. 313,829, 13.10.28).

[Roller] apparatus for treating with liquids artificial films prepared from viscose. FELD-MÜHLE, PAPIER- U. ZELLSTOFFWERKE A.-G. (B.P. 290,232, 10.5.28. Ger., 10.5.27).

Papermaking [machines]. A. KOLITSCH (B.P. 298,653, 13.10.28. Can., 13.10.27).

[Roller machine for] colouring, sizing, impregnating, or otherwise treating paper [on one surface]. I. G. FARBENIND. A.-G. (B.P. 293,000, 22.6.28. Ger., 29.6.27).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Cause of uneven dyeing of cotton yarn. H. J. KIST and C. W. ZAHN (Textilber., 1929, 10, 471–472).—Uneven results obtained in the dyeing (large-scale) of skeins of Egyptian and American cotton were traced to a previous uneven exposure of the skeins to frost. A

normal air-dry skein of cotton after exposure to -10° while in a sealed waterproof box had an increased affinity for direct dyes. When the same skein was again similarly frozen together with another normal skein, both gained an equal affinity for dyes. A. J. HALL.

Discharges [using resorcinol] on cellulose acetate silk. SCHEURER, LAUTH & CIE., and L. DISERENS (Sealed Note No. 2662, 23.6.26. Bull. Soc. Ind. Mulhouse, 1929, **95**, 349—352). Report by A. WOLFF (*Ibid.*, 352—354).—The difficulty of obtaining clear white discharges on cellulose acetate silk owing to poor penetration of the silk by the sodium hyposulphite used in such discharge processes is largely overcome by adding resorcinol to the discharge paste. Although resorcinol is a solvent for cellulose acetate its action in the discharge paste is not due to this property alone since other solvents such as phenol and acetone are not effective. When cellulose acetate is heated with a 50% solution of resorcinol, addition of ferric chloride fails to give the characteristic violet coloration, thus suggesting that a reaction between resorcinol and cellulose acetate occurs which is similar to that between acetic anhydride and phenol. In assisting discharges resorcinol is effective with a larger range of dyes than a thiocyanate (cf. B.P. 262,254; B., 1927, 71) under similar circumstances, and yields purer discharges. Wolff reports favourably on the use of resorcinol, but finds its utility restricted as in the case of a thiocyanate. If the quantity of resorcinol used exceeds 20 g. per kg. of discharge paste the silk is liable to deterioration. Attention is drawn to a similar process described by Schneevogt (B., 1926, 662). A. J. HALL.

Detection of indigosols on the fibre. J. C. LIVINGSTON (Bull. Soc. Ind. Mulhouse, 1929, **95**, 230).—It is possible to determine whether cotton fabric coloured with an indigoid dye has been dyed by the usual vat method or by means of an indigosol, by stripping it with an alcoholic solution of a hyposulphite, then boiling it in a 0.025% solution of methylene-blue, washing, and drying; fabric dyed with an indigosol has a definite affinity for the basic dye whereas fabric dyed in the vat has not. This affinity for methylene-blue is independent of the method of developing the indigosol, and is not due to the presence in the fabric of oxycellulose; it is suggested that the sulphuric acid formed by hydrolysis of the indigosol simultaneously combines with the cotton, thereby conferring on it an affinity for the basic dye. A. J. HALL.

[Production of] crêpe effects on delaines. C. FAVRE (Sealed Note No. 2284, 8.10.13. Bull. Soc. Ind. Mulhouse, 1929, **95**, 359—360). Report by A. WOLFF (*Ibid.*, 360).—In treating a large number of pieces by the method previously described (B., 1926, 123) it is difficult to obtain evenness of crêpe. It is found preferable to treat each piece separately in a tub, allowing 20 min. per piece. Those portions of the fabric previously printed with a solution of sodium silicate (*d* 1.384) resist the crêping action of the sulphuric acid used. Wolff confirms the resist action of sodium silicate, and states that whilst solutions of albumin and sucrose are equally effective, that of a gum containing barium chloride is not. A. J. HALL.

PATENTS.

Dyeing and printing [of mixed textiles]. A. THAUSS and A. GUENTHER, Assrs. to GRASSELLI DYE-STUFF CORP. (U.S.P. 1,711,002, 30.4.29. Appl., 10.11.27. Ger., 3.12.26).—In the dyeing of mixed fabrics, the wool or silk is first dyed and the direct cotton dye is then applied from a bath containing sulphonated colophony, which resists the wool or silk against the direct dye. Tin salts may be added to assist the result.

C. HOLLINS.

Ageing or reducing printed or dyed fabrics. A. E. WHITE. From DELTEX Co. (B.P. 312,423, 13.3.28).—In an ageing machine for fabrics printed or dyed with vat dyes (especially in the case of discharge printing) the steam is kept wet by supplying water in regulated quantity at points in the immediate vicinity of the runs of fabric. A machine is described.

C. HOLLINS.

Solution for dyeing purposes. A. GUENTHER, A. THAUSS, and G. MAUTHE, Assrs. to GRASSELLI DYE-STUFF CORP. (U.S.P. 1,709,976, 23.4.29. Appl., 16.12.27. Ger., 17.12.26).—See B.P. 307,777; B., 1929, 639.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Organic inhibitors in the acid corrosion of iron. J. C. WARNER (Amer. Electrochem. Soc., May, 1929. Advance copy. 10 pp.).—The rate of dissolution of iron in an aqueous solution may be diminished not only by substances which produce passivity at anodic areas, but also by substances which increase the hydrogen overvoltage at cathodic areas. This conclusion from the local-element theory is supported by experiments on the effect of gelatin, petroleum bases, and coal-tar bases on the rate of dissolution of a low-carbon iron in dilute sulphuric acid and on the hydrogen overvoltage at the metal surface in this solution. But aniline and quinoline, which definitely retard dissolution, actually lower the hydrogen overvoltage measured at 10 milliamp. per cm.² Overvoltage measurements at lower current densities might remove this anomaly.

H. J. T. ELLINGHAM.

Action of carbonic acid under high pressures on iron. E. MÜLLER and H. HENECKA (Z. anorg. Chem., 1929, **181**, 159—171).—Under high pressures of carbon dioxide, *e.g.*, 20—50 atm., carbonic acid reacts on aqueous suspensions of metallic iron to produce soluble ferrous hydrogen carbonate. As the concentration of the hydrogen carbonate ion increases the hydrogen-ion concentration of the medium decreases and the rate of dissolution of iron approaches zero. At temperatures above 30° partial decomposition of ferrous hydrogen carbonate into insoluble ferrous carbonate occurs. The dissolution-time curves for these temperatures hence show maximum values. Above 30° the eventual quantity of soluble iron decreases with increasing temperature. The eventual amount of soluble iron increases with increasing pressure of carbon dioxide, but is almost independent of pressure for pressures greater than 30 atm.

J. S. CARTER.

Corrosion of steel in dilute acids. H. ENDO and K. NAKAWAGA (J. Study Met., 1928, **5**, 301—313).

Addition of cobalt, nickel, molybdenum, chromium, titanium, vanadium, phosphorus, or much silicon to steel (containing 0.3–0.6% C) increases the resistance to 5% sulphuric, hydrochloric, or nitric acid, whilst the resistance is decreased by carbon, manganese, or sulphur. The relation between the annealing temperature after quenching and the loss of weight in 1% sulphuric acid was studied, the results being explained in terms of Honda's theory of quenching. CHEMICAL ABSTRACTS.

Tensile tests [on alloy steels] at elevated temperatures. L. GUILLET, J. GALIBOURG, and M. SAMSOEN (Compt. rend., 1929, 188, 1328–1330).—Earlier experiments (B., 1929, 601) have been extended to include tensile tests on nickel and nickel-chromium steels at 450°, and determinations of their elasticity and Brinell hardness numbers. The values obtained depend on the previous thermal treatment of the metal; e.g., oil-tempering produces a higher instantaneous extension limit than when air is the tempering medium.

J. GRANT.

Tungsten steel. W. ZIELER (Arch. Eisenhüttenw., 1929–30, 3, 61–78; Stahl u. Eisen, 1929, 49, 1083–1084).—The system iron-carbon-tungsten at the iron corner of the ternary diagram comprises the following constituents: cementite, iron tungstide, Fe_2W , two double carbides I and II distinguished from one another by their stability on annealing and their behaviour on etching with sodium picrate, and tungsten carbide. Normal cooling of alloys with 10–20% W favours the formation of a double carbide, but prolonged annealing above a critical temperature causes the separation of WC; this decomposition is retarded by quite small amounts of chromium. The metastable double carbide is coloured brown by sodium picrate and is strongly etched, whereas the stable double carbide is only slightly attacked, but is coloured dark brown. In steels with less than 0.3% C the A3 transformation point is progressively raised by addition of tungsten and its intensity decreased. In steels with 0.7–1.4% C, on the other hand, the Acl and Ac3 points are fairly constant with all proportions of tungsten, but the Ar1 and Ar3 points are lowered. The hardness of steels with 0.3% C, after quenching in oil or water, is at a maximum with 5% W; with 0.7% C steels after oil-quenching that with 1% W has the maximum hardness. A high silicon content tends to reduce the hardness. Steels with more than 1.1% C have maximum hardness with 20% W after quenching from about 1000°; the alloy with 1.4% C and 20% W, after this treatment, has a hardness of 750, which is increased still further by the presence of 0.6–0.7% Cr.

A. R. POWELL.

Determination of gases in metals, especially oxygen in iron and steel, by the hot-extraction method. H. DIERGARTEN (Arch. Eisenhüttenw., 1928–9, 2, 813–828; Stahl u. Eisen, 1929, 49, 1053–1054).—A furnace is described and illustrated for the determination of gases in iron and steel by heating *in vacuo* at 1500–1800°. It comprises a water-cooled cylindrical vessel containing a graphite heating coil enclosing a graphite crucible and provided with a water-cooled cover having openings for charging the crucible, for exhausting the furnace, and for removing the gases

evolved. The oxygen is recovered as carbon monoxide, but the results are low when more than 0.7% Mn is present owing to absorption of the gas by the volatilised manganese.

A. R. POWELL.

Rapid determination of vanadium in alloyed and unalloyed steels. K. RIES (Chem.-Ztg., 1929, 53, 527).—The steel (1–2 g.) is dissolved in dilute sulphuric acid, 10 c.c. of phosphoric acid being added if tungsten is present, the iron oxidised with nitric acid, and the solution evaporated until fumes of sulphuric acid begin to be evolved. After cooling and dilution, a few c.c. of 3% ferrous sulphate solution are added to reduce the vanadic acid to vanadyl sulphate, the excess of iron is oxidised with ammonium persulphate, and the cold solution titrated with permanganate.

A. R. POWELL.

System nickel-iron. G. J. Sizoo and C. ZWIKKER (Z. Metallk., 1929, 21, 125–126).—Single-crystal rods of iron-nickel alloys have been prepared by drawing the molten alloy by suction up a quartz tube at 50° above the m.p. of the alloy, and subsequently removing the polycrystalline skin by etching with nitric acid. The iron used contained less than 0.1%, and the nickel less than 0.4%, of impurities, and both were free from manganese. Using these alloys wires of 0.25 mm. diameter were prepared, and their sp. electrical conductivity and the temperature coefficient were determined. The conductivity curve has two rather ill-defined maxima at compositions corresponding with Fe_3Ni and FeNi_2 , and two minima at Fe_2Ni and about 90% Ni. The temperature coefficient curve has maxima and minima at the same points, but in all cases they are very well defined.

A. R. POWELL.

Influence of aluminium, lead, iron, and zinc in brass. First report of a Committee of investigation of the effect of impurities in brass (Z. Metallk., 1929, 21, 152–159).—Addition of up to 4% Pb to brass with 57, 60, or 65% Cu has little effect on the yield point or breaking strain, but it decreases the elongation of the 57% Cu alloy and the reduction in area, bending strength, and torsion of all the alloys; on the other hand addition of 1.8% Pb to the 57% Cu alloy renders it much more readily workable on the lathe. Iron up to 2% in 60 and 62% Cu brass tends to increase the strength but reduce the ductility, although the results obtained are usually very irregular. Aluminium increases the tensile strength of 68 and 62% Cu brass, but reduces the ductility and the Erichsen value. Substitution of part of the zinc in 82% Cu brass by the equivalent quantity of tin improves all the tensile properties, especially the yield point.

A. R. POWELL.

Influence of sulphur dioxide on bronze and copper. P. RÖNTGEN and G. SCHWITZKE (Z. Metallk., 1929, 21, 117–120).—When bronze with 10% Sn is melted in an atmosphere containing sulphur dioxide and then allowed to cool no porosity is observed, whereas under the same conditions copper becomes very porous. Prolonged heating of bronze in the presence of sulphur dioxide results in the formation of a sulphide layer on top of the metal, but this does not occur with copper. These results indicate that the gas is not really soluble

in the molten metal, but reacts with it forming sulphide and oxide; in the case of copper the reverse reaction proceeds with sufficient velocity during cooling of the metal to cause appreciable amounts of sulphur dioxide to be re-formed, thus giving rise to porosity. On the other hand, with a bronze the primary reaction is $\text{SO}_2 + 2\text{Cu} + \text{Sn} = \text{Cu}_2\text{S} + \text{SnO}_2$, and this reaction is irreversible at lower temperatures. Aluminium bronzes, brass, and nickel silver should behave similarly to tin bronze. A. R. POWELL.

[Solid] solubility of copper in silver. M. HANSEN (Z. Metallk., 1929, 21, 181—184).—The curve showing the boundary of the α -solid solution field in silver-copper alloys has been determined by microscopical examination of alloys after prolonged annealing at various temperatures and quenching. Up to 250° the solubility is 1.7% Cu, at 400° 2.2%, at 500° 3.2%, at 600° 5%, at 700° 7.2%, and at the eutectic temperature, 779°, 9.0%. The solidus of the α -phase is a straight line joining the m.p. of silver to the eutectic line at 9% Cu. These results are in agreement with Norbury's observations on the age-hardening of quenched silver-copper alloys (cf. B., 1928, 267). A. R. POWELL.

Rapid method of analysis of bearing-metal alloys. J. F. ILTSCHENKO and R. M. STACHORSKI (Ukraine Chem. J. [Tech.], 1928, 3, 237—240).—Analysis of alloys consisting of antimony, tin, lead, and copper is described. Antimony after conversion into the sulphate is determined volumetrically by titration with a solution of potassium permanganate, and the presence of salts of tin, lead, bismuth, zinc, and of small quantities of copper does not affect the result. Tin is determined iodometrically after conversion into the sulphate and separation from antimony and copper. Antimony, copper, arsenic, bismuth, iron, nickel, and zinc may be present without affecting the results. Lead is determined gravimetrically, and copper colorimetrically as copper ammonium salt. A. FREIMAN.

Determination of tin and antimony in bearing-metal alloys. A. P. AFANASIEV (Ukraine Chem. J. [Tech.], 1929, 4, 11—17).—On heating a sample of the alloy with nitric acid, then diluting with water, filtering, and igniting the residue, a mixture of stannous and antimony oxides is produced, in which the antimony is determined as sulphate by titrating its solution with standard potassium permanganate. The tin is found by difference. Lead present as impurity is determined as lead sulphate and copper as copper oxide. The procedure to be adopted when an excess of either alloy component is present is also fully discussed. A. FREIMAN.

Determination of bismuth [in ores]. G. J. HUGH (Chemist-Analyst, 1929, 18, No. 2, 3—4).—The ore (0.5 g.) is evaporated nearly to dryness with nitric acid, 2—3 c.c. are added in excess, the solution (50 c.c.) is warmed and filtered, ammonia added to opalescence, followed by 1 c.c. of 6*N*-hydrochloric acid and 200 c.c. of cold water. After stirring and keeping for several hours (or 1 hr. at 100°), the precipitate is decanted with hot water and washed through the filter with 25 c.c. of hot water containing ammonium chloride (5 g.) and 7.5*N*-sulphuric acid (1 c.c.). The filtrate and

washings are diluted to 150—200 c.c., boiled for 30 min. with aluminium foil, and decanted, the bismuth being dissolved by warming with saturated ferric chloride solution (10—15 c.c.). After addition of cold water (200 c.c.) and syrupy phosphoric acid (5 c.c.) the ferrous iron is titrated with permanganate.

CHEMICAL ABSTRACTS.

Determination of manganese by the Volhard method. F. J. WATSON (Chem. Eng. Min. Rev., 1929, 21, 352—353).—The electrometric determination of the end-point of manganese titrations by the Volhard method is slow, owing to the slowness of the reactions at the end of the titration. Good results may be obtained by adding a slight excess of permanganate followed by sufficient thiosulphate to destroy this excess, and then electrometrically titrating the excess of thiosulphate at 80° with permanganate. A. R. POWELL.

Use of Ganelin's method in the treatment of Altai lead ores. N. N. EFREMOV and E. M. YAKIMETZ (J. Chem. Ind. Moscow, 1927, 4, 662—664).—A preliminary examination of the applicability of Ganelin's method (G.P. 97,943/1897, 124,846/1899) to the treatment of concentrates containing Pb 45.3, Zn 12.70, Cu 0.77%, and a little silver and gold.

CHEMICAL ABSTRACTS.

Importance of the method of sampling for the analysis of alloys. ZWICKER (Chem.-Ztg., 1929, 53, 546—547).—Examples are given of the variation in composition to be expected in large ingots of red bronze or bearing metals. For sampling consignments of the former the ingots should be sawn right through at two places and all the sawings thoroughly mixed for analysis. White metal ingots may be sampled by taking drillings from several parts of the ingot, melting them in a carbon crucible, and casting in a large iron mould so that solidification is rapid. Drillings from this small ingot should represent the bulk. A. R. POWELL.

Reagent for macroscopic metallography. G. D'HUART (Rev. Mét., 1929, 26, 300—306).—An etching reagent for developing the macrostructure of cast iron, mild steels, and copper alloys comprises a mixture of solutions containing (a) 16 g. of nickel chloride dissolved in 100 c.c. of hydrochloric acid, *d* 1.18, and 50 c.c. of water, and (b) 40 g. of chromic acid in 50 c.c. of water. This reagent reveals the segregation of sulphur and phosphorus, the presence of fissures, and lines of slip in highly deformed mild steels. For enhancing the development of these lines in slightly worked steels 8 g. of copper chloride should be added to solution (a).

A. R. POWELL.

Method of electrolytic gold refining adopted by the Hitachi copper works. T. KIMATA and T. YAMAMOTO (Japan. J. Min., 1928, 44, 785—814).

Electrolytic recovery of metals. G. EGER (Chem. Fabr., 1929, 323—324, 333—335, 351—352).—See B., 1929, 560.

PATENTS.

Annealing furnaces. SIEMENS-SCHUCKERTWERKE A.-G., Asses. of SIEMENS-SCHUCKERTWERKE GES.M.B.H. (B.P. 291,437, 8.5.28. Ger., 3.6.27).—The furnace is provided with an inner jacket of heat-insulating material and a gas-tight outer jacket preferably of sheet metal;

in both jackets are apertures which can be closed or opened independently of one another, and the cooler is mounted between them and within the outer casing, thus preventing any escape of gas from the annealing chamber to the immediate vicinity of the furnace. Alternatively, the furnace has a removable cover with a hood over it, the edge of the latter being immersed in a sealing groove filled with liquid, whilst the cooler is arranged around the edge of the cover under the hood.

M. E. NOTTAGE.

Continuous annealing process. H. M. NAUGLE and A. J. TOWNSEND, ASSRS. to COLUMBIA STEEL CO. (U.S.P. 1,714,040, 21.5.29. Appl., 4.4.24. Renewed 13.10.28).—For the continuous bright annealing of ferrous metal wire or strip the material is passed into a long tube provided with water seals at both ends and containing a non-oxidising atmosphere. The first portion of the tube serves as the heating furnace and the remainder as a cooler in which the temperature of the annealed strip is reduced to below 100° before it passes the second water seal.

A. R. POWELL.

Manufacture of alloy pig iron. H. P. PARROCK (U.S.P. 1,716,181, 4.6.29. Appl., 24.12.26).—A portion of the pig iron tapped from the blast furnace is transferred while molten to an open-hearth furnace, there treated with the requisite amount of previously melted alloy metal, and cast, the remainder of the pig iron from the blast furnace being cast separately.

A. R. POWELL.

Production of cast iron. E. PIVOVARSKY, ASSR. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,715,509, 4.6.29. Appl., 17.6.27. Ger., 15.6.26).—Pig iron of such a composition that it exhibits a grey fracture when cast in the usual sand moulds is cast into chill moulds having a cross-sectional area of at least 100 cm.²; cooling is hastened by spraying the castings with water, and the metal is remelted at a high temperature to redissolve any residual graphite. Subsequent castings have a high-grade white structure.

A. R. POWELL.

Bearing metal and its manufacture. R. J. SHOEMAKER, ASSR. to S. & T. METAL CO. (U.S.P. 1,717,469, 18.6.29. Appl., 25.3.26).—Lead is melted, together with a metal which prevents oxidation, at a temperature above the m.p. of the second metal, allowed to cool, and treated with sodium at a temperature between the m.p. and b.p. of sodium.

A. R. POWELL.

Extraction of mercury from cinnabar. W. C. BAXTER (U.S.P. 1,718,103, 18.6.29. Appl., 13.4.26).—Ores containing cinnabar are leached with a solution containing an alkali sulphide and hydroxide, and the resulting liquors are electrolysed to recover the mercury and regenerate the solution.

A. R. POWELL.

Treatment of light-metal [magnesium] alloys. J. A. GANN, ASSR. to DOW CHEM. CO. (U.S.P. 1,712,988—1,712,990, 14.5.29. Appl., [A] 28.11.21, [B] 13.1.27, and [C] 29.3.27).—(A) A magnesium alloy containing 8% Al is annealed at 482–493° for about 1 hr., whereby the greater part of the eutectic goes into solid solution. (B) A magnesium alloy containing a metal which forms both a solid solution and eutectic with magnesium is annealed at a temperature just below the m.p. of the

eutectic for a sufficient time to cause all the eutectic to enter into solid solution. (C) A magnesium alloy containing zinc and aluminium is annealed at 340° until the zinc enters completely into solid solution, then at 490–500° to cause the greater part of the aluminium to dissolve.

A. R. POWELL.

Aluminium-beryllium alloy and its [heat] treatment. R. S. ARCHER and W. L. FINK, ASSRS. to ALUMINUM CO. OF AMERICA (U.S.P. 1,716,943, 11.6.29. Appl., 22.11.26).—Aluminium containing beryllium is annealed at a temperature slightly below the m.p. of the eutectic to cause the greater part of the beryllium to pass into solid solution, quenched, and subsequently aged at such a temperature and for such a time as to produce the desired hardening effect.

A. R. POWELL.

XI.—ELECTROTECHNICS.

PATENTS.

Electrolyte condenser. TELEGRAPH CONDENSER CO., LTD., and W. J. COLE (B.P. 314,160, 18.4.28).—Metal sheets, e.g., of aluminium 0.001–0.004 in. thick, interleaved with sheets of absorbent fibrous material are rolled spirally, and the absorbent material impregnated, preferably *in vacuo*, with an electrolyte containing dextrin, gelatin, agar-agar, sugar, gum arabic, or glycerin.

J. S. G. THOMAS.

Electrolytic condenser. STANDARD TELEPHONES & CABLES, LTD. FROM WESTERN ELECTRIC CO., INC. (B.P. 314,565, 29.3.28).—Electrodes, of which one at least is made of tantalum, and an electrolyte of jelly-like consistency composed of sodium silicate and dilute sulphuric acid are arranged in a container having a non-liquid seal, e.g., of cork impregnated with paraffin, which is normally gas-tight but becomes pervious to gases before the gas pressure in the container becomes excessive.

J. S. G. THOMAS.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Kauri resin. J. R. HOSKING (Rec. trav. chim., 1929, 48, 622–636).—An attempt is made to estimate and compare the constituents of kauri resins of varying age. Four varieties of the resin have been examined, viz.: (1) fresh from the tree *Agathis australis*, (2) the same after remaining in air or in casks for a long period, (3) fossil resin ("range gum"), and (4) from peat moors. (1) The thick, milk-white liquid was distilled with steam until all volatile oils had passed over (average yield 8.4%); the distillate, d_4^{20} 0.8588, n_D^{20} 1.4673, $[\alpha]_{5461}^{20} +17.47^\circ$, by fractionation afforded a 92% yield of d - α -pinene, b.p. 154–158°, d_4^{25} 0.8535, n_D^{25} 1.4658, $[\alpha]_{5461}^{25} +22.26^\circ$, but no trace of β -pinene could be detected. The aqueous liquors which remained in the distilling apparatus yielded (1.1%) a gum by concentration and precipitation with excess of alcohol, which was initially bright red, but rapidly became black in air. The colour change is due to the presence of small quantities of manganese derivatives which undergo oxidation. By shaking the aqueous solution of the gum with kaolin and reprecipitating it with alcohol, a nearly colourless product which no longer reduces Fehling's solution was obtained; after

drying in air it contained 14.3% of water and yielded 5.1% of ash, mainly calcium carbonate, giving a positive reaction for manganese. The aqueous solution of the purified gum was inactive. Oxidation with nitric acid (*d* 1.15) furnished a 13% yield of mucic acid; hydrolysis with 5% sulphuric acid at 100° for 18 hrs., followed by destruction of galactose with yeast, afforded an uncrystallisable syrup, dextrorotatory in solution, from which an osazone, m.p. 158°, and a *p*-bromophenylhydrazone, m.p. 160° (decomp.), were obtained, and which was therefore probably *l*-arabinose. The mother-liquors from the precipitation of the gum after concentration and treatment with a solution of basic lead acetate gave a strongly dextrorotatory solution which yielded an uncrystallisable syrup (osazone, m.p. 186—190°) which possibly contained mainly *d*-glucose. The insoluble resin remaining from the steam-distillation (77.6% of the original resin) was powdered, and separated from ligneous matter by dissolution in an ether-alcohol mixture; the product, obtained by removal of the solvent, gave an equivalent of 103 by titration, and values from 104 to 108 were obtained by hydrolysis for varying periods at 15° or at 100°; the resin therefore contained no esters. The powdered product was extracted thrice by shaking with ether, and the acid fraction of the extract separated by treatment with alcoholic potassium hydroxide, followed by acetic acid. This "α"-resin acid was purified through the potassium salt, the product so obtained constituting 63% of the crude steam-treated resin. Further purification by repeated conversion into the sodium salt yielded the "α"-resin acid as a white, amorphous powder, softens 98°, m.p. 121—124°. The neutral portion of the ethereal extract yielded a yellow solid, m.p. 60—70°, representing 10% of the crude steam-treated resin, and corresponding with the "kauroresin" of Tschirch and Niederstadt (A., 1901, i, 398). The "β"-resin acid was obtained by shaking the residue from the ether extraction with alcohol, and isolated as the potassium salt; this salt by treatment with alcoholic hydrochloric acid gave the free "β"-resin acid as a white, gelatinous mass, converted by drying into a brown powder, softens 210°, m.p. 233—236°. The "β"-resin acid constitutes 19% of the crude steam-treated resin. The original resin contained 3.1% of impurity, and some water (water + loss = 9.8%). The other varieties, by similar treatment, gave the following results: (2) The specimens examined were 2 and 5 years old, respectively, and gave 6.8, 4.85% of volatile oil; 0.45, 0.3% of gum; and 89.4, 92.65% of resin. The oil from the older specimen contained 89% of *d*-α-pinene, *d*₂₀²⁵ 0.8529, *n*_D²⁵ 1.4633, [α]₅₄₆₁ +10.08°, whilst the gum contained manganese and closely resembled that obtained from the fresh resin (1). The resin from the older specimen (titration value 76.6) yielded an "α"-resin acid, m.p. 120—124° (39%), a "β"-resin acid, m.p. 235—237° (45%), together with 9% of neutral products. (3) The specimen afforded 4.0% of volatile oil, 0.15% of gum, 94.05% of resin; fractionation of the oil gave a trace of a solid, m.p. 123°, which was acid to litmus, a 63% yield of *d*-α-pinene, *d*₂₀²⁵ 0.8622, *n*_D²⁵ 1.4664, [α]₅₄₆₁ +10.96°, and a 6% yield of a fraction, b.p. 190—215°, which was probably

mainly fenchyl alcohol. The gum was obtained only in small quantity and in an impure state; it contained manganese. The resin (titration value 64.9) yielded an "α"-resin acid, m.p. 120—125° (35%), a "β"-resin acid, m.p. 232—234° (48%), and 9% of neutral products. (4) The specimen gave 0.85% of volatile oil, 0.15% of gum, and about 85% of resin. The crude steam-distilled oil (titration value 1.9; ester value zero), by acetylation and hydrolysis, had an alcohol content of 12.9%, calculated on the assumption that the alcohol present was fenchyl alcohol. By fractionation were isolated: *d*-α-pinene, b.p. 155—156°/758 mm., *d*₂₀²⁵ 0.8632, *n*_D²⁵ 1.4653, [α]₅₄₆₁ +11.24° (30.5%); dipentene containing some limonene (38.5%); fenchyl alcohol, m.p. 39—41° (fenchyl hydrogen *o*-phthalate, m.p. 145—146°, [α]₅₄₆₁ +15.08°; fenchoxime, m.p. 163—164°) (12.9%, of which 7.5% was isolated). The amount of gum was very small, but contained manganese. The resin (titration value 63.5) was not further examined. The variation with age of the components isolated and their probable mode of origin is discussed; in general, the amount of volatile oil and gum decreases, and the amount of resin increases, with increasing age.

C. W. SHOPPEE.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rational classification of the principal accelerators of vulcanisation [of rubber]. R. THIOLLET and G. MARTIN (Caoutchouc et Gutta-Percha, 1929, 26, 14494—14497).—In grading vulcanisation accelerators it is desirable to consider, in addition to mere activity, the following features:—times necessary at different temperatures for "fixation" of mixtures, and for vulcanisation to maximum mechanical properties, ageing, plastifying effect, and influence of other ingredients on the activity of the accelerator. The point of "fixation" is reached when the mass can be just removed cleanly from the mould, but cut pieces are still capable of being pressed together; immediately before arrival at this point the mass softens if heated rapidly, whereas just after this point it becomes stronger. A mixture of two accelerators, e.g., mercaptobenzthiazole and diphenylguanidine, may cause fixation in a shorter time than the same total quantity of either constituent alone, but this is not always so.

D. F. TWISS.

XV.—LEATHER; GLUE.

Analysis of artificial bating materials [for leather]. II. V. KUBELKA and J. WAGNER (Collegium, 1929, 247—262; cf. B., 1929, 140).—The authors' method is fully described. It differs from the Schneider-Ulcek method (B., 1927, 662) in principle, in the concentrations used, and in the mode of expression of the results. The enzyme activity of the samples is determined by the Schneider-Ulcek method only after they have been brought to the same ammonium salt content, whereas in the authors' method the bate is tested as it is. Temperature of extraction affects the analysis; 37° is used by the authors and 18° in the Schneider-Ulcek method.

D. WOODROFFE.

"Syntan" [synthetic tannin] tannage. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1929, 21,

698—701).—Experiments to determine the amount of tannin fixed by hide powder from solutions of quebracho and 1, 5, and 15% respectively of a synthetic tannin, quebracho and sulphuric acid, quebracho, sulphuric acid, and sodium sulphate, and quebracho alone, showed that the addition of synthetic tannin increased the initial rate of fixation of tannin. The amount of tannin fixed in a given time from the different quebracho liquors was increased by small additions, but diminished by larger amounts of synthetic tannin, which can be explained by the lowering of the pH value of the liquors. The tannin fixed from wattle liquors was decreased by the addition of synthetic tannin, but was greater than when sodium sulphate was added. The greater fixation from tan liquors containing synthetic tannin and of the same pH value as tan liquors containing sodium sulphate pointed to some other effects of the synthetic tannin besides acidity. The tanned powder was more "leathery" and softer. D. WOODROFFE.

Tannin analysis. Report of a committee of the International Association of Leather Trades' Chemists. M. BERGMANN (Collegium, 1929, 233—247).—Higher results for the total soluble matter were obtained by the use of calcium chloride instead of sulphuric acid or phosphorus pentoxide in the desiccators. Quicker evaporation was obtained in metal basins than in porcelain, but the same weight of dry residue was obtained in each. The greatest degree of concordance of the results of different analysts on different tanning materials respectively was obtained by the Grassier-Allen method of extraction (B., 1911, 909) as compared with the Koch methods of extracting 1 or 2 litres respectively. Solutions of extracts have been prepared by the proposed official international method, by dissolving in boiling water and diluting to 1 litre with boiling water, and also by dissolving in 100 c.c. of boiling water, heating on the water-bath for 30 min., and diluting to 1 litre with boiling water. The results obtained by these three methods on different extracts were very similar. A suggestion to moisten the ground tanning material before determining the moisture was proved unnecessary. The total soluble matter in a solid untreated quebracho extract was best determined by using a Berkefeld filter candle and suction. Ready-chromed powder gives as good results as freshly-chromed hide powder. Good results have been obtained with the Darmstadt (B., 1929, 140), Freiberg (B., 1928, 722), and Keigueloukis (B., 1929, 221) apparatus for avoiding the handling of the hide powder. Comparisons of the "filter-bell," old official "shake," and provisional international methods of analysis have shown that the greatest degree of concordance was obtained by the "filter-bell" and the least by the international method. D. WOODROFFE.

XVI.—AGRICULTURE.

Use of alcoholic salt solutions for the determination of replaceable bases in calcareous soils. O. C. MAGISTAD and P. S. BURGESS (Ariz. Agric. Exp. Sta. Tech. Bull., 1928, 20, 481—497).—The ammonium chloride method is inaccurate on account of dissolution of organic matter and of calcium from calcium carbonate,

and owing to mechanical losses. Ethyl-alcoholic (68%) barium chloride solution (0.1N) is therefore used as displacing agent. The alcohol is removed by evaporation, the barium precipitated as chromate, and the calcium, magnesium, and other bases are determined in the supernatant liquid. Alkali salts, if present in the soil, must first be extracted with water.

CHEMICAL ABSTRACTS.

Apparatus for measurement of pH . A. UHL (Fortsschr. Landw., 1928, 3, 973—975; Chem. Zentr., 1929, i, 133).—An apparatus for field work is described.

A. A. ELDRIDGE.

Soil of the forest areas of E. Norway and the Trondhjem district. H. GLÖMME (Publ. Norwegian Forestry Res. Service, 1928, 3, No. 10 [i]; Proc. Internat. Soc. Soil Sci., 1929, 4, 97—99).—Interrelationships of soil properties, surface vegetation, forest growth, and the nature of the humus formed are examined. The humification of acid material poor in lime and nitrogen (e.g., heather lichens and pine needles) mainly by the action of fungi is slow and leads to a poor acid humus. Examination of forest profiles is recorded.

A. G. POLLARD.

Distribution of phosphorus compounds in soils, with reference to soil-surface formation. F. TERLICHOWSKI and S. MICHNIEWSKI (Rocz. nauk. Roln. i Lesnych, 1927, 18; Proc. Internat. Soc. Soil Sci., 1929, 4, 57).—The changes occurring in the form of combination of soil phosphorus and its ultimate assimilation by the plant are largely dependent on the nature and general direction of soil solution movements, and these in turn are controlled by the mechanical and chemical composition of the soil. By the examination of the distribution of phosphorus in different soil horizons certain relationships are established.

A. G. POLLARD.

Preservation of soil profiles. K. SCHLACHT (Z. Pflanz. Düng., 1929, 13A, 426—431).—A sheet of celluloid or other suitable material is coated with a solution of a colourless water-soluble condensation product of urea and formaldehyde. This is pressed against the vertical, smoothly-faced profile wall. After 5—10 min. the sheet is peeled off with a thin layer of soil adhering; when dry a permanent "monolith" of the profile is obtained. Negative reproductions can be made with paraffin wax casts and subsequently transferred to plaster of Paris positives.

A. G. POLLARD.

Denitrification in uncultivated soils. A. KARLSEN (Bergens Museum Årbok, 1927. Naturv. rekke No. 4; Proc. Internat. Soc. Soil Sci., 1929, 4, 28—29).—Denitrifying organisms were present in numerous cultivated, forest, heath, and moorland soils. No relationship between the intensity of denitrification and the nature of the flora or geological strata was observed.

A. G. POLLARD.

The phosphate question [and crop yields]. O. ARRHENIUS (Z. Pflanz. Düng., 1929, 14A, 121—140).—On the basis of Mitscherlich's growth curves a close relationship is established between the amount of soil phosphate soluble in 2% citric acid solution and crop yields. The results of field trials combined with simultaneous analysis of the citric-soluble phosphate allow

of the preparation of soil charts from which the phosphate requirement of crops may be calculated.

A. G. POLLARD.

Conditions of application and of action of phosphates [on chernozem soils]. V, VI. Reversion of phosphates in soils. VII. Organic parts of the exchange complex. M. A. EGOROV (Ukraine Chem. J., 1928, 3, 241—262; cf. B., 1928, 618).—V. It is shown that the specific action of sodium phosphates on soils is not due only to their alkalinity. The amount of organic matter extracted by a sodium phosphate solution increases with time, but the rate of increase for even the same soil type, *e.g.*, chernozem, varies with the locality. Time and degree of humidity of the soil are of great importance in determining the amount of organic matter extracted. For chernozem soils there exists an optimum humidity, but this again varies from locality to locality. Also the concentration of the sodium phosphate solution used is of importance; in some cases this concentration is sharply defined, and in others less so.

VI. Shaking of a soil with solutions of the different sodium phosphates gives the corresponding calcium salts. These are considered to be the separate steps in the reversion process taking place in the soil, and they are manifested by the different colour intensities of the water extracts of the soils. The reversion process, however, does not in all cases preserve its initial intensity or its continuity. In some cases both the continuity and the intensity remain unchanged, in others it remains continuous but less intense, whilst again in others both factors suffer.

VII. It is confirmed that non-chernozem soils on extraction with a sodium phosphate solution furnish more organic matter than does a chernozem soil. Again, the fraction due to crenic and apocrenic acids in the total organic matter extracted is particularly small in the case of the chernozem soils, whereas the humic acid fraction is largest in chernozem soils and smallest in other types.

A. FREIMAN.

Nitrates in soil and plant as indexes of the nitrogen need of a growing crop. B. E. GILBERT and J. B. SMITH (Soil Sci., 1929, 27, 459—468. Cf. B., 1927, 825; A., 1928, 1162).—The artificial maintenance of the nitrate contents of the soil and plant solutions above the "sub-optimum" values for a number of market-garden crops yielded uniformly increased crops. The nitrogen requirement of soil for young growing crops can be calculated from the nitrate content of the soil, but the nitrate content of the plant solution is also necessary in the evaluation of the nitrogen requirement for the total crop.

A. G. POLLARD.

Fertilising action of some potash salts used singly and in mixtures. H. LIESGANG (Z. Pflanz. Düng., 1929, 8B, 62—65).—Comparison is made of the efficiency of the chloride, sulphate, and nitrate of potassium in pot-culture experiments with oats. Contrary to the conclusions of Hellriegel, mixtures of the chloride and sulphate or chloride and nitrate produced no greater crops than equivalent amounts of potassium applied as nitrate or sulphate alone. In the relatively high concentrations used, potassium chloride yielded definitely

smaller crops than the sulphate or nitrate applied singly.

A. G. POLLARD.

Liming as a factor in the mobilisation of phosphoric acid in podsol soils. D. L. ASKINASI and S. S. JARUSSOV (Wiss. Inst. Düngemittel, 1928, No. 57; Z. Pflanz. Düng., 1929, 13A, 294—295).—The liming of podsoles increases the energy of decomposition processes and the mineralisation of humus. The latter is connected with the enrichment of the soil solution in inorganic nitrogen and phosphorus. Following liming, the crop yield and also its nitrogen and phosphate content increased. These effects gradually decrease as the effect of the lime is obliterated during a period of about ten years. The changing condition of phosphate nutrition during this time is confirmed by examination of Neubauer's method. The slow-acting phosphates of aluminium and iron and the organic phosphorus compounds existing in acid soils are assumed to be converted into calcium phosphate by liming.

A. G. POLLARD.

Agricultural value of Nile silt held fallacious. E. MCK. TAYLOR (Eng. News-Rec., 1929, 102, 993—995).—Field trials extending over a number of years show that Nile silt has not the fertilising power usually ascribed to it. The essential factor in the maintenance of the soil fertility of the Nile basin is the summer fallow, and the decline in crop yields in recent years is attributed to the elimination of this from the system of agriculture rather than to the decreased amounts of silt reaching the land during irrigation.

A. G. POLLARD.

Significance [to crop yields] of the solubility in ammonium citrate of Rhenania phosphate and basic slag. A. WILHELMJ [with H. K. and S. GERICKE] (Z. Pflanz. Düng., 1929, 8B, 42—61).—In pot-culture experiments with oats there was no relationship between the crop yields and the ammonium citrate solubility of a number of samples of Rhenania phosphate and basic slag. The nutrient value of these two fertilisers is best expressed by their solubility in citric acid. The alleged superiority of Rhenania phosphate over basic slag could not be substantiated.

A. G. POLLARD.

Plant nutrition studies in relation to the triangular system of water cultures. H. F. CLEMENTS (Plant Physiol., 1928, 3, 441—458).—Length, weight, and content of ash, nitrate, or soluble carbohydrates do not adequately indicate the best balance of salts for the growth of plants.

CHEMICAL ABSTRACTS.

Mud fertiliser from sewage fields. HEINE (Landw. Jahrb., 1928, 68, Suppl. I, 355—356; Chem. Zentr., 1929, i, 130).

Effect of lime materials on the outgo of sulphur from Hagerstown silt loam soil. W. B. ELLETT and H. H. HILL (J. Agric. Res., 1929, 38, 697—711).—The results are recorded of the examination of the drainage waters in lysimeter experiments with soil treated with various materials containing lime. Such treatment did not markedly increase the leached sulphur, but stimulated the biological transformation of sulphur in soils. The addition of subsoil to surface soil decreased the sulphur losses in the drainage.

A. G. POLLARD.

Is sulphur a limiting factor of crop production in some Utah soils? J. E. GREAVES and W. GARDNER

(Soil Sci., 1929, 27, 445—457).—A relationship is established between the crop-producing power of Utah soils and their sulphur content as the latter decreases during continuous cropping. The addition of sulphur-carrying salts increases bacterial activity in these soils, particularly nitrogen fixation. A. G. POLLARD.

Comparative acid tolerance of some southern legumes. G. JANSSEN (Soil Sci., 1929, 27, 469—497).—The growth of a number of legumes in sand cultures was examined, and in some cases good plants were obtained in moderately acid media provided nitrate was present. In soil the optimum growth reaction occurred at pH 6.0—6.8 for the various species. Sweet clover grew better in acid soils neutralised with soda than when lime was used. A. G. POLLARD.

Nitrogen problem in sugar cane culture in Java. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, III, No. 3, 91—152; Proc. Internat. Soc. Soil Sci., 1929, 4, 87—89).—The rate of intake of nitrogen by sugar cane reaches a maximum after the 30th week, and the total assimilated increases with the concentration of nitrogen in the nutrient. Optimum crop yields are attained with concentrations of 10.5—31.5 mg. of nitrogen per litre, corresponding to a total consumption of 640—800 kg./hectare. Low nitrogen concentrations in the early growth period of cane have an unfavourable effect on the crop, but in later stages low concentrations are tolerated without loss. The nitrate-producing power of soils is more important for sugar cane culture than the actual amount of nitrate present at any one time. Ammonium sulphate manuring was profitable on soils the nitrate-producing capacity of which was less than 10 mg. NO_3 per kg. The inverse relationship between nitrate-producing capacity and nitrogen deficiency is sufficiently close to allow of the calculation of the latter from the former in practice. A. G. POLLARD.

The chlorine question in sugar cane culture in Java. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, I, No. 3, 90—100; Proc. Internat. Soc. Soil Sci., 1929, 4, 85—87).—In sand culture with sugar cane sodium chloride was added in varying proportions to the nutrient. Maximum yields were obtained with 0.006% of chloride. With 0.018% of chloride yields had fallen below those from chloride-free nutrients. The chlorine content of the cane was but little affected by variations in that of the nutrient. Plants growing in light soils were more sensitive to the effects of chlorides than those in heavy soils. In practice, drainage and leaching are necessary for the removal of excessive chlorides. A. G. POLLARD.

Has manuring with ammonium sulphate a noxious effect on sugar cane? O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1927, No. 37, 939—941; Proc. Internat. Soc. Soil Sci., 1929, 4, 85).—Ammonium sulphate in amounts commonly used can only affect the yield of sugar cane on very acid soils. In Java soils no difference in manurial effect was observed on acid, alkaline, or neutral soils. A. G. POLLARD.

Noxious effect of molasses on soil. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1927, No. 31, 791—798; Proc. Internat. Soc. Soil Sci., 1929, 4, 90—91).—

The reduced crop yields following the application of molasses to soil result from the withdrawal of nitrate from the soil by organisms decomposing the molasses. The more severe effects noted in heavy soils are solely the outcome of the longer period of decomposition of molasses, compared with sandy soils, wherein decomposition is complete in about 4 weeks. A. G. POLLARD.

Manurial effects of phosphorites on podsol soils. A. T. TIULIN and WOSBUTSKAJA (Agric. Exp. Stat. Perm, No. 1, 1927; Proc. Internat. Soc. Soil Sci., 1929, 4, 76—77).—The principal factors affecting the availability of rock phosphates in soils are the acidity and base-saturation of the soil, nitrification, and the properties of the soil minerals. In pot experiments, acidification of soils with nitric and sulphuric acids, with the sulphates of iron and aluminium, and also treatment with sodium bicarbonate increased the availability of rock phosphate over that in neutral soils. Availability was, however, decreased by acidification with gypsum and with aluminium sulphate in the presence of lime. The latter effect is ascribed to the greatly increased concentration of calcium in the soil solution and the consequent depression of the solubility of calcium phosphate. After the application of soluble phosphates, the greater assimilation and crop yields in neutral soils are due to the more favourable conditions for nitrification. Analysis of drainage waters from the treated soils showed the greater concentration of calcium in acid soils, and the increased nitrate in neutral soils. In alkaline soils the enhanced concentration of phosphorus appears to result from the formation of soluble organic phosphorus compounds. A. G. POLLARD.

Liming of soil and the use of heavy dressings of potash salts. VINCENT and HERVIAUX (Ann. Sci. Agric., 1928, 45, 335—357; Proc. Internat. Soc. Soil Sci., 1929, 4, 72—73).—Pot experiments and field trials show that preliminary saturation of the soil colloids leads to a better assimilation of potash fertilisers by plants. Heavy applications of potash for this purpose are expensive, and may involve the necessity of frequent liming. By the use of sufficient lime to control soil acidity, and bring about a satisfactory condition of the soil humus, practically complete assimilation of potash fertilisers may be attained. A. G. POLLARD.

Value of by-products of carbonate filters as a fertiliser. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1928, No. 5, 100—108; Proc. Internat. Soc. Soil Sci., 1929, 4, 91—92).—The by-product contains finely divided chalk and appreciable amounts of nitrogen and phosphorus, and is valuable as a sugar cane fertiliser. A. G. POLLARD.

Importance of nitrogen nutrition of crops. O. ARRHENIUS (Med. No. 313, Centralanst. försöksv. jordbruks., 1927, 1—17; Proc. Internat. Soc. Soil Sci., 1929, 4, 92—93).—The intake of nitrate by cereals and sugar beet takes place in two definite periods, and it is essential for maximum crops that optimum quantities of nitrate should be present at these times. The period of nitrogen intake varies with the nature of the crop. High concentrations of nitrate induced root rot in sugar beet and intended to produce sterile grain in maize. A. G. POLLARD.

Importance of potash and phosphate nutrition of crops. O. ARRHENIUS (Med. Centralanst. försöksv. jordbruks., Avdeln. lantbruksbot., 1927, 1—12; Proc. Internat. Soc. Soil Sci., 1929, 4, 74).—In sand culture experiments increasing concentrations of phosphate in the nutrient yielded increasing crops up to a maximum of 9 mg. PO_4 /litre. Higher concentrations in some cases reduced the yields, except in the case of clover, where the optimum concentration was 30 mg./litre. Similar experiments with increasing concentrations of potassium indicated an optimum concentration of 6 mg./litre for cereals and about 20 mg./litre for clover and sugar beet. Comparison of the total intake of potash and phosphate by plants with the concentration of these nutrients in soil solutions showed that, in general, the concentration of potassium in soils approached the optimum, but that of phosphate averaged about one half of the optimum value. The ability of plants to take up nutrients by other means than direct absorption of soil solutions is suggested. Manuring should be directed to increasing the concentration of the soil solution to the optimum, rather than to supplying the total nutrient required by the plant. A. G. POLLARD.

Root rot and soil properties. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, 129—143; Proc. Internat. Soc. Soil Sci., 1929, 4, 59).—The prevalence of root rot could not be correlated with the reaction or phosphate content of soils, but was related to the nitrifying power and certain physical properties. It is not clear whether these last two factors are interdependent or are separately effective. A. G. POLLARD.

Passage of ash-substance into the plant. I. Reaction of the medium as a factor in the mineral nutrition of plants. D. A. SABININ and G. S. KOLOTORA (Agric. Exp. Stat. Perm., 1926, 1, 91—113; Proc. Internat. Soc. Soil Sci., 1929, 4, 81—82).—Maize grown in water cultures showed maximum growth with nutrients at pH 4 and minimum growth at pH 8. The reaction of the plant sap was not influenced by that of the nutrient, but the latter greatly affected the relative proportions of anions and cations entering the plant system. Acid media favour the intake of a preponderance of anions, and *vice versa*. The rate of absorption of mineral matter varies irregularly during the growth of the plant, the maximum in the case of phosphates occurring during the first 3—4 weeks of development. A. G. POLLARD.

Chlorate method for determination of nitrate, nitrogen, total nitrogen, and other elements in soils and plant tissues. E. M. EMMERT (J. Assoc. Off. Agric. Chem., 1929, 12, 240—247).—*Nitrate nitrogen*: The ground, dried tissue (0.2—0.5 g.) or green tissue or soil (1—3 g.) is heated in a Kjeldahl flask with 25 c.c. of 50 vol.-% sulphuric acid, and the expelled gases are led through a condenser into an absorption tower containing 150 c.c. of freshly prepared chlorine dioxide solution made by dropping concentrated hydrochloric acid on to 15 g. of sodium chlorate and passing the gas evolved into water until a deep yellowish-green coloration develops. The exit from the tower is fitted with a Kjeldahl (Hopkins) trap adjusted so that some of the solution is forced into it. The heating of the flask is continued until white fumes begin to be evolved. The

combined washings from condenser, trap, and connexions are boiled until colourless, and an aliquot portion containing at least 0.25 mg. of nitrogen is shaken first with 0.05—0.1 g. of silver sulphate and later with 0.5—1 g. of calcium oxide; an aliquot portion of the clear filtered liquor, containing at least 0.2 mg. of nitrogen, is then evaporated to dryness, and 2—3 c.c. of phenoldisulphonic acid solution are added, followed after 5—10 min. by 20—30 c.c. of water so as to dissolve most of the salts. Sodium hydroxide solution (25%) is now added, a few c.c. in excess of that required to develop a yellow colour. The solution is made up to 100 c.c., shaken with 0.5 g. of calcium hydroxide, and sufficient filtered to compare colorimetrically with a standard solution containing 0.0025 mg. of nitrogen per c.c. The volume of the unknown is adjusted until its strength is nearly equal to that of the standard, and the nitrate nitrogen in the sample is calculated from the colorimetric readings. In some samples from which gaseous evolution is violent, *e.g.*, clay loam soils, much of the nitrate nitrogen was lost. *Reduced nitrogen*: The residue from the distillation of nitrate nitrogen is cooled, diluted with 10 c.c. of water, and again cooled. Then 1 g. of sodium chlorate is added for each 0.1 g. of dry tissue or each 0.5 g. of green tissue or soil, and the mixture is shaken and heated rapidly in an open Kjeldahl flask until the green fumes formed decompose and only white fumes are present. When the temperature reaches 100° the flame is lowered, the flask connected to the distillation and absorption apparatus, and, after the violent reaction ceases, the nitric acid is distilled rapidly into water or dilute chlorine peroxide solution. When white fumes appear and the solution remains colourless, the flask is disconnected before turning out the flame. The method is continued as in that for nitrate nitrogen. *Total nitrogen*: An appropriate quantity of the sample is placed in a Kjeldahl flask with sodium chlorate in the same proportion as for reduced nitrogen and 25 c.c. of 50 vol.-% sulphuric acid are added. The method is carried out as for reduced nitrogen, care being taken to prevent accumulations of chlorate and sample near the flame. *Non-volatile elements*: The flask is now tilted so that the neck slopes downward without the contents running out, and the sulphuric acid is distilled off and collected. The residue is cooled, and made up to a known volume or used as a whole. When sodium is to be determined, potassium chlorate may be substituted for the sodium salt. *Carbon*: The gases evolved in the oxidation are collected and measured, and an aliquot part is passed through a suitable absorption train, the carbon dioxide being absorbed and weighed in a soda-lime-phosphorus pentoxide bulb. Preliminary tests gave good results. W. J. BOYD.

XVIII.—FERMENTATION INDUSTRIES.

Proteins of different barleys and pasteurisation turbidities. W. HESSELBURGER (Woch. Brau., 1929, 46, 285—288).—By treatment with suitable adsorbents beers of normal flavour and head retention can be obtained which remain bright when submitted to the most adverse conditions, independently of age, temperature, treatment, malt quality, and mashing

conditions. Examination of the heat-coagulum from untreated and treated beers after hydrolysis by Van Slyke's method shows a marked difference in the adsorption of the different nitrogen compounds. Thus, arginine- and histidine-nitrogen is mostly adsorbed, but not cystine- and lysine-nitrogen. Applications of the method to barleys, malts, and beers are quoted from which it appears that these qualitative differences may relatively greatly exceed the difference in total nitrogen between two barleys, and may prove to be a determining factor in the brewing quality of barley and malt.

F. E. DAY.

Determination of the volatile acids in beer. W. WINDISCH, P. KOLBACH, and E. SCHILD (Woch. Brau., 1929, 46, 245—248, 255—259).—Acetic acid is not volatilised by passing a stream of air through 370 c.c. of a solution requiring 3.77 c.c. of 0.1*N*-caustic soda for neutralisation, nor is formic acid lost under similar conditions. Added acetic acid cannot be quantitatively recovered from beer by steam distillation at ordinary pressure, or by evaporating to dryness *in vacuo*. On adding successive quantities of 50 c.c. of water to the dry residue of beer distilled *in vacuo* and again distilling, a point is reached after about two such additions at which the amount of additional volatile acid obtained is small, and the total yields from duplicate tests agree satisfactorily. If the acidity of the beer is increased by adding a non-volatile acid, *e.g.*, phosphoric acid, the yield of volatile acid changes only slightly between p_H 3 and 2.5, but is markedly increased at p_H 2.0, and at p_H 3.0 the increased volatile acidity obtained after the second addition of water and redistillation is at a minimum. Tests with beers buffered to definite reactions show that above p_H 3 acetic and formic acids are not completely removed, and that lactic acid volatilises from more acid beers. No appreciable quantity of volatile acid is formed under the conditions of distillation. Though in a few cases a third addition of water and redistillation gave a distinctly higher result, exceeding 0.1 c.c. of 0.1*N*-caustic soda per 100 c.c. of beer, this is regarded as abnormal, possibly due to butyric acid. For the determination the beer is aspirated with air freed from ammonia and carbon dioxide till free from the latter. To 100 c.c., 0.1*N*-phosphoric acid is added to bring the p_H to 3.0 (1.3 c.c. for each 1% of original extract), and 30 c.c. of tartaric acid-sodium tartrate buffer solution, p_H 3.0, 0.5*M* in respect to total tartaric acid. The mixture is distilled from a Claisen flask of which the exit is bent first upwards, then down, through a 100-cm. Liebig condenser. A few drops of octyl alcohol, renewed with the water additions, and a little paraffin, m.p. 40—42°, prevent frothing. The distillate is collected in 100 c.c. of water in a 500-c.c. cylinder cooled in ice. The flask is heated on a water-bath, heat being applied after vacuum is established. The bath temperature should be 50° at the syrupy stage and 60° at dryness, at which it is kept till not more than one drop of distillate per minute falls at the adapter. 50 c.c. of water are added to the flask, the residue is dissolved and the distillation repeated, and this yet again. The distillate is freed from carbon dioxide by aeration and titrated with 0.1*N*-caustic soda, using 4 drops of 1% phenolphthalein as indicator. Results are quoted showing the amount of volatile acid in various

types of beer and in beer during fermentation. Steam distillation of beers which have been brought to p_H 3.0 give results approximating to those of the method described if 600 c.c. of distillate are collected from 100 c.c., but differences occur in some cases and the results of duplicate determinations are less consistent.

F. E. DAY.

Divinylethylene glycol as cause of the bitter taste in the disease of bitterness of wines. E. VOISENET (Compt. rend., 1929, 188, 1271—1273).—The compound isolated from bitter wine (B., 1929, 413) has now been identified with divinylethylene glycol, $CH_2 \cdot CH \cdot CH(OH) \cdot CH(OH) \cdot CH \cdot CH_2$ (Griner, A., 1893, i, 237). It would appear to be formed in the wine by the action of reductases on acetaldehyde.

R. K. CALLOW.

XIX.—FOODS.

Effect of dry skim milk on the water absorption of doughs and the plasticity of flour suspensions. J. L. ST. JOHN and C. H. BAILEY (Cereal Chem., 1929, 6, 140—150).—Addition of dry skim milk increases the water-imbibing capacity of dough so that it is necessary to add an equal weight of water to maintain the same degree of plasticity as measured by means of the power-driven dough mixer and wattmeter. Extensibility of dough measured with the Chopin extensimeter is not substantially affected by the inclusion of 10% or less of dry skim milk. The mobility of flour-water suspensions decreases with addition of dry skim milk. In order to maintain the same mobility, water must be added equal in weight to that of the milk added. The mobility of flour-water or flour-water-skim milk suspensions measured by Bingham and Murray's method increases with time during several hours. W. J. BOYD.

Granulation of flour and its relation to baking quality. C. B. KRESS (Cereal Chem., 1929, 6, 202—214).—Baking tests have been carried out on portions of flour separated on various sieves, No. 10—16. It is concluded that, on the whole, granulation of the flour makes little difference to its baking quality, the medium granulation giving a more even grain. The best loaf (volume, colour, texture, and grain) is made from the 30—40% of flour remaining after removing the coarsest and the finest flour. The fineness does not affect appreciably the rate of fermentation or development of acidity.

E. B. HUGHES.

Correlation between diastatic power of flour and crust colour in the test loaf, and its significance. M. J. BLISH, R. M. SANDSTEDT, and H. PLATENIUS (Cereal Chem., 1929, 6, 121—127).—When flours are baked by a fixed procedure, all factors being precisely controlled, depth of crust colour is positively correlated with residual sugar in the baked loaf on the one hand and with relative diastatic power and fermentation tolerance of the flour on the other. W. J. BOYD.

Quick viscosimetric method for measuring the staleness of bread. L. P. KARACSONYI (Cereal Chem., 1929, 6, 241—243).—The viscosity at 20° of a 10% suspension in water of finely-sieved crumb is measured in an Ostwald viscosimeter with slightly modified capillary. Examples quoted show a reduction of viscosity of about 35% when bread is stored for 48 hrs.

E. B. HUGHES.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 27, 1929.

L—GENERAL; PLANT; MACHINERY.

Economic factors in chemical plant location. C. W. CUNO (Ind. Eng. Chem., 1929, 21, 738—740).—Attention is called to the continual migration of industry in the United States and to the fact that location is usually more important than technical or commercial efficiency. Factors affecting location are enumerated.

C. IRWIN.

Fundamental calculations for the flue-gas drum dryer. K. D'HUART (Z. Ver. deut. Zucker-Ind., 1929, 79, 327—365).—An exposition of thermal calculations relating to revolving drum dryers in which the material is dried by direct contact with flue gas travelling in the same direction. Methods of calculating calorific values of solid fuels from easily obtainable analytical data are described, and also some simplified methods for calculating thermal efficiency.

J. H. LANE.

Spray drying and the drying of dairy products. J. E. NYROP (J.S.C.I., 1929, 48, 136—139 r).—The statement that the dehydration in a spray dryer is so rapid, requiring only a fraction of a second, that time reaction can be neglected, is negatived by calculations showing that the evaporation B_s in an efficient spray dryer, working under ordinary conditions as in a milk dryer, is $B_s = 0.9Sz$, where S is the surface in m^2 of 1 kg. of the sprayed product and z the drying time in hrs. When a Niro-atomiser is used it takes 20 sec. to evaporate 1 kg. The surface area of the sprayed product is without influence on the difference in temperature between the drying current and the particles of the product; a high velocity of the current in relation to the surface of the particles increases the difference in temperature, and this is decreased when the vapour pressure in the drying current is raised. Different types of atomiser are described. Nozzles give—up to a certain capacity and viscosity—a very fine spray, but reversible soluble powders are difficult to produce from concentrated emulsions. Rotating pipes do not give a fine spray, and a rotating funnel-shaped pipe (parallel with the axis) or a flat or bell-shaped disc is only able to atomise satisfactorily small amounts of not very viscous materials. With atomisers where the product is passed over rapidly rotating vanes a very large capacity and satisfactory results are obtained. The use of such dryers in preparing milk and cream powders, chocolate, etc. is discussed.

Incrustation of well-borings and its removal by chemical means. G. WIEGAND (Gas- u. Wasser-fach, 1929, 72, 741—744).—When the filter or metal lining of a deep well is corroded and encrusted by the action of hydrogen sulphide, the deposits can be removed by treatment with 30% hydrochloric acid. This acid

must be free from arsenic, and the hydrogen sulphide which is produced must be allowed to escape in such a way that no risk of poisoning can arise.

R. H. GRIFFITH.

Glass plates prolong life of distilling columns. A. A. BACKHAUS (Chem. Met. Eng., 1929, 36, 429).—Glass plates of both the bubbling-hood and perforated types used in the distillation of anhydrous solutions of alcohol and hydrogen chloride are illustrated. The column is made of lead-lined steel or cast-iron flanged sections, the joint with the glass being made with a lead ring. In this case lead plates lasted only a few months, whilst the lead lining is durable. C. IRWIN.

Industrial measurements. I. Weighing. E. P. PARTRIDGE (Ind. Eng. Chem., 1929, 21, 740—744).—For the weighing of raw materials hopper scales which release a hopper charge when a required weight has been added are useful. The telepoise conveyor scale and conveyor weightometer automatically integrate weights passing over a conveyor. For batch compounding, as in a glassworks, a special truck which is really a container mounted on a platform scale may be used. The poidometer gives automatic control to a continuous feed as in cement manufacture. The conveyor scale may be modified to weigh continuously a sheet product, e.g., paper or rubber. C. IRWIN.

Plastometer. E. KARRER (Ind. Eng. Chem. [Anal.], 1929, 1, 158—160).—The construction and operation of an automatic plastometer for measuring the plasticity of rubber are described with reference to detailed drawings for particulars of which the original must be consulted.

A. R. POWELL.

See also A., Aug., 903, **Centrifugal filtration device** (SKAU). **Apparatus for fractional distillation under vacuum** (ASHWORTH).

Vaporisation of complex mixtures. PODBIELNIAK and BROWN. **Waste-heat recovery.** GREGSON.—See II. **Drying and mixing viscous liquid.** JESSUP.—See XIII. **Filter cloths.** SANDERA.—See XVII.

PATENTS.

Regeneration of kieselguhr. A. M. DUPERU (U.S.P. 1,708,362, 9.4.29. Appl., 25.10.26).—Moist spent kieselguhr is mixed with dry regenerated kieselguhr and dried, pulverised, and calcined in air. The dust from the drying and burning kilns is collected in water, the dust water mixed with regenerated calcined kieselguhr, and the mixture filtered.

R. BRIGHTMAN.

High-pressure joint. F. H. BRAMWELL, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,722,623, 30.7.29. Appl., 23.7.26. U.K., 23.7.25).—See B.P. 249,001; B., 1926, 472.

Apparatus for mixing, compacting, degassing, or grinding viscid material. A. SONSTHAGEN and G. M. POVERUD (U.S.P. 1,722,115, 23.7.29. Appl., 26.8.27. U.K., 6.4.27).—See B.P. 279,707; B., 1923, 41.

Production of intimate mixtures of substances and of chemical products therefrom. J. W. SPENSLEY (U.S.P. 1,722,687, 30.7.29. Appl., 5.3.23. U.K., 4.4.22).—See B.P. 201,968; B., 1923, 962 A.

Centrifugal separator. E. B. MÖLBACH (U.S.P. 1,721,230, 16.7.29. Appl., 19.10.28. Nor., 29.10.27).—See B.P. 299,703; B., 1929, 498.

Manufacture of ultrafilter membranes. J. DUCLAUX (U.S.P. 1,720,670, 16.7.29. Appl., 30.8.23. Fr., 11.9.22).—See B.P. 203,714; B., 1924, 657.

Apparatus for charging furnaces, refuse destructors, producers, etc. WOODALL-DUCKHAM (1920), LTD., and F. A. HORNBY (B.P. 316,841, 15.12.28. Addn. to B.P. 305, 451).

[Ejector] means for producing foam, more particularly for fire-extinguishing purposes. EXCELSIOR FEUERLÖSCHGERÄTE A.-G., Assees. of C. WAGENER (B.P. 289,444, 26.4.28. Ger., 27.4.27).

Absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM A.-G. (B.P. 292,480, 18.6.28. Ger., 16.6.27).

Adsorption refrigerating apparatus. ELECTROLUX, LTD., From PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 315,292, 11.4.28).

Jaws of stone breakers or crushers. W. SOMERVILLE (B.P. 315,730, 12.4.28).

Mixing apparatus [for disinfecting grain etc.]. J. S. J. FRUERGARD (B.P. 315,742, 17.4.28).

Screening plant [with mass-balanced screens]. BAMAG-MEGUIN A.-G. (B.P. 311,219, 17.7.28. Ger., 7.5.28).

Pumps for forcing under pressure liquefied gases at low temperatures. L'AIR LIQUIDE SOC. ANON. POUR L'ÉTUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 302,681, 30.11.28. Fr., 20.12.27).

Hermetic closures for pressure vessels. K. BREDSCHNEIDER (B.P. 292,614, 22.6.28. Ger., 24.6.27).

Apparatus for generating vapours (B.P. 298,905).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Terminology in coal research. R. THIESSEN and W. FRANCIS (Fuel, 1929, 8, 385—405).—The nomenclatures proposed, respectively, by Stopes in England (B., 1919, 452 A) and Thiessen in America (B., 1925, 433) for distinguishing the types of coal present in a seam have been correlated. Vitrain corresponds with anthraxylon and clarain with attritus. The thin bright streaks in clarain, however, are anthraxylon. Durain has no counterpart in American coals. Fusain corresponds with mineral charcoal or "mother of coal" in America. The terms anthraxylon, attritus, and fusain may be used irrespective of the rank or geologic age of the coal. The terms vitrain, clarain, and durain should be limited to coals of bituminous rank. Of the

German terms, Glanzkohle and Mattkohle, the former may have been used for vitrain or clarain or both, whilst the latter is probably more nearly equivalent to durain.

A. B. MANNING.

Accuracy in coal sampling. C. F. KINGDON (J. Inst. Fuel, 1929, 2, 361—363).—The gross sample should be crushed to a maximum size of about $\frac{1}{8}$ in. diam. and then reduced in quantity to about 6 lb. by quartering or riffing. Considerable variation in results may obtain from dividing and mixing by hand, and a sampling apparatus is recommended. This consists essentially of a rotating cylinder inside a larger container; the inner cylinder has a cone-shaped cover provided with a slot through which a proportion of the material passes during the rotation, the remainder being rejected into the outer container. Moisture should be determined before the final grinding.

C. A. KING.

Comparative measurement of the plastic state of coals. F. SCHIMMEL (Brennstoff-Chem., 1929, 10, 319—321).—The methods of Foxwell (B., 1924, 4, 737, 738, 896) and of Agde and von Lyncker (B., 1929, 462) have been compared with coals from the Ruhr, Saar, and Saxon districts. Curves are obtained which show the change in resistance of a standard column of coal as it is heated, and of the rate at which a weighted needle can penetrate the coal as the temperature rises. The plastic ranges determined in these two ways are in very close agreement if the maximum values obtained are always taken to indicate the end of this range; this is a modification of Foxwell's method in which the whole duration of resistance has previously been measured.

R. H. GRIFFITH.

Modification of Parr's total carbon determination in coal. R. E. BREWER and E. P. HARDING (Ind. Eng. Chem. [Anal.], 1929, 1, 145—148).—The residue from a fusion of the coal sample with sodium peroxide and potassium chlorate or perchlorate as accelerator is dissolved in gas-free distilled water and the solution transferred to a modified Parr apparatus in which the carbon dioxide is liberated with hydrochloric acid. The difference between the volumes of the combined gases before and after absorption with caustic potash solution gives the volume of carbon dioxide generated. Suitable corrections are made for the carbon dioxide in the reagents and in inorganic combination in the coal, and tables are given for facilitating the temperature and pressure corrections for gas volumes and for calculating the weight of carbon per c.c. of carbon dioxide.

H. S. GARLICK.

Combined water content of coals of different ages. D. J. W. KREULEN and B. L. ONGKIEHONG (Brennstoff-Chem., 1929, 10, 317—319).—Dehydration and hydration curves have been constructed for a number of coals, and the method of Rabinovitch and Fortunatov (B., 1929, 1) has been also applied to calculation of the size of pore which absorbs the moisture. The coals were treated, under standard experimental conditions, with sulphuric acid of varying concentrations, and the loss of water was determined by titration of the acid. It is found that the distribution of the water varies with the geological age of the coal; in a brown

coal, for instance, it is adsorbed far more evenly by pores of different sizes than in an older coal, where it occurs chiefly in the smallest pores.

R. H. GRIFFITH.

Determination of the moisture content of coal and similar substances. M. MANNHEIMER (Ind. Eng. Chem. [Anal.], 1929, 1, 154—156).—The coal, ground to pass 60-mesh, is weighed and shaken for at least $\frac{1}{2}$ min. with sufficient anhydrous methyl alcohol to give a final water concentration not greater than 2%. The alcohol is sucked through a filter into a test-tube containing a float and the equilibrium temperature of flotation in the aqueous alcohol determined. From the equilibrium temperature, the amount of coal, and the quantity of methyl alcohol, the moisture content can be determined, the whole procedure taking less than 5 min. The method shows a small but fairly constant higher value for water content than that obtained by extraction with xylene, this difference varying slightly with length of time of extraction, final concentration of water in the alcohol, and species of coal. The values obtained agree with one another at least as well as those obtained by other methods.

H. S. GARLICK.

Extraction of mineral substances from coal. N. M. KARAVAEV and I. B. RAPOPORT (Izvestia Teplotekh. Inst. [Moscow], 1929, No. 5, 31—34).—It is shown that the usual methods of analysis of coals give results inaccurate with regard both to their mineral matter content and to the composition of the combustible material. Boiling a coal with dilute hydrochloric acid (1 : 10) extracts hardly any mineral matter, whilst hydrofluoric acid of the same dilution is much more effective, even in the cold, and dissolves any silicates present. Hydrofluoric acid has the additional advantage that there is greater certainty of the organic matter in the coal not being attacked. Any analytical errors due to the presence of moisture, which must not and cannot be removed by heating to high temperatures at which the organic matter may begin to decompose, can be corrected by treatment with hydrofluoric acid (1 : 10) in the cold for 24—48 hrs. Analyses of coal samples after heat treatment and hydrofluoric acid treatment showed differences in the carbon content up to 3% and of hydrogen up to 0.32%.

A. FREIMAN.

Fusain. J. D. DAVIS (Min. Congress J., 1929, 197—200; Fuel, 1929, 8, 375—379).—The literature on the occurrence, separation, physical and chemical properties of fusain is summarised.

A. B. MANNING.

Electrostatic method for determining fusain in bituminous coal. J. D. DAVIS and J. A. YOUNKINS (Ind. Eng. Chem. [Anal.], 1929, 1, 165—167).—The fusain in a 0.5-g. sample of 60-mesh air-dried coal is subjected to preliminary separation in an impure state by shaking with a mixture of gasoline and carbon tetrachloride of d 1.40—1.45, followed by centrifuging. The coal which floats is removed and the remaining impure fusain dried, weighed, and transferred to the tray of an electrostatic separator, where the fusain is removed from the electric field by a current of an inert gas, and the residue re-weighed. The completeness of separation is checked microscopically and the process

repeated if necessary. Practically complete separation is possible, although the results have a tendency to be slightly high due to mineral matter being carried over with the fusain. The electric field is produced by the output of a 500-watt high-tension transformer connected to a 110-volt supply, the secondary winding giving about 20,000 volts, rectified by a G.E.C. Kenotron valve. With electrodes $\frac{3}{4}$ in. apart a filament current of 4.5 amp. was adequate.

H. S. GARLICK.

Some properties of coal dust and pulverised coal. F. S. SINNATT (Fuel, 1929, 8, 362—370).—The utilisation of coal in the form of pulverised fuel has directed attention to certain properties, such as ease of grinding, proportion of fusain in the dust, the temperature of ignition and the speed and mechanism of combustion of the finely-divided material, the m.p. and behaviour of the ash, etc. Some recent investigations relating to these properties are summarised, and their bearing on practice is briefly indicated. Attention is directed to the possibility of a wide variation in the properties of the individual particles of the powdered coal.

A. B. MANNING.

"C.O.L." chamber ovens at the works of the Wandsworth, Wimbledon, and Epsom District Gas Co. C. M. CROFT (Gas J., 1929, 187, 251—256).—The plant consists of five settings of six chambers, each holding 3 tons of coal. A detailed description of the plant and its auxiliaries is given, and the results of tests with Durham and Yorkshire coals are shown. In a 28-day test with Durham coal, containing 2.65% of moisture, 31.38% of volatile matter, and 7.93% of ash, 4277 tons of coal, 503 tons of breeze, and 399 tons of steam were used, whilst the producers consumed 710 tons of coke; 73,093,000 cub. ft. of gas of 485.6 B.Th.U./cub. ft. were produced, equivalent to 85.25 therms per ton, and 13.59 cwt. of dry coke remained from each ton of coal. A 4-day test with Yorkshire coal gave 83.3 therms of gas and 13.84 cwt. of dry coke per ton of coal.

R. H. GRIFFITH.

Low-temperature coking of Chelyaba brown coals. N. N. ROGATKIN (J. Chem. Ind. Moscow, 1928, 5, 1278—1279).—The yield of liquid products increases but little over 400° and is maximal at 470°. Above 450° the yield of gas rapidly increases at the expense of that of the coke. Data concerning the composition of the brown coal and gas and the yields are given.

CHEMICAL ABSTRACTS.

Primary tar of Chelyaba coals. I. Y. POSTOVSKI (J. Chem. Ind. Moscow, 1928, 5, 1279—1280).—Rogatkin's tar (cf. preceding abstract) contained C 73.36, H 7.86, N 1.42, S 0.25, O 17.11%, and had d_{20}^{25} 1.0350, f.p. below 0°, mech. impurities 4.6, moisture 12.3, ash 2.46, phenols 40.0%, calorific value 8873 g.-cal. The tar is low in paraffin and high in phenol, and the tar water contains 0.3% of ammonia.

CHEMICAL ABSTRACTS.

Spontaneous combustion of [Japanese] coal. I. MIYAGAWA, M. YAMADA, and J. INABA (J. Fuel Soc. Japan, 1929, 8, 81—85).—Experiments to determine the liability of a coal to spontaneous combustion by its examination in an adiabatic calorimeter are described, and it is shown that coals requiring less than $4\frac{1}{2}$ hrs.

for the temperature to rise from 101° to 130° due to oxidation are the most liable to spontaneous combustion in the mine. It is also indicated that the α -compounds are the most spontaneously combustible. Examination of the oxidisable substance in coal by its reactions with iodine, chromic acid, sulphuric acid, hydrogen peroxide, ozone, and bromine show that, with the exception of the last-named, these reagents can be used to determine the liability to spontaneous combustion provided that conditions are carefully standardised.

C. B. MARSON.

Waste-heat recovery with particular reference to the carbonising and steel-making industries. W. GREGSON (J. Inst. Fuel, 1929, 2, 329—344).—Surplus heat may be recovered either by direct return to the furnace by means of regenerative settings or by external means, *e.g.*, waste-heat boilers. Regenerative recovery is limited by the volumes of ingoing air and outgoing products, and increase in the capacity of regenerators beyond this determined value is detrimental; further recovery can be obtained only by waste-heat boilers. In gas-works' retort practice both methods in conjunction are standard. The emission of heat from coke discharged from horizontal retorts represents as much as 25% of the total heat to the settings, and the steaming of vertical retorts has a distinct advantage in this respect. In steel-furnace work higher heat efficiency can be effected by feed-water heaters, but the capital and maintenance costs of the latter outweigh any advantage obtained by the additional heat recovery.

C. A. KING.

Action of hydrogen on coal. J. I. GRAHAM and D. G. SKINNER (J.S.C.I., 1929, 48, 129—136 T).—The early history of the hydrogenation of coal is outlined, and the work of Bergius is described. The paper records the results obtained by the authors on the hydrogenation of over thirty British coals in presence of phenol as a suspending medium. The products in each case have been separated into (A) portion insoluble in phenol, (B) portion soluble in phenol, insoluble in chloroform, (C) portion soluble in phenol and soluble in chloroform and boiling above 150°, (D) light fraction boiling below 150°, (E) water, (F) gases. In general it was found that coals of the parabituminous group (Seyler) give the highest yield of oil and smallest amount of residue. For comparison, the results of destructive distillation tests on each coal are given, and the increased yield of liquid products resulting from hydrogenation is clearly shown. Although Bergius has stated that any coal (of carbon content below 80%) may be hydrogenated almost completely, the authors consider that the composition and constitution of the coal are of considerable importance in regard to the degree of its conversion into oil by the action of molecular hydrogen. The properties of the products of hydrogenation are described in detail. The residue (a) is richer in carbon and usually poorer in hydrogen than the original coal. Decomposition of this product is observed at a temperature below 400°, and its stability is evidently dependent on the partial pressure of hydrogen in the surrounding atmosphere. On distillation to 600°, 3—4% by weight of gas is evolved, and of this 65—80% by vol. is hydrogen. Product B, which is an intermediate product in

the hydrogenation process, may contain 85—90% C, 4.3—4.7% H, and 0.8—6% O; the C/H ratio is in general about 19. The volatile matter is usually about 33%. On distillation it decomposes at a temperature considerably below that obtaining in the hydrogenation process; it is stable at temperatures above 250°, only in presence of a high concentration of hydrogen molecules, and is capable of further hydrogenation, when simpler products are obtained. Distillation at 600° yields 7—8% of oil and about 2—3% of gas very rich in hydrogen. Mol. wt. determinations by the b.p. method using phenol as solvent gave values of the order of 3000 for this product. Considerable loss in weight occurs when heated in air at 150°, indicating oxidation of carbon and hydrogen, but with little fixation as a solid complex such as occurs with the parent coal. In no case has product C (the tar oil), been found to consist of hydrocarbons only, oxygen being usually present to the extent of about 4%, whilst the C/H ratio is about 11.5. The iodine values of the fractions of this product and also of product D show that these possess a fair degree of saturation. The water produced in hydrogenation depends on the oxygen percentage of the original coal. The quantity of gas produced in hydrogenation is rather larger than that from destructive distillation at the same temperature. Methane may be present in amount up to 13% of the original coal. Unsaturated hydrocarbons are never present. It is shown that the greater part of the hydrogen consumption takes place during pyrogenic decomposition of certain of the coal constituents, although some absorption also takes place by the direct action of hydrogen. In an attempt to discover which constituents of the coal are most amenable to the action of hydrogen, vitrain from the Slate Seam, Warwickshire, was extracted with phenol for a prolonged period, and the residue subjected to hydrogenation in the usual manner. Less residue (A) was yielded than in the case of the original coal, and thus it seems that the extract would not be readily hydrogenated. On the other hand, extraction of another coal with pyridine, and subsequent hydrogenation of the residue, left a residue, A, practically equal in amount to that obtained by direct hydrogenation of the coal, indicating that in this case the extract must be almost completely hydrogenated. The conclusion is reached that, in the application of catalysis to the hydrogenation of coal or its products, the prospect of the process becoming a commercial success is greatly enhanced. The use by Bergius of iron oxide paste is considered to have a catalytic effect. It has been found that other substances, such as nickel oxide and ammonium molybdate, have a greater effect than has iron oxide.

Calculations on a lignite dryer with psychometric chart. I. LAVINE and R. L. SUTHERLAND (Chem. Met. Eng., 1929, 36, 425—428).—A chart gives the relations between temperatures from 40° to 680° F. and humidity, latent heat, total heat, etc. for air and gases (*e.g.*, flue gases) having similar specific heats. The application of these curves to the problem of drying lignite with 36% of moisture by hot flue gases is given in detail. This can be dried to a 20% moisture content, which amount is in equilibrium with the atmosphere

under average conditions. In making the calculation the operations of heating the lignite and adiabatic evaporation are considered separately. It is assumed that the gas on leaving the dryer is saturated. The relative humidity of the gas should be reduced along the dryer in the same ratio as the moisture in the lignite to maintain a uniform humidity gradient. This is obtained by the use of a countercurrent. The use of the minimum quantity would produce disintegration of the lignite, and a much larger quantity of gas must be recirculated to maintain desirable humidity conditions.

C. IRWIN.

Laboratory tests of the chemical activity of ashes of solid fuels in relation to refractories. O. V. NEFEDIEVA and N. G. PACUKOV (*Izvestia Teplotekh. Inst. [Moscow], 1929, No. 5, 20—30*).—Two methods of testing refractory bricks as to their behaviour towards the chemical activity of ashes and slags was used. One method was to determine the fusibility of mixtures of refractories and ashes; the other was a modification of Nesbitt and Bell's method (*J. Amer. Ceram. Soc., 1923, 6, 296*) of surface interaction. According to the first method the sample of solid fuel was finely powdered (900-mesh/sq.cm.) and burnt. The ash was then powdered and samples were taken for fusion determinations. From the refractories small samples were taken from different parts and the powder was moistened with water, and, after addition of a 10% dextrin solution, made up into small cones for fusion experiments. Mixtures of refractory and ash were next prepared (3:1, 1:1, and 1:3) and each was thoroughly rubbed together in an agate mortar; a portion was then subjected to the fusion tests, and the rest heated in a current of oxygen, again thoroughly powdered, and oxidised in an electric oven for 1 hr. Conical samples for fusion experiments of both the mechanical mixture and the heated mixture were prepared as described above. Curves showing the relation between constitution of the mixture and temperature of fusion exhibited in all cases, except one, a rapid decrease of fusion point to a 25% ash content, after which the decrease was more gradual and regular. It was also found that refractory bricks with a high silica content are more resistant. In the surface-interaction method a brick sample was weighed and measured, and the size of the hollow in one of its sides was determined by means of a volumometer, using pure sand. Its porosity was also determined. The hollow was then filled with ash and slag and the brick with the hollow upwards was heated for a known period at a known temperature. After being cooled, weighed, placed with the hollow downwards on a fire-resisting crucible, to allow any molten ash which might escape to collect in the crucible, it was placed in an oven and kept for the same length of time at the same temperature. After cooling it was again weighed and measured, and from the measurements of the hollow the degree to which the brick was attacked by the given ash sample could be determined.

A. FREIMAN.

Adsorptive characters of activated ash-free charcoals. E. LANDT and K. K. BHARGAVA (*Z. Ver. deut. Zucker-Ind., 1929, 79, 470—484*).—Inactive char produced from pure sugar at 250—300° was activated by heating for 7 hrs. at constant temperatures ranging

from 550° to 1050° in a regulated current of air in an electric tube oven. The yields of activated products ranged from 27 to 70% of the inactive carbon used, and for any given temperature of production the activities varied inversely with the yields. Towards iodine the products were as active as good commercial decolorising carbons. Their behaviour towards sodium hydroxide and hydrochloric acid varied widely according to the conditions of production. Propionic acid was adsorbed by all the products more readily than acetic acid, and the latter more readily than formic acid (Traube's rule). None of the products showed more than feeble adsorptive action on the colouring matters of molasses.

J. H. LANE.

Mechanical separation and flotation of Koureika's graphite. V. I. TRUSHLEVITSCH (*Trans. Inst. Econ. Min. Met. Moscow, 1928, No. 39, 47 pp.*).—A study of the conditions for the elimination of sulphur and ash from graphite from Touroukhansk, Siberia.

CHEMICAL ABSTRACTS.

Gas from brown coal. F. FRANK (*Z. angew. Chem., 1929, 42, 773—776*).—The carbonisation and gasification of brown coal under varying conditions and the production of further quantities of gas by cracking the tar vapours evolved have been studied on a laboratory scale. The apparatus comprised an electrically-heated retort, through which could be passed an accurately-measured quantity of carbon dioxide, steam, or other gas or vapour, an electrostatic purifier for the precipitation of tar fog, ordinary gas purifiers, and a condensing system. The results indicate the possibility of producing town's gas by the gasification of brown coal, combined with the cracking of the volatile products evolved by passing them over the highly reactive brown coal coke at a suitable temperature. The process can also be made to yield appreciable quantities of motor spirit.

A. B. MANNING.

[Oxides of nitrogen in] coke-oven gas. P. SCHUF-TAN (*Z. angew. Chem., 1929, 42, 757—760*).—The presence of nitrogen peroxide in coke-oven gas has been established and its concentration determined colorimetrically by using a solution of *m*-phenylenediamine. Nitric oxide, however, formed the greater proportion of the oxides of nitrogen present in the gas. This was determined by the addition of oxygen, whereby it is partly converted into nitrogen peroxide. From the volume of oxygen added, the time allowed for the reaction, and the amount of peroxide formed, the original concentration of the nitric oxide could be calculated. The concentrations found were of the order of 0.0001—0.003%. The results were confirmed by hydrogenation of the nitric oxide over copper and determination of the ammonia formed. The presence of the nitric oxide was traced to the flue gases, which may contain up to 0.2—0.4%, and are drawn into the chambers by the suction of the exhausters. The formation of Roussin's salts by the action of the nitric oxide on ferrous sulphide in the presence of alkali has been observed in coke-oven plants.

A. B. MANNING.

Carbon monoxide in gases. H. KEMMER (*Gas- u. Wasserfach, 1929, 72, 744—749*).—If the removal of carbon monoxide were necessary in order to make coal

gas non-poisonous, three methods might be available: (1) absorption, (2) catalytic process, or (3) condensation. The practicability of these alternatives and their costs are reviewed, and it is concluded that absorption with ammoniacal copper formate solution, catalytic conversion into methane, and fractional distillation are all technically possible.

R. H. GRIFFITH.

Separation and utilisation of the constituents of coke-oven gas. J. BRONN (*Z. angew. Chem.*, 1929, 42, 760—768).—The composition of coke-oven gas and the separation of its constituents by fractional condensation at low temperatures are discussed. The most recent form of Linde's apparatus is described in detail. In this the gas, freed from carbon dioxide by washing successively with water under 12 atm. pressure and with caustic soda, is subjected to a preliminary cooling to -30° to -50° by heat interchange with the gases issuing from the apparatus. After entering the actual gas separation apparatus it undergoes further cooling by the already separated gases, whereby all the constituents are condensed except the hydrogen, nitrogen, and part of the carbon monoxide. The residual gas, still under a pressure of about 10 atm., then passes through tubes immersed in a bath of liquid nitrogen boiling under reduced pressure. This brings about condensation of the carbon monoxide and almost all the nitrogen, leaving hydrogen of 98—99% purity. If the gases are required for the synthesis of ammonia the process is slightly modified, the residual mixture of hydrogen, nitrogen, and carbon monoxide being washed with a current of liquid nitrogen under conditions which completely remove the carbon monoxide and leave a mixture of hydrogen and nitrogen in the requisite 3:1 ratio. Various possibilities for the economical utilisation of the hydrocarbons, e.g., as motor fuels, in the synthesis of alcohols, oils, etc., are discussed.

A. B. MANNING.

Removal of sulphur compounds from gases and recovery of the sulphur. G. LORENZEN (*Z. angew. Chem.*, 1929, 42, 768—773).—The various processes for removing sulphur compounds from gases are briefly described and compared, and the present position of the problem is discussed. One of the most recent developments is that due to Petit, in which the gas is washed with a solution of potassium carbonate, this being regenerated by treatment with carbon dioxide to remove the hydrogen sulphide, and then by heating to remove the excess carbon dioxide. The hydrogen sulphide mixed with carbon dioxide is passed through an iron oxide purifier and the sulphur thereby recovered. With a sulphur content in the gas of 7—8 g./m.³ the value of the sulphur recovered just pays the cost of its removal.

A. B. MANNING.

Simplified iodine pentoxide apparatus for determination of carbon monoxide in flue gas. F. E. VANDAVEER and R. C. GREGG (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 129—133).—The apparatus and procedure are described in detail; carbon monoxide is determined by titration of the iodine liberated, an accuracy of 0.002% being claimed. The gas sample passes successively through two chromic acid towers, solid potassium hydroxide, an iodine pentoxide tube, and a solution of potassium iodide. Carbon dioxide, oxygen, hydrogen,

methane, ethane, hydrogen sulphide, sulphur dioxide, nitrogen, and oxides of nitrogen when present in small quantities do not interfere. If small amounts of formaldehyde are present one of the chromic acid towers must be heated at 100° . Correct results cannot be obtained if ethylene is present.

J. S. CARTER.

Air-gas ratio apparatus. C. Z. ROSECRANS (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 156—158).—Illustrations and wiring diagrams of a gas analysis apparatus of the thermal conductivity type for determining the proportion of fuel gas in air are given and briefly described. It comprises two thermal conductivity tubes, one for dry air as standard and one for the gas mixture, mounted in a constant-temperature block controlled by a bimetallic thermostat and combined in an improved Wheatstone bridge circuit. Readings are taken for pure air and pure fuel gas of varying compositions, and by joining these points on a graph by a straight line the composition of any fuel gas-air mixture may be quickly determined.

A. R. POWELL.

Nomography. O. LIESCHE (*Chem. Fabr.*, 1929, 305—306).—A nomogram for determining the heat lost by incomplete combustion from the analysis of the flue gases is reproduced.

A. R. POWELL.

Determination of the sp. gr. of asphalt by means of a hydrometer. H. R. BERRY, E. H. SMITH, and F. R. LANG (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 164).—The asphalt samples are heated to a temperature slightly above that of an oil-bath, maintained within the range 143.3 — 148.9° ($\pm 0.56^{\circ}$), and poured into metal cylinders $6 \times 1\frac{1}{4}$ in. immersed therein. A thermometer is placed directly in the asphalt, and when a constant temperature is reached a 4-in. A.P.I. scale hydrometer is floated therein and the gravity and temperature are recorded. A table gives the conversion directly into sp. gr. The hydrometer method gives results which agree closely with those obtained by the pycnometer method.

H. S. GARLICK.

Base combination and base exchange with humic acids. H. HEIMANN (*Brennstoff-Chem.*, 1929, 10, 301—303).—Humic acids obtained from peat, brown coal, sucrose, or quinol exhibit base-exchange properties which differ slightly from those shown by zeolites or by permutits; this is due to the fact that the free humic acid can exist as such, but free "permutit acid" cannot. The behaviour of humic acid with a number of salt solutions is discussed, and the changes which take place are differentiated from those due to adsorption.

R. H. GRIFFITH.

Production of gaseous, liquid, and solid hydrocarbons from methane. II. Action of the spark discharge on methane. H. M. STANLEY and A. W. NASH (*J.S.C.I.*, 1929, 48, 238—242 T).—The action of the spark discharge between copper, aluminium, and iron electrodes on methane, at atmospheric pressure and higher, is found to result in a variety of processes the extent and course of which are influenced by a number of factors such as current, spark gap, electrode material, but above all by the duration of sparking. The main products of the reaction are: (a) Gaseous: acetylene (up to nearly 14% of reaction gas), and smaller quan-

ties of ethylene and some diacetylene; (b) liquid: light oil, highly unsaturated with a pronounced tendency to polymerisation to resins. Hydrocarbons containing the group CH: appear to be present with only comparatively small amounts of benzene; (c) chloroform-soluble tar, from which small amounts of naphthalene and acenaphthylene were isolated; (d) resinous substances, insoluble in chloroform; (e) free carbon which under certain conditions may be obtained in light, flocculent form, resembling carbon black, in yields of nearly 20%. Acetylene appears to be the main primary product of the reaction, the higher hydrocarbons being probably formed by the further action of the discharge on the acetylene-hydrogen mixture produced. Longer periods of sparking favour the formation of liquid and solid products, whilst higher current discharges tend to produce greater quantities of flocculent carbon. The use of different electrode materials as well as of increased pressures up to 5 atm. did not materially alter the course of the reaction or the type of products. The formation of long carbon rods on the electrodes, thus reducing the electrode distance, was one of the experimental difficulties encountered.

Thermal production of acetylene from methane. K. PETERS and K. MEYER (Brennstoff-Chem., 1929, 10, 324—329).—The decomposition of methane has been studied in an apparatus consisting of a tungsten spiral, of 0.5-mm. wire, 3 mm. in width, which is heated electrically and carried by two iron tubes through which the gas is passed; the whole is mounted in a glass flask from which the products are drawn and examined. Temperatures are measured approximately by means of an optical pyrometer. The life of the wire is limited by the gradual formation of tungsten carbide, which is very brittle. Experimental data are summarised for 44 runs, showing temperature, pressure, gas velocity, time of heating, and production of oil and acetylene. It is found that as the yield of acetylene increases to a maximum, at the highest temperatures and under reduced pressure, the formation of oil falls to nil; similar results are obtained with coke-oven gas, and curves are given showing the range of conditions necessary for production of carbon, benzol, or acetylene from methane or from coal gas. A similar apparatus has been used to demonstrate the formation of acetylene by a single passage of methane through an electric arc, with a time-contact of less than 1/10,000 sec.; only very small amounts of acetylene are produced from naphthalene under the same conditions.

R. H. GRIFFITH.

Sulphur content of Transvaal oil shales. I. J. BLOM (Brennstoff-Chem., 1929, 10, 307—309).—Oil shales from Ermelo and Wakkerstroom contained total sulphur 0.09—0.77%, sulphide sulphur 0.02—0.36%, and organic sulphur 0.07—0.53%; the results are, in general, lower than those for American shales (cf. Harding and Thordarson, Ind. Eng. Chem., 1926, 18, 731). Determinations of sulphates and phenol-soluble sulphur are recorded, and other analytical data are tabulated in full.

R. H. GRIFFITH.

Vaporisation of complex mixtures [of petroleum hydrocarbons]. W. J. PODBIELNIAK and G. G. BROWN

(Ind. Eng. Chem., 1929, 21, 773—779).—Computations have been made on the basis of Raoult's law to give comparisons between the efficiencies of continuous and batch distillation applied to petrol. If w is the amount of liquid in mols. and x and y are the compositions of the liquid and of the vapour in mol. fractions, then $\log w_2/w_1 = \int_{x_2}^{x_1} dx/(y-x)$, either in the case of a continuous distillation at fixed temperature or of an infinitesimal step in a batch distillation. For a complex mixture, integration can only be performed by giving final values to the steps. Experimental results were also obtained and compared with the calculations. It was found that the deviations from Raoult's law (for hydrocarbons up to hexane) were within the limits required for calculations for engineering practice. For continuous distillation a series of equations giving the relationship between mols. vaporised and partial pressure for each component are used. It is shown that the batch method gives the more efficient fractionation in most cases. The detailed conclusions summarised in this form are independent of the validity of Raoult's law.

C. IRWIN.

Colloid-chemical factors in the formation and separation of crude oil emulsions. A. LOTTERMOSER and N. CALANTAR (Kolloid-Z., 1929, 48, 362—377).—The effectiveness of a number of substances in bringing about the separation of crude oil emulsions into their components has been examined by shaking the emulsion with a small quantity of the substance for 5 min. and observing the clarity of the oil layer after keeping it for a week. Among the best substances for this purpose are aniline, phenol, dihydroxystearic acid, carbamide, pepsin, and saponin. The effect of casein as an emulsifying agent has been studied, and the 0.2% of casein recommended by Seifriz (B., 1925, 556) is found to be insufficient. Casein is not regarded as a good emulsifier. Generally oil-in-water emulsions are formed, but in the presence of hydrophobic colloids and also in the emulsification of unpurified oil at certain concentrations, both kinds of emulsions are produced. Hard asphalt and petroleum tar are hydrophobic colloidal emulsifiers in petroleum which favour the formation of a natural water-in-oil emulsion; they lower the interfacial tension between oil and water, and consequently accumulate at the boundary with the formation of an elastic membrane; in addition, they raise the viscosity of petroleum, thus increasing the stability of natural petroleum emulsions. Hard asphalt helps to produce more stable emulsions than petroleum tar. Natural petroleum emulsions can be separated into their components by extraction of the tar by means of alcoholic sodium hydroxide, by adsorption of the asphalt substances by means of fuller's earth, or by precipitation of the asphalt and tar by anhydrous stannic chloride. Fractions of petroleum of low b.p. are the least readily emulsified, owing to their poverty in colloidal emulsifiers and to the difference in the density of the two phases. Sapamine acetate, hydrochloride, and lactate are unsuitable for the emulsification of petroleum or for the separation of the emulsions.

E. S. HEDGES.

Purification of crude benzol by means of sulphur. R. ULOTH (Brennstoff-Chem., 1929, 10, 297—301).—

A crude benzol from a coke-oven plant, which contained 0.324% S, was heated in an autoclave with sulphur. The liquid product thus obtained contained only traces of free sulphur, and the combined sulphur was only about 50% of that originally present, being almost entirely in a small quantity of pitch that is formed. As the temperature of the reaction is increased, the removal of unsaturated compounds, as measured by loss on washing with sulphuric acid, becomes more nearly complete; changes begin at 110°, and are almost ended at 270°. Differences in pressure have only a small effect on the reaction, but a minimum quantity of sulphur, generally 1–2%, is necessary. Experiments were carried out similarly with cyclohexene, thiophen, cyclopentadiene, and indene; all except the first became deeply coloured and produced hydrogen sulphide when heated with sulphur. cyclohexene is polymerised by heating in the presence of copper at 150° to a dark, viscous mass, no benzene being produced; in absence of copper, this reaction commences only at 200°. R. H. GRIFFITH.

Naphthenesulphonic acids [in oil refining]. S. PILAT and E. DAVIDSON (Przemysl Chem., 1927, 11, 141–146).—Compounds, having formulæ approximating to $C_{24}H_{33} \cdot SO_3H$, which do not lose the sulphonic group even on prolonged boiling with alcoholic alkali, are formed in the acid refining of oils. The sodium salts, even in fairly acid solutions, produce a foam in aqueous solution. CHEMICAL ABSTRACTS.

Volumetric determination of sulphur in crude petroleum. G. WOODWARD (Ind. Eng. Chem. [Anal.], 1929, 1, 117–118).—Enough oil is burned in the bomb to produce 0.03–0.25 g. of sulphuric acid. The contents are washed out using the minimum quantity of water, and a small amount of potassium iodide solution is added as indicator. The solution is evaporated to a volume slightly less than 50 c.c. with sufficient powdered aluminium (approx. 0.01 g.) to effect decolorisation, and then sufficient 95% alcohol added to bring it to a 50–70% alcohol solution. Lead nitrate solution (0.02N), previously standardised against 0.1N-sulphuric acid, is then run in until a permanent yellow colour is produced. With crude oil where the amount of sulphuric acid formed was 0.1–0.25 g. the deviation between the results of this and the barium sulphate methods did not exceed 1.8%. H. S. GARLICK.

Determination of sulphur in petroleum oils. R. C. GRIFFIN (Ind. Eng. Chem. [Anal.], 1929, 1, 167–169).—In the usual bomb method for determining sulphur in petroleum oils part of the sulphur is in most cases oxidised to a sulphonic acid or similar substance, of which the barium salts are soluble in water, with consequent low results in the determination. The sulphur of the sulphonic acid may be recovered as sulphuric acid by refluxing with concentrated hydrochloric acid. Both the usual bomb and the lamp methods give low results with oils containing mercaptans, which in the bomb are largely oxidised to sulphonic acids. When burned in the lamp they form some other compound than sulphur dioxide or trioxide and are lost. The determination of sulphur in heavy crudes by the lamp method by diluting with kerosene and burning the mixture is unreliable.

For very heavy crudes the recovery of sulphur by this method may be as low as 65%. H. S. GARLICK.

Sp. heats of mineral oils. L. M. HENDERSON, S. W. FERRIS, and J. M. McILVAIN (Ind. Eng. Chem. [Anal.], 1929, 1, 148–151).—The sp. heats between 37° and 250° of a wide range of mineral oils were determined in a calorimeter in which the electrical energy required to raise the temperature of a given weight of oil a definite number of degrees in a definite length of time under controlled conditions was measured. The results show that the sp. heat at elevated temperatures can be calculated with the aid of the equation of Fortsch and Whitman (B., 1926, 810) with sufficient accuracy for engineering requirements. H. S. GARLICK.

Analysis of motor fuels containing alcohol. A. NOLL (Z. Spiritusind., 1929, 52, 242–243, 247–248).—To 10 c.c. of the fuel in a flask graduated from 100 to 110 c.c. at the neck, calcium chloride solution (d 1.3) is added, the mixture shaken for 15 min., and made up with calcium chloride solution to 110 c.c. The volume of the hydrocarbons is measured on the graduated neck, and the water and alcohol are determined by difference. A further sample (100 c.c.) is twice extracted with calcium chloride solution in a separating funnel, the extracted alcohol determined by distillation, and the residual hydrocarbons are dried over ignited sodium sulphate and filtered. A portion (10 c.c.) is shaken in a 100–110-c.c. flask for 15 min. with 50 c.c. of a mixture of sulphuric acid and oleum or nitric acid (4:1 by vol.). The contents are made up to 110 c.c. with sulphuric acid and the proportion of paraffins is read, the benzene being obtained by difference. The method is shown to give consistent results with mixtures containing known proportions of benzene, petrol, and alcohol, but it is pointed out that since either hydrocarbon may be present in commercial samples of the other the method is less accurate in determining the proportion of commercial constituents in a mixed fuel. F. E. DAY.

Comparison of McIlhiney's and Rosenmund's methods for determining unsaturated linkings in vaselines. P. AGOSTINI (Annali Chim. Appl., 1929, 19, 241–253).—With the normal hydrocarbons, octylene and hexadecylene, these two methods (B., 1900, 176; 1902, 1542; 1923, 798, 1151; 1925, 214) give virtually identical bromine values. With amylene a small proportion, and with diisobutylene a larger amount, of hydrobromic acid is formed by the McIlhiney method; with diisobutylene, Rosenmund's procedure results in the formation of substitution products and gives values above the theoretical ones. With most vaselines, hydrogen bromide is formed by the McIlhiney method, which gives sensibly the same bromine values as the Rosenmund method, so that it seems that dibromopyridine sulphate solutions also yield substitution products in such cases. The McIlhiney method is preferred. T. H. POPE.

Use of mixtures of petroleum products and creosote for preservation of timber. A. I. VORONOV (J. Chem. Ind. Moscow, 1928, 5, 1227–1230).—The conditions of formation, particularly with Russian

heavy oils and mazouts, of the undesirable tarry precipitate on admixture with creosote were examined.

CHEMICAL ABSTRACTS.

Cylinder stocks of high quality from lubricating oil residues. V. L. GURVICH and M. A. PENGU (*Neft. Choz.*, 1928, 15, 55—64).

See also A., Aug., 903, **Apparatus for fractional distillation under vacuum** (ASHWORTH). 925, **Conversion of naphthenic acids into naphthenes** (KOMPPA). 948, **Determination of ethylene** (MORRIS).

Determination of phenols. SHAW. **Humic acids.** FUCHS and STENGEL.—See III.

PATENTS.

Fuels and firelighters. J. THIECKE, MINIMAX A.-G., and H. HERZOG (B.P. 315,232, 10.1.28).—A fuel or fuel-igniting substance is made by moulding into a suitable shape a mixture of ammonium nitrate and an organic substance, *e.g.*, charcoal, together with igniting means, *e.g.*, a mixture of potassium chloride, antimony sulphate, and lead peroxide, the whole being given a waterproof coating of paraffin or shellac. A. B. MANNING.

Fuel distillation. E. H. THIERRY and F. B. GRANT (B.P. 314,782, 2.4.28).—The fuel, *e.g.*, a briquetted mixture of anthracite duff and bituminous coal, is carried on trucks through a retort comprising two horizontal, parallel, and relatively long passages connected at the ends by bridging conduits and forming a closed circuit through which steam or other fluid medium is circulated in the direction opposed to the motion of the fuel. Two branch passages at one end of the loop formed by the retort are provided with chambers closed by trap doors through which the fuel enters and leaves the retort. At the other end of the loop the retort is provided with a superheating jacket in which the gaseous products obtained from the carbonisation of the fuel are burnt. These are withdrawn from the retort near the exit end and pass through a scrubber before being burnt. A. B. MANNING.

Apparatus for production of carbon black. R. H. UHLINGER, Assr. to THERMATOMIC CARBON Co. (U.S.P. 1,718,720, 25.6.29. Appl., 22.11.24).—A number of decomposing furnaces are connected to a conduit which conducts the mixture of gas and carbon black to a separating means and conveys the separated carbon black therefrom. The connexions from the furnaces to the conduit are provided with means for spraying the mixture of hot gases and carbon black with a cooling liquid. The arrangement is such that the mixture of gases and carbon black from at least one furnace enters the portion of the conduit containing separated carbon black and comes into intimate contact therewith. A. B. MANNING.

Production of coloured smoke clouds. J. C. SAVAGE (U.S.P. 1,716,797, 11.6.29. Appl., 3.4.24).—An oil-soluble colouring agent, *e.g.*, a dye, anthracene, or iodine, is incorporated with a light mineral lubricating oil, and the mixture ejected into the air, in the vapour state. Trichloroethylene or carbon tetrachloride may be added to the oily mixture. F. G. CLARKE.

Production of carbon monoxide. W. C. ARSEM, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,719,867, 9.7.29. Appl., 27.4.25).—A mixture of carbon dioxide and hydrogen is heated to 1000—1200° in the presence of a catalyst, and is then rapidly cooled to below 100° and the condensed water removed; the alternate rapid heating and cooling of the gaseous mixture is continued for the production of further quantities of carbon monoxide. A. B. MANNING.

Purification of gases in continuous working of plants for manufacture of light hydrocarbons. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (SOC. INTERNAT. DES PROC. PRUDHOMME-HOUDRY) (B.P. 295,272, 7.5.28. Fr., 8.8.27).—In processes for the hot purification of gases by passing over agents such as the oxides of nickel, which can be regenerated in known manner (*cf.* B.P. 274,846; B., 1929, 4), residual traces of sulphur are removed in a small supplementary purifier charged with a metallic purifying agent, *e.g.*, copper or its oxide, the sulphide of which is stable at the working temperature (approx. 250°). A. B. MANNING.

Apparatus for gas analysis. C. D. ZIMMERMAN (U.S.P. 1,719,864, 9.7.29. Appl., 17.1.27).—A number of gas burettes, each connected with its own series of absorption pipettes, are mounted on a stand and arranged to draw samples of gas simultaneously from different points of a gas passage or passages. A manifold connects the lower ends of the burettes to a liquid-levelling bottle, which, when raised, displaces gas simultaneously from each of the burettes into the corresponding pipette for the absorption of one of the constituents, and, when lowered, simultaneously withdraws the residual gas into its corresponding burette. A. B. MANNING.

Treatment of (A) tar or (B) pitch. O. D. CUNNINGHAM, Assr. to P. C. REILLY (U.S.P. 1,711,448—9, 30.4.29. Appl., 27.4.25).—Coal tar or pitch is heated to 160—170° with excess of sulphuric acid, run into water, and the product washed and neutralised to obtain material for decolorising or for use as filler in rubber or pigment mixes. R. BRIGHTMAN.

Emulsions of mixtures of pitch and bitumen. C. H. THOMPSON and W. J. MCGIVERN (B.P. 315,057, 23.5.28).—A mixture of bitumen and stearine pitch, containing a large proportion of the latter, preferably 40%, is emulsified with a dilute alkaline solution. A solution containing $\frac{1}{2}$ pt. of caustic soda in 15 pts. of water is suitable for the emulsification of 50 pts. of the mixture. China clay, ground limestone, etc. may be added as a filler. A. B. MANNING.

Manufacture of infusible asphalt masses of high elasticity. R. LICHTENSTERN (U.S.P. 1,720,487, 9.7.29. Appl., 28.4.28. Austr., 19.4.27).—Asphalt, to which not more than 10% of zinc, ferric, or aluminium chloride has been added, is heated to 300°, tung oil, not more than the amount of asphalt, is added, and the heating continued until coagulation occurs. T. A. SMITH.

Apparatus for mineral oil distillation. A. E. PEW, JUN., and H. THOMAS, Assrs. to SUN OIL Co. (U.S.P. 1,719,235, 2.7.29. Appl., 13.3.25).—Lubricating oil is distilled by means of mercury vapour which is used to

heat a thin wall over which the oil is flowing. Several mercury condensing chambers are supplied from one mercury vapour generator, and the oil is passed in series through the units heated by these chambers.

T. A. SMITH.

Cracking oil in liquid phase. J. C. BLACK (U.S.P. 1,720,070, 9.7.29. Appl., 26.10.26).—Oil is passed, under pressure, through tubes arranged in series in a furnace setting. The temperature of the oil is controlled by injecting additional oil at points along the tube, where pyrometer indicators show this to be necessary. Oil from the cracking tube is discharged into a space at the bottom of a dephlegmator column, whence the liquid fractions are passed through heat exchangers to preheat the cracking stock.

T. A. SMITH.

Cracking hydrocarbon oils. H. L. PELZER, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,718,375, 25.6.29. Appl., 11.4.28).—High-sulphur crude oil is cracked in the vapour phase. A mixture of caustic alkali with the crude oil is added to the resultant vapours during a scrubbing operation to separate tar. The remaining vapour mixture is refluxed and the vapours heavier than those suitable as components of gasoline are condensed and returned to the cracking operation. The tar separated from the product of the cracking and from the crude oil, including any unconsumed alkali and its reaction products, is discharged directly from the scrubbing operation.

H. S. GARLICK.

Cracking of hydrocarbon oils. H. L. PELZER, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,719,486, 2.7.29. Appl., 16.4.27).—In cracking stills in which the oil is circulated through a shell containing a bed of finely-divided material, and through a heating coil, the former rests on a screen, which is supported by a grating built of strips on the edges of which the screen rests.

T. A. SMITH.

Decomposition of hydrocarbons into hydrocarbons containing fewer carbon atoms. A. J. VAN PESKI (B.P. 315,193, 18.1.28).—The hydrocarbons are passed over metallic carbides, or metallic compounds which form carbides under the conditions of the reaction, at elevated temperatures and in the absence of hydrogen or gases capable of yielding hydrogen. Thus, by passing propane over granulated calcium carbide, or molybdenum mixed with pumice, at 600–800°, it is decomposed into ethylene and methane without deposition of carbon or loss of activity of the catalyst. Under similar conditions gas oil, b.p. 220–340°, gives a gas containing a high percentage of unsaturated hydrocarbons and a smaller or larger amount of a liquid condensate of b.p. 60–180°.

A. B. MANNING.

Refining of petroleum. E. B. PHILLIPS and J. G. STAFFORD, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,709,149, 16.4.29. Appl., 7.8.23).—The oil is agitated with sulphuric acid, and the mixture is fed on the upper surface of a rapidly rotating disc, from the edge of which it is thrown in a thin, uniform film against baffles on the walls of the vessel. The sludge is separated by gravity and the oil agitated with clay or fuller's earth and filtered.

R. BRIGHTMAN.

Refining petroleum oil. R. CROSS, Assr. to CROSS DEVELOPMENT CORP. (U.S.P. 1,718,218, 25.6.29. Appl.,

17.12.24).—A suitable hydrocarbon stock is treated successively with a sodium plumbite solution and with sulphuric acid before being cracked.

H. S. GARLICK.

Purifying hydrocarbon oils. L. LINK and M. B. AMIS, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,718,714, 25.6.29. Appl., 9.12.25).—An uncracked light hydrocarbon distillate containing difficultly removable corrosive sulphur is brought into intimate contact with the spent alkali from the treatment of a light fraction obtained from a cracking process.

H. S. GARLICK.

Preparing hydrocarbon products. J. SIMPSON, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,781,713, 25.6.29. Appl., 30.1.24).—A naphtha distillate containing corrosive sulphur is subjected to the action of a sodium plumbite solution which has already been used for treating a sour naphtha distillate.

H. S. GARLICK.

Treating hydrocarbon oils. C. J. GREENSTREET, Assr. to GASOLINE CORP. (U.S.P. 1,718,913, 25.6.29. Appl., 9.8.20).—Heavy hydrocarbon oils are mixed with steam and passed under pressure through a pipe extending backwards and forwards through a furnace and maintained at a substantially constant temperature sufficient to produce cracking. The vapours from the coils are brought directly into contact with a quantity of liquid sufficient to chill them suddenly below cracking temperature.

H. S. GARLICK.

Purifying and imparting a green fluorescence to lubricating oil. W. D. RIAL and W. R. BARRATT, Assrs. to PAN AMER. PETROLEUM Co. (U.S.P. 1,708,592, 9.4.29. Appl., 7.6.27).—Lubricating oil, e.g., "over-head stock," is treated with 1½% of coal-tar pitch and 2 oz. of hydrated lime per gal. at 120°, filtered, and clarified and decolorised at 110–160° with a decolorising clay.

R. BRIGHTMAN.

Reclaiming waste oil. R. R. ROSENBAUM (U.S.P. 1,708,488, 9.4.29. Appl., 1.6.27).—Used lubricating oil is heated under vacuum with sodium silicate and percarbonate, perborate, or persulphate, again agitated under vacuum with fuller's earth heavily impregnated with sulphur dioxide, and the gasoline and lighter fuel oils are then distilled off.

R. BRIGHTMAN.

Separating wax from mineral oils. R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 1,719,350, 2.7.29. Appl., 18.7.22).—A low-viscosity oil containing wax is treated with an oil-miscible liquid, consisting essentially of an aliphatic alcohol and an agent for promoting the miscibility of the alcohol and oil, to precipitate the wax.

H. S. GARLICK.

Pressing of wax-containing oil. F. W. SULLIVAN, W. J. MCGILL, and H. L. WALKER, Assrs. to STANDARD OIL Co. (U.S.P. 1,720,300, 9.7.29. Appl., 1.6.25).—Oil is removed from wax cake, formed in the filter press in the usual manner by forcing water, at a temperature below the m.p. of the wax, through the cake.

T. A. SMITH.

Preparing carbon dopes. H. W. A. DIXON (U.S.P. 1,718,949, 2.7.29. Appl., 23.3.27).—Colloidal mixtures containing oil and wax which are solid at ordinary temperatures are prepared in an apparatus consisting

of two containers and the necessary pumps and emulsifying mill. Oil is heated in one container and passed through the pumps and mill to the other container to heat the whole apparatus. It is then passed backwards and forwards through the mill, the necessary ingredients to form the emulsion being added. T. A. SMITH.

Recovery of entrained oils from clay. G. F. OLSEN (U.S.P. 1,720,144, 9.7.29. Appl., 22.1.27).—Lubricating oil having a viscosity greater than 150 sec. (Saybolt) at 100° F. is recovered from filter-press cake by forcing through the cake, while still in the press, a lubricating oil having a viscosity of 75–150 sec. (Saybolt). T. A. SMITH.

Lubricant. C. B. DE HART (U.S.P. 1,709,311, 16.4.29. Appl., 28.1.27).—Equal weights of jefferisite and aluminium stearate are mixed cold with lubricating oil, and the mixture is heated nearly to the flash point of the oil. R. BRIGHTMAN.

Method and apparatus for testing coal. R. GEIPERT (U.S.P. 1,721,813, 23.7.29. Appl., 7.10.26. Ger., 4.3.26).—See B.P. 267,082; B., 1927, 516.

Coking-retort oven. J. BECKER, ASSR. to KOPPERS Co. (U.S.P. 1,721,763, 23.7.29. Appl., 13.10.22).—See B.P. 216,579; B., 1924, 625.

Low-temperature distillation of carbonisable material. K. W. J. H. JACOBS (U.S.P. 1,721,997, 23.7.29. Appl., 24.8.21. Ger., 28.7.19).—See B.P. 157,794; B., 1922, 578 A.

Apparatus for determining the calorific value of gases. O. DOMMER (U.S.P. 1,720,782, 16.7.29. Appl., 25.9.24. Holl., 1.10.23).—See B.P. 222,839; B., 1925, 616.

Continuous rectification of [hydrocarbons and other] liquids of high b.p. E. A. BARBET (U.S.P. 1,722,322, 30.7.29. Appl., 11.6.23. Fr., 11.12.22).—See B.P. 208,175; B., 1925, 533.

Fuel burners. BABCOCK & WILCOX, LTD. From DEUTS. BABCOCK U. WILCOX DAMPFKESSEL-WERKE A.-G. (B.P. 315,466, 14.4.28).

Pulverised fuel burner. E. C. R. MARKS. From RILEY STOKER CORP. (B.P. 316,341, 26.4.28).

Pulverulent fuel burners. GEN. ELECTRIC CO., LTD., and G. S. WOOLLATT (B.P. 316,242, 26.4.28).

Gas burners. J. P. AUTER (B.P. 315,918, 21.4.28).

Method and apparatus for burning liquid fuel. FILMA OIL BURNERS, LTD., and A. MARSDEN (B.P. 315,252, 10.4.28).

Apparatus for separating benzol from waste water. W. LINNMANN, JUN. (B.P. 304,595, 16.1.29. Ger., 21.1.28).

Flotation agent (U.S.P. 1,709,447).—See X.

III.—ORGANIC INTERMEDIATES.

Rapid method for determination of phenols. J. A. SHAW (Ind. Eng. Chem. [Anal.], 1929, 1, 118 121).—The aqueous phenol sample (10 c.c., diluted, if necessary, to contain less than 1 g. per litre) is acidified and distilled from a pyrex test-tube by means of a

current of air saturated with steam by being first passed through hot water in a similar test-tube, both tubes being contained in a chamber through which live steam is passing. The volume of the sample thus remains approximately the same while 25 c.c. of distillate are collected. Aliquot portions of the distillate are successively diluted until the phenol content is between 30 and 35 pts. per million, and the turbidity produced by a few drops of bromine water is estimated by comparison with standards containing 30 and 35 p.p.m., respectively. Alcohols, amines, aldehydes, organic bases, oils, and inorganic salts interfere and must be removed, *e.g.*, by previous distillation from alkali or from acid and alkali in succession. The temperature of the standards and diluted distillate should be adjusted to 18–20°, and comparison be made within 15–20 sec. of precipitation. The sensitivity is about 30 p.p.m. C. HOLLINS.

Hydroxyl and carboxyl groups in humic acids. W. FUCHS and W. STENGEL (Brennstoff-Chem., 1929, 10, 303–307).—Humic acids were isolated from two brown coals (Cassel and Rhenish) by treatment with aqueous hydrochloric acid, and extraction with a mixture of benzene and alcohol until bituminous substances were removed. Further reaction with dry methyl alcohol and dry hydrochloric acid gave a product which could be extracted with ether, and was finally completely methylated by means of diazomethane; the product contained 17–19% total OMe, 7–4% ester OMe, and about 3% of carbonyl groups. Oxidation of a humic acid was effected by the use of nitric acid, and the methylation of the oxidation products was also investigated; the completely methylated products in this case contained 19% total OMe, 8.5–9.5% ester OMe, and 2.4–2.8% CO. Analyses show that the substances are hydroxy-oxy-carboxylic acids, the oxidation products of which are very similar to the so-called "nitrohumic acid" (B., 1928, 555). Differences in the elementary composition of humic acids from various sources are attributed to alterations in the degree of reduction.

R. H. GRIFFITH.

See also A., Aug., 874, **Salting-out of ethyl acetate** (SCHLESINGER and KUBASOWA). 886, **Electrolytic oxidation of organic substances** (MARIE and LEJEUNE). 923, **Catalytic reduction of nitro-compounds** (BUSCH and SCHULZ). 924, **Preparation of aminonaphthols** (BROWN and others). 930, **1:2-Phenanthraquinone and its derivatives** (FIESER). 934, **Condensation of di-*o*-thiobenzoic acid with aromatic hydroxy-compounds** (SEN and SEN-GUPTA). 948, **Micro-determination of methoxyl and ethoxyl groups** (FRIEDRICH).

Acetylene from methane. PETERS and MEYER. **Naphthenesulphonic acids.** PILAT and DAVIDSON.—See II. **Determination of formaldehyde.** HEIM.—See XX.

PATENTS.

Manufacture of polyalkylene glycol esters. O. LOEHR, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,710,424, 23.4.29. Appl., 16.12.26. Ger., 18.12.25).—Organic carboxylic acids are treated with an olefine oxide in presence of an esterification catalyst; *e.g.*, acetic acid and ethylene oxide in presence of sulphuric

acid at 125° yield glycol diacetate, b.p. 90—120°/20 mm., and acetates of polyethylene glycols. R. BRIGHTMAN.

Condensation products [thioglycollic acids] from reaction products of sulphur chloride and primary arylamines. R. HERZ, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,699,432, 15.1.29. Appl., 16.5.28. Ger., 11.4.14).—The cyclic products from arylamines and sulphur chloride are treated with water and alkali and condensed with chloroacetic acid to give thioglycollic acids. C. HOLLINS.

Production of benzidine and derivatives. R. A. NELSON, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,718,373, 25.6.29. Appl., 22.6.20).—By the addition of sulphuric acid gradually to a solution of a hydrazo compound in a low-boiling solvent of the benzene series with vigorous stirring, the benzidine compound separates as sulphate in the form of a stiff paste. H. S. GARLICK.

Manufacture of benzyl phthalate. A. ZIMMERLI, Assr. to RHODIA CHEM. CO. (U.S.P. 1,708,404, 9.4.29. Appl., 28.2.26).—A solution of phthalic anhydride in an excess of benzyl alcohol is boiled for about 8 hrs., the solution is distilled to remove water, and the residue distilled in a vacuum. Benzyl phthalate has m.p. 42—44°, b.p. 260—285°/10 mm. R. BRIGHTMAN.

Catalytic oxidation of organic compounds. A. O. JAEGER, Assr. to SELDEN CO. (U.S.P. 1,722,297, 30.7.29. Appl., 26.8.27).—See B.P. 296,071; B., 1929, 236.

Manufacture of acetic acid. H. W. MATHESON, Assr. to CANADIAN ELECTRO PRODUCTS CO., LTD. (Re-issue 17,377, 16.7.29, of U.S.P. 1,410,207, 21.3.22).—See B., 1922, 347 A.

Conversion of cyanonaphthalenesulphonic acids. R. HERZ, F. SCHULTE, and W. ZERWECK, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,721,882, 23.7.29. Appl., 24.2.27. Ger., 17.5.26).—See B.P. 296,010; B., 1928, 780.

IV.—DYESTUFFS.

See A., Aug., 930, 1:2-Phenanthraquinone and its derivatives (FIESER). 934, Colour and molecular strain (DEY and DUTT). 934, Condensation of di-*o*-thiobenzoic acid with aromatic hydroxy-compounds (SEN and SEN-GUPTA). 947, Alizarin and mercuric acetate (WHITMORE and LEUCK). 962, Pigments of red algæ (LEMBERG).

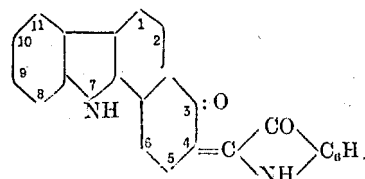
PATENTS.

Dyes [for domestic use]. E. R. SCHLATTER, Assr. to DIP-IT, INC. (U.S.P. 1,710,076, 23.4.29. Appl., 7.5.25).—The usual mixture of dyes (which may contain incompatibles) is dissolved in glycerin, *e.g.*, to 2% concentration. The solution may be diluted largely without precipitation. C. HOLLINS.

Brown sulphur dyes. O. SPENGLER and R. WEIDENHAGEN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,705,840, 19.3.29. Appl., 31.10.25. Ger., 10.1.25).—Aldehyde or acyl derivatives of leucoindophenol, particularly 4-benzylideneamino-4'-hydroxydiphenylamine, are melted with sulphur at 200° to give benzthiazole compounds which are dark brown sulphide dyes. C. HOLLINS.

Disazopyrazolone colouring matters [chrome wool dyes] which contain a 1:5-dihydroxynaphthalene nucleus. L. W. GELLER, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,706,878, 26.3.29. Appl., 23.12.25).—A *p*-phenylenediamine is coupled on the one hand with a pyrazolone and on the other with 1:5-dihydroxynaphthalene; *e.g.*, *p*-nitroaniline → 1-*p*-sulphophenyl-3-methyl-5-pyrazolone, reduced → 1:5-dihydroxynaphthalene dyes wool maroon, changing to brown on chroming. C. HOLLINS.

Vat dyes [of the indigoid series]. M. P. SCHMIDT and O. LIMPACH, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,706,902, 26.3.29. Appl., 17.11.25. Renewed 12.2.29).—An isatin α -anil is condensed with a 3-hydroxy- α -naphthacarbazole to give black dyes (annexed formula). 3-Hydroxy-8-methyl- α -naphthacarbazole is thus con-



densed with isatin α -anil or 5-chloroisatin *p*-chloroanil. Improved fastness to chlorine results when the carbazole nitrogen is alkylated. C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Regain of stretched fibres and the porous structure of wool. A. T. KING (Trans. Faraday Soc., 1929, 25, 451—458).—The visible pores, after the scales had been removed from stretched wool fibres, were observed to have become elongated when examined microscopically, and this is believed to be the case of those pores that are too small to be so observed. Dry stretching of fibres was shown with the aid of a specially designed micro-balance to be accompanied by an increase in weight, whilst fibres which had been steamed in the stretched condition and having microscopic pores which tended to seal up and disappear decreased in weight. A theory of the pore structure of wool is advanced to account for these observations, namely, that the initial absorption of water, say up to 5%, consists of an adsorption layer on the internal surfaces of the pores, the subsequent addition, 5—25%, consisting of the filling up of the pores, and the final 25—33% being an osmotic absorption. The regain is determined by the dimensions of the pores that are filled with water, so that any alteration in the size of the pores should alter the regain. H. T. S. BRITTON.

Viscose. XXIV. Increasing the tensile strength of viscose silk. G. KITA, T. NAKASHIMA, J. ONOHARA, and K. MASUI. **XXV. Stretching of finished artificial silk in concentrated sulphuric acid.** G. KITA, S. IWASAKI, and S. MASUDA (J. Cellulose Inst., Tokyo, 1929, 5, 147—153, 175—177).—XXIV. Artificial silk of greater tensile strength is obtained by increasing the friction of the guides on the partly coagulated thread or by using a higher spinning velocity while maintaining the same pressure on the spinning liquid. Breaking of the thread will occur, however, if certain limiting values

for friction and spinning velocity are exceeded, which values depend on the properties of the viscose, viz., degree of ripeness, on the composition and temperature of the coagulating bath, and on the direction of spinning etc. Increased tensile strength may also be imparted by stretching the half-coagulated thread under the same conditions through a greater distance, preferably in the coagulating bath. Conditions and results are given numerically.

XXV. Artificial silk, after being wound on to a rotating glass cylinder, was passed through a bath of concentrated sulphuric acid and received on a second cylinder the speed of which was greater than that of the first, so that the thread could be subjected to varying degrees of tension depending on the relative speeds of the two cylinders. The effect of acid concentration, length of bath, temperature, and tension on the properties of the threads was thereby examined. By this treatment it was found possible to increase the tensile strength and elasticity of artificial silk by 20–50% and to make it more resistant to water.

F. R. ENNOS.

Viscosity of viscose. II. Structure viscosity of viscose. T. NAKASHIMA (*Kolloid-Z.*, 1929, 48, 326–328; cf. Kita and co-workers, *B.*, 1929, 713).—The measurements of the viscosity of viscose carried out by Tomihisa (*J. Soc. Chem. Ind. Japan*, 1928, 31, 866) are examined and shown to be in accordance with Wo. Ostwald's equation. The equation is also applicable to the spinning experiments, and it follows that the thickness of the thread bears no direct relation to the pressure.

E. S. HEDGES.

"Sthénosage" of viscose. L. MEUNIER and R. GUYOT (*Rev. gén. Colloid.*, 1929, 7, 53–66).—The swelling of natural and artificial cellulose has been studied, and it is inferred that the hydrophilic properties of cellulose are related to the number of free hydroxyl radicals present. A study has been made of the change in the degree of swelling produced by Eschallier's process of "sthénosage" (a kind of tanning effected by the simultaneous action of acid and formaldehyde): by this process the swelling is greatly reduced and the hydroxyl radicals are replaced in pairs by the group $\cdot\text{O}\cdot\text{CH}_2\cdot\text{O}\cdot$. The best conditions for this process have been worked out, (a) by soaking the fibre in acid, drying, and then subjecting to the vapours of formaldehyde, (b) by soaking the fibre directly in a mixture of acid and formaldehyde, drying, and finally heating at a moderate temperature. In each case the effect of variation of all the factors involved has been examined. The second method is considered to be the more advantageous and also the simpler in practice. Attempts have been made to replace the formaldehyde by acetaldehyde, paraldehyde, and acetone, but the reduction in swelling produced by these reagents was considerably less.

E. S. HEDGES.

Absorption of soda by cellulose. Y. KAMI and T. YAMASHITA (*J. Cellulose Inst.*, Tokyo, 1929, 5, 178–185).—The relation between the concentration of sodium hydroxide and its absorption by cotton is expressed by a stepped curve; in the case of tissue paper and wood cellulose the curve exhibits no sharp breaks. Yields of over 90% of regenerated cellulose (viscose)

may be obtained without difficulty, provided the sodium hydroxide absorption is over 0.17 g. per g. of cellulose, which requires the use of a solution containing more than 14.7% of sodium hydroxide. Higher temperatures tend to diminish the absorption, the most favourable steeping temperature being 20°. These results correspond very closely with practical experience in the viscose factory, where the average absorption is 0.18–0.23 kg. of sodium hydroxide per kg. of wood cellulose.

F. R. ENNOS.

Turpentine obtained as by-product in the manufacture of cellulose by the sulphate method.

I. Y. POSTROVSKI and V. G. PLUSNIN (*J. Chem. Ind. Moscow*, 1928, 5, 1161–1165).—The turpentine is contaminated with methyl mercaptan and sulphide arising from interaction between by-product sodium sulphide and methyl alcohol. The contamination is partly prevented by liquefying the emitted gases; the sulphur compounds not otherwise removed from the oil are then oxidised with dilute aqueous bleaching powder.

CHEMICAL ABSTRACTS.

Weighing. PARTRIDGE.—See I. **Viscosity of nitrocellulose solutions.** MERZ. **Nitrocellulose lacquers.** WOLFF and ROSEN.—See XIII. **Sugar-factory filter cloths.** SANDERA.—See XVII. **Cellulose from corn.** LINK.—See XIX.

PATENTS.

Manufacture of moulded indurated-fibre articles.

H. H. CLARK, Assr. to CLARK FIBRE PRODUCTS CORP. (U.S.P. 1,717,092, 11.6.29. Appl., 15.1.27. Fr., 4.6.26).—Old cotton cloth is digested in an aqueous solution containing about 5% of sodium hydroxide and 3% of sodium carbonate for about 4 hrs. under a pressure of 30–50 lb./in.² and the resulting pulp is run into a perforated mould. After drying, the mass is compacted by pressure to expel any entrapped air, impregnated with the indurating fluid (zinc chloride solution), and its walls are further compacted and shaped by moulding under pressure.

F. R. ENNOS.

Manufacture of cellulose pulp. J. J. DE LA ROZA, Assr. to BAGASSE PRODUCTS CORP. (U.S.P. 1,721,376, 16.7.29. Appl., 11.6.27).—Material containing cellulose is treated first with hot aqueous solutions containing sulphur, then with hot dilute alkaline solution (at 160–173° in both cases), and is finally refined and washed.

A. G. POLLARD.

Apparatus for spinning solutions of cellulose ethers or esters. M. KLEIN, Assr. to RUTH-ALDO CO., Inc. (U.S.P. 1,716,781, 11.6.29. Appl., 27.3.28. Fr., 24.6.27).—The spinning cell is provided with an inlet for cold air in its upper part where the downwardly directed spinning nozzle is situated, and the thread issuing therefrom passes down the vertical passage between a number of diaphragms set obliquely in the cell. Hot air and solvent vapours are withdrawn from the lower part of the cell.

F. R. ENNOS.

Apparatus for dehydrating viscose. S. A. NEIDICH (U.S.P. 1,717,118, 11.6.29. Appl., 21.6.26).—Fixed inside a retort having a removable lid is an annular spool seat coaxial with a conduit, which projects upwards into the bottom of the retort. A dehydrating fluid

(alcohol) is passed up the conduit, is vaporised by a heating arrangement surrounding it, and is condensed at the bottom of the retort by a refrigerant container below.

F. R. ENNOS.

Manufacture of cellulose products. H. P. BASSETT and T. F. BANIGAN, Assrs. to MEIGS, BASSETT, & SLAUGHTER, INC. (U.S.P. 1,709,513, 16.4.29. Appl., 11.5.23).—The acetylating solution containing acetylnitrocellulose is treated, *e.g.*, with glacial or dilute acetic acid to reduce the consistency to a point just short of precipitation, and the ester is spun through a precipitating bath without removing it from the solution.

R. BRIGHTMAN.

Cellulose nitrate plastic. G. L. SCHWARTZ, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,710,078, 23.4.29. Appl., 16.6.21. Renewed 6.7.28).—*n*-Butyl phthalate, mixed with a non-solvent, *e.g.*, glycerides or esters of higher fatty acids and liquid petrolatum, is used as softener for nitrocellulose.

R. BRIGHTMAN.

Manufacture of no-glare writing paper. W. G. REYNOLDS (U.S.P. 1,721,576, 23.7.29. Appl., 12.3.27).—A calendered writing sheet consists of a filler having a coloured material worked therein containing sulphite-stock, auramine-double O, and soluble-blue.

A. G. POLLARD.

Treatment of highly-porous paper-like material. K. L. MOSES (U.S.P. 1,716,654, 11.6.29. Appl., 28.5.23. Renewed 4.8.28).—To enable the paper web to support its own weight while being impregnated with an aqueous filler, *e.g.*, rubber latex, a small quantity of a strength-giving element, *e.g.*, rubber, is incorporated with the pulp, either before or after the formation of the web. Latex is sprayed upon the latter, or the web is passed through a hydrocarbon solution of the rubber before impregnation with the filler. The minimum quantity of strength-giving element is used, so as not to diminish the porosity of the web.

F. G. CLARKE.

Apparatus for spinning solutions of cellulose esters or ethers. RUTH-ALDO Co., Inc., Assees. of (Miss) M. KLEIN (B.P. 292,608, 22.6.28. Fr., 24.6.27. Addn. to B.P. 292,561; B., 1929, 595).—See U.S.P. 1,716,781; preceding.

Manufacture of cellulose products. M. CUSIN (Assr. to SOC. LYONNAISE DE SOIE ARTIFICIELLE), and P. A. A. CHEVALET (U.S.P. 1,722,202, 23.7.29. Appl., 7.1.27. Fr., 22.2.26).—See B.P. 266,300; B., 1928, 520.

Manufacture of cellulose derivatives, and of artificial materials. L. LILIENFELD (U.S.P. 1,722,927—S, 30.7.29. Appl., 17.3.25. Austr., 4.4.24. Renewed [A] 1.6.29, [B] 29.6.28).—See B.P. 231,807—8; B., 1925, 913—4.

Pumps for viscose. W. CHATELAIN (B.P. 302,357, 10.12.28. Fr., 16.12.27).

Production of glazed and/or coloured paper. V. ANTOINE (B.P. 297,098, 11.6.28. Belg., 15.9.27).

[Machine for] treatment [printing and waxing] of paper etc. A. C. McMASTER (B.P. 315,925, 28.4.28).

Coating composition (U.S.P. 1,710,453).—See XIII. **Sulphite liquors** (U.S.P. 1,710,272).—See XVI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Application of azoic colours to wool. A. E. EVEREST and J. A. WALLWORK (J. Soc. Dyers & Col., 1929, 45, 235—237).—Naphthols and arylides of hydroxy-naphthoic acid show a substantive affinity for wool and silk when applied from a soap solution. The impregnated wool is then coupled with the base, using a bath more dilute than that usually employed in cotton dyeing. The general fastness of these insoluble azo colours on wool is very good.

L. G. LAWRIE.

X-Ray studies of dyed cellulose. Theory of dyeing. F. BRON (Helv. phys. Acta, 1928, 1, 165—185; Chem. Zentr., 1929, i, 808).—Inorganic dyes are, in general, deposited as irregularly oriented crystals in the (ramie) fibre, whilst organic dyes appear to be adsorbed in the amorphous form at the micellar surfaces. Silver and gold appear to adopt an orientation in addition to the irregular arrangement. Iodine is adsorbed. In the dyeing process the size of the crystallites of the fibre appears to be altered by aggregate-crystallisation or coagulation, the increase being effected chiefly by the hot salt solution of the dye bath, and not by the dye solution itself.

A. A. ELDRIDGE.

PATENTS.

Process of dyeing. H. E. GRIER, Assr. to R. N. COMEY BROOKLYN Co. (U.S.P. 1,720,595—6, 9.7.29. Appl., 16.11.26).—(A) Loosely-woven articles are treated with a solution of wax with which a dye is incorporated. (B) A resin is added to the solution.

L. G. LAWRIE.

Loading of silk fabrics. R. CLAVEL (B.P. 303,129, 9.3.29. Ger., 29.12.27).—The fabric is passed continuously through a series of baths, being treated successively with at least two substances which yield insoluble compounds by reaction with one another. A suitable apparatus is described.

L. G. LAWRIE.

Loading and dull-lustring of silk. R. CLAVEL (B.P. 303,128, 1.3.28. Ger., 30.12.27).—Natural or artificial silk is alternately treated with solutions of heavy-metal salts such as stannic chloride or lead acetate and with disodium phosphate or other alkaline phosphate solution. The silk may first be treated with a small quantity of an acid reagent.

L. G. LAWRIE.

Preparation of effect threads. CHEM. FABR. VORM. SANDOZ (B.P. 295,936, 18.8.28. Ger., 20.8.27).—In the process of B.P. 195,619 (B., 1924, 128) the mercerised and esterified cotton is steamed to restore its lustre.

L. G. LAWRIE.

Protecting fabric. R. A. PHAIR, Assr. to H. KOHN-STAMM & Co., Inc. (U.S.P. 1,708,519, 9.4.29. Appl., 9.3.25).—To the final rinse waters in laundering textile fabrics protective agents are added, which will remain in the fabrics either to neutralise any sulphuric acid etc. absorbed from the atmosphere or to convert the acid into a harmless substance; chalk, calcium borate or hydroxide, magnesium hydroxide, sodium acetate or phosphate may be used.

R. BRIGHTMAN.

Colouring organic fibres or articles such as skins, furs, paper, etc. consisting of animal and vegetative fibres. N. WOROSHTZOW (U.S.P. 1,719,159, 2.7.29. Appl., 30.6.25).—See G.P. 402,503; B., 1926, 96.

Apparatus for dyeing textile materials. B. JOKL (B.P. 316,154, 23.1.28).

Dyeing jigs etc. J. LITIGOW (B.P. 315,536, 5.6.28).

Treating artificial fibres [in cake form] with liquids. DU PONT RAYON Co., Assees. of E. K. GLADDING (B.P. 293,767, 11.7.28. U.S., 11.7.27).

Winding and wet-treating of textile threads, particularly artificial silk threads. M. SCHOENFELD (B.P. 304,197, 10.1.29. Switz., 16.1.28).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Comparison of modern chamber sulphuric acid plants. T. R. HARNEY (Chem. Met. Eng., 1929, 36, 402—406).—In view of the difficulty of reducing comparisons between different plants to a basis of capital and operating costs the comparison is made between radiation surfaces relative to throughput. On various standard-type chamber sets this ratio varies from 1.0 to 1.6 sq. ft./lb. of sulphur/24 hrs.; with Pratt sets it is rather less, on the basis of similar nitre consumption. The Pratt system consists of a large first chamber, a small tower from which the gases are returned to the first chamber, and 2 or 3 small auxiliary chambers. With Mills-Packard chambers the ratio varies from 1.0 to 1.4. Gaillard tower chambers with turbo-dispersers have ratio 0.67—0.80, and the Schmiedel box 0.57—0.62. A system of water-cooled flues will operate at about the same figure. These ratios require further modification with variation in cost per sq. ft. of surface, maintenance cost, etc. No quantitative data for these are given, but a classification in order of merit based on a general allowance for these variables puts Pratt and Mills-Packard chambers first. The first part of an ordinary chamber set can, however, work at a ratio of 0.7 sq. ft./lb. of sulphur/24 hrs., and a combination of rectangular chambers in the forward part of the set with Schmiedel boxes or pipe systems at the rear appears attractive. C. IRWIN.

Safety in manufacture of sulphuric acid by the contact process. S. H. KERSHAW (Ind. Eng. Chem., 1929, 21, 762—763).—The use of gas-masks is desirable when treating the contact mass for revivification. Abundant safety showers should be provided. Acid valves are best placed not more than 18 in. above ground level, so that the operator can keep his face away. The usual precautions on entering storage tanks are described.

C. IRWIN.

Manufacture of potassium chlorate by double decomposition. C. MAZZETTI (Annali Chim. Appl., 1929, 19, 273—282).—The various aqueous ternary systems involved in the manufacture of potassium chlorate from calcium chlorate and potassium chloride are investigated, and the best conditions for the reaction determined.

T. H. POPE.

Determination of calcium carbide in technical calcium cyanamide. D. STROHAL (Arh. Hemiju, 1929, 3, 118—124).—50 c.c. of 50% alcohol and 150 c.c. of 10% sodium chloride solution are added to 100 g. of calcium cyanamide. A stream of nitrogen is passed first through the flask, and then through a red-hot

combustion tube packed with copper oxide, the resulting gases being absorbed in a weighed absorption U-tube. The reaction is complete after 3 hrs. The weight of carbon dioxide produced multiplied by 0.7821 gives the percentage carbide content.

R. TRUSZKOWSKI.

Manufacture of Epsom salts. N. N. EFREMOV and M. M. NARKEVICH (J. Chem. Ind. Moscow, 1928, 5, 1179—1183).—Whilst magnesium sulphate heptahydrate is unchanged at the ordinary temperature, the other hydrates (industrially preferable but not in demand) effloresce. For the preparation of the heptahydrate, solutions are concentrated at 80—82° to d 1.30, then cooled slowly, especially below 30—35°; stirring is discontinued below 35°. At 30° the liquid is seeded and the crystals are left in contact with the mother-liquor for several days.

CHEMICAL ABSTRACTS.

Hot and cold decomposition of carnallite, and Wilson's rule. W. ALTHAMMER (Mitt. Kali-Forschungs-Anst., 1928, 21—51; Chem. Zentr., 1929, i, 1139).—A mathematical study of the decomposition of carnallite under various industrial conditions. A. A. ELDRIDGE.

Influence of temperature on the precipitation of nickel carbonate. M. SERGEYEV (Oil Fat Ind. Russia, 1928, No. 11, 15).—Nickel carbonate for preparing nickel catalyst is preferably precipitated from nickel sulphate solution (6.47 g. Ni per litre) by 10% sodium hydroxide solution in slight excess at the b.p.

CHEMICAL ABSTRACTS.

Electrolysis of molten zinc chloride. (Sir) R. THRELFALL (J.S.C.I., 1929, 48, 210—223 T).—During the years 1903 to 1916 the manufacture of very pure zinc and chlorine by fusion electrolysis was developed by the author at the works of Messrs. Albright & Wilson, Ltd., at Oldbury, England. The original intention was to make use of both the zinc ash and "pickle" liquor (impure ferrous chloride) produced by the galvanising works of the district, but it soon appeared that it was better to dissolve the ash in crude hydrochloric acid, of which a supply was available within piping distance. A detailed account is given of the manufacture of pure dry fused zinc chloride on the large scale, and of its subsequent electrolysis. The various types of electrolytic cells investigated are described and illustrated by working drawings, as is the apparatus devised for the production of pure, dry, fused zinc chloride. Incidentally, the manufacture of large tanks and slabs of hard carbon is also described with all necessary detail. Some notes on the relation of the colour of ferric oxide paint to its mode of preparation are included, as well as notes on various pieces of apparatus which it is thought may have a wider application. The original paper must be consulted for further details.

See also A., Aug., 891, **Synthesis of ammonia by the glow discharge** (BREWER and WESTHAVER). **Electrolytic preparation of ammonium permanganate** (RAPIN). 892, **Precipitation of manganese dioxide by electrolysis** (ROLLET).

Sulphur compounds from gases. LORENZEN.

Nitrogen oxides in coke-oven gas. SCHUFTAN.

Separation of coke-oven gas. BRONN.—See II. **Acid-resistant alloys.** FIELD; BEDWORTH.—See X.

Analysis of insecticides. HART.—See XVI.

PATENTS.

Production of calcium carbide and pig iron. H. ELDRIDGE, Assr. to M. E. MCGHEE, M. E. WALDRON, and M. K. ELDRIDGE (U.S.P. 1,719,970, 9.7.29. Appl., 8.3.27).—A mixture of calcium carbonate, carbon, and iron oxide is heated by an electric arc, and a current of air is injected above the mass to cause combustion of liberated carbon monoxide and to give a reduced pressure above the mixture. C. A. KING.

Recovery of iodine. J. W. TURRENTINE (U.S.P. 1,708,287, 9.2.29. Appl., 18.2.25).—Iodine vaporised with a current of steam is passed countercurrent with sodium or potassium hydroxide solution and the iodine is precipitated by acidifying. R. BRIGHTMAN.

Preparation of solutions of hydrofluosilicic acid. W. MÖLLER and W. KRETH (U.S.P. 1,720,965, 16.7.29. Appl., 22.12.26. Ger., 23.12.25).—See B.P. 263,779; B., 1928, 90.

Combustion of ammonia. H. PAULING (U.S.P. 1,722,339, 30.7.29. Appl., 29.12.27. Ger., 22.10.26).—See B.P. 279,511; B., 1928, 402.

Treating synthesis gases of synthetic-ammonia plants. W. H. KNISKERN, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,721,455, 16.7.29. Appl., 13.12.23).—See B.P. 226,224; B., 1925, 713.

Drying process for ammonium nitrate solutions. C. TONIOLO (U.S.P. 1,721,798, 23.7.29. Appl., 27.1.26. Ital., 7.2.25).—See B.P. 247,229; B., 1927, 440.

Manufacture of calcium hypochlorite. L. MELLERSH-JACKSON. From MATHIESON ALKALI WORKS (B.P. 316,447, 18.8.28).—See U.S.P. 1,713,650 and 1,713,654; B., 1929, 643.

Decomposition of chlorides and sulphates of alkaline-earth metals in molten state by steam. J. KERSTEN (U.S.P. 1,721,485, 16.7.29. Appl., 18.11.24. Ger., 15.11.23).—See B.P. 243,104; B., 1926, 88.

Solutions from leaching ores (U.S.P. 1,720,138).—See X. **Sulphite liquors** (U.S.P. 1,710,272).—See XVI.

VIII.—GLASS; CERAMICS.

Ancient glasses. IV. B. NEUMANN (Z. angew. Chem., 1929, 42, 835—838. Cf. B., 1925, 992; 1928, 670).—Descriptions are given of a number of samples of ancient glassware, with analyses of pieces of Egyptian glass from Gorub Medinea (1500 B.C.), Babylonian-Assyrian from Nippur (1400 B.C.), and Roman "millefiori" glass from Sacrau (300 A.D.). Colouring oxides are those of iron, manganese, and copper, opalescence, in the older glasses, being produced by tin oxide. A. COUSEN.

Crystal growth in opal glass. J. F. HYSLOP (J. Soc. Glass Tech., 1929, 13, 129—130).—The inference previously drawn (cf. B., 1928, 192) that for equal times of heating of opal glass below the critical point, particle size is proportional to the viscosity of the glass, is shown to be incorrect. A. COUSEN.

X-Ray study of the zonal structure of silica brick from the roof of a basic open-hearth furnace. G. L. CLARK and H. V. ANDERSON (Ind. Eng. Chem., 1929, 21, 781—785).—By noting the diffraction of X-rays by a

fine powder and recording the reflexions from the various faces of the crystals on a photographic film, a reliable method of differentiating between the various forms of crystalline silica is afforded. Unused silica brick contains about 70% of cristobalite with about 25% of tridymite and unaltered quartz. Used silica brick was found to exhibit 7 zones, samples from each of which were subjected to X-ray analysis. The quartz lines were found still to exist in the hotter zones, with about an equal intensity to those in the cooler zones, though the cristobalite lines showed increased intensity in the former. Unidentified lines are considered to be reflexions of the atomic planes of low cristobalite. Probable cristobalite interplanar distances are indicated. C. IRWIN.

Colour problems of coloured potter's clays. G. GOGUEL (Kolloid-Z., 1929, 48, 305—318).—The origin of coloured clays is discussed. An attempt has been made to investigate the production of rhythmic layers in clays by allowing solutions of ferrous sulphate and ferric chloride to diffuse into pastes of aluminium hydroxide, magnesium carbonate, and calcium carbonate; lamellæ of basic iron compounds were formed behind the diffusion front, but rhythmic layers were not observed. Iron compounds are the most effective colouring agents in clays, producing mainly greens and reds. Artificial colours were made in silica gels by precipitation with solutions containing both ferrous and ferric iron in various ratios, and these were compared with the colours of natural clays. The conclusion is reached that the colouring agent in natural clays is of colloidal nature. By means of colour filters, a method has been developed for analysing the mixture of colours present in most natural clays. Analysis of 10 different coloured clays indicates that the colouring cannot be accounted for purely by chemical composition, but that the degree of dispersion of the colloidal matter is an important factor. E. S. HEDGES.

Making the glass disc for a 70-inch telescope reflector. A. N. FINN (Ind. Eng. Chem., 1929, 21, 744—750).

See also A., Aug., 877, **Suspensions of kaolin** (DUBRISAY and others).

Weighing. PARTRIDGE.—See I. **Activity of fuel ashes towards refractories.** NEFEDIEVA and PACUKOV.—See II.

PATENTS.

Means [rotary furnace] for melting glass, silicates, and like mineral substances. G. ZOTOS (B.P. 300,576, 24.10.28. Switz., 15.11.27).

Lehrs [for bottles etc.]. THERMAL ENGINEERING CORP., Assees. of O. SHACKELFORD (B.P. 289,790, 30.4.28. U.S., 29.4.27).

Manufacture of unsplinterable glass. D. G. and C. J. CARR (B.P. 316,496, 23.11.28).

[Composite] strengthened glass and the like. J. CHAMBERLAIN and H. PERIAM (B.P. 316,755, 11.7.28).

Manufacture of compound glass sheets. D. MOSELEY & SONS, LTD., and A. NIELD (B.P. 316,565, 26.4.28).

[Apparatus for] manufacture of reinforced or

compound glass, and coating and treatment of sheets of other material. G. B. RILEY (B.P. 316,238, 24.4.28).

Making sheets of glass wool or like vitreous material. S. G. S. DICKER. From GOULD STORAGE BATTERY Co., INC. (B.P. 315,498, 26.4.28).

Production of glass in strip form and apparatus therefor. PILKINGTON BROS., LTD., and E. B. LEMARE (B.P. 316,354, 1.5.28).

Apparatus for colouring [inside of] glass [bulbs]. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of J. T. FAGAN and R. G. PHELPS (B.P. 301,841, 6.12.28. U.S., 6.12.27).

Manufacture of [moulded] pottery ware. W. J. MILLER (B.P. 308,836, 23.1.28. U.S., 24.2.27).

[Apparatus for] manufacture of [moulded] pottery. W. J. MILLER (B.P. 285,871, 23.1.28. U.S., 24.2.27).

IX.—BUILDING MATERIALS.

Unsoundness factor in Portland cement manufacture. A. J. BLANK (Rock Products, 1929, 32, 72—74).—Unsoundness of Portland cement can be attributed directly to the presence of excessive quantities of uncombined lime, which itself is due to faulty burning, poor chemical control, or insufficient grinding of raw mixtures. The composition of some materials produces inherent unsoundness in the product; *e.g.*, a clinker containing 8.32% Al_2O_3 was 50% unsound initially, whilst one with 6.17% Al_2O_3 was comparatively easy to burn. No appreciable difference in cements containing 0.3—1.2% of free lime has been noted, and from comparative tests over a range of 0.34—1.68% of free lime it is not considered that more rigid specification of this factor is necessary. C. A. KING.

Action of acids on cement and mortar. S. NAGAI (J. Ceram. Ind. Japan, 1928, 36, 382—394).—Blast-furnace cement was more resistant than a Portland cement or "solidit" to the action of sulphuric or hydrochloric acid. CHEMICAL ABSTRACTS.

Effect of type and gradation of coarse aggregate on the strength of concrete. W. F. KELLERMANN (Rock Products, 1929, 32, 86—90).—The strength of concrete is affected appreciably by the character of the coarse aggregate to a degree equal in some cases to that produced by variation of the water-cement ratio. Calcareous aggregates give consistently higher tensile and flexural strength than siliceous aggregates, and, in general, rounded fragments result in lower strengths than aggregates composed of crushed or angular fragments. Variation in grading of coarse aggregates has little effect on strength apart from workability or uniformity of the concrete. There seems to be no relation between the quality of material as measured by the abrasive test and the strength of concrete. C. A. KING.

Corrosion of concrete. C. HARI (Kogyoshirjo, 1928, 16, 1340—1352).—A discussion of the causes and prevention of corrosion. CHEMICAL ABSTRACTS.

Tests of reagents used for preventing the decay of wood. III. K. TANAKA, S. SHOJI, and N. FUNAKOSHI (Bull. Reconstr. Bur. Tokyo, 1927, ii, 2, 125—127).

—Of the wood preservatives tested for corroding action on iron, creosote was least corrosive.

CHEMICAL ABSTRACTS.

Tests of fire-resisting reagents for wood. K. TANAKA and S. SHOJI (Bull. Reconstr. Bur. Tokyo, 1927, ii, 9, 111—118).—A mixture of ammonium phosphate and magnesium borate is effective; it is not removed by exposure to water as is a mixture of ammonium phosphate and boric acid.

CHEMICAL ABSTRACTS.

See also A., Aug., 884, System $CaO-CO_2-SiO_2$ and setting of mortar (HÜTTIG and ROSENKRANZ).

Weighing. PARTRIDGE.—See I. Preservation of timber. VORONOV.—See II.

PATENTS.

Lime-hardening composition. D. M. HARRISON, Assr. to MCKENZIE MORTAR Co. (U.S.P. 1,718,954, 2.7.29. Appl., 13.4.25).—Commercial lime mortars and plasters are hardened by the addition of pulverised shale, calcium chloride, and finely-divided iron sulphide.

T. A. SMITH.

Compound asbestos-cement sheets and slabs. E. C. R. MARKS. From E. RUYTER (B.P. 316,564, 31.1.28).

[Metal-coated] bituminous roofing or damp-proof course and its preparation. W. BRIGGS & SONS, LTD., and A. S. BRIGGS (B.P. 316,241, 26.4.28).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

High-frequency fatigue [of metals]. C. F. JENKIN and G. D. LEHMANN (Proc. Roy. Soc., 1929, A, 125, 83—119).—Tests have been made on annealed copper, 0.11% carbon steel (both in the rolled and normalised conditions), 0.89% carbon steel (hardened), rolled aluminium, and normalised Armco iron to determine the effect on the fatigue limit of the frequency of alternation of stress, for frequencies varying from 600 to 20,000 periods per sec. An account is given of a number of preliminary investigations, and the apparatus finally used is described in detail. High frequencies are obtained by using fluctuations of air pressure acting directly on the test pieces, which consist of freely vibrating bars supported by means of two wires placed at each node, arranged so that they are also at the nodes for anticlastic bending. The apparatus consists essentially of the two blowers used to vibrate the specimen, each blower being a small, adjustable, resonating chamber into which air is admitted by a throttle valve at the back, while the front is closed by one face of the specimen. The position of the specimen is arranged so that as it vibrates to and fro it alternately releases the air pressure or allows it to mount up in the chamber. Measurements were made of the amplitude of vibration at the centre of the bar (to calculate the strains), the frequency, and the time of fracture. Brinell hardness tests were also carried out. Rayleigh's theory for the stresses in a long thin beam (*cf.* Jenkin, *ibid.*, 1925, A, 109, 119) is modified to allow for the anticlastic bending that takes place in short bars of moderate width, such as are used in the present investigation. Fatigue limit stress curves are

plotted both for the corrected (A) and uncorrected (B) formulae, showing that the stresses calculated from (B), which neglects anticlastic bending, increase more rapidly with the frequency than those calculated from (A). The graphs show that the fatigue limits of all the metals rise as the frequency increases, but that a frequency is reached at which this rise ceases, and in the normalised Armco iron and the hardened high-carbon steel the fatigue limit actually fell again at the highest frequencies. The maximum rise in fatigue limit is about 60% above the value at 50 periods per sec., and the greatest drop about 9% of the maximum. The Brinell hardness of the metal near the fracture also rises with the frequency, and for normalised Armco iron shows a drop at the highest frequencies.

L. L. BIRCUMSHAW.

Hydrogen and carbon monoxide contents of some metals melted in vacuo. A. VILLACHON and G. CHAUDRON (Compt. rend., 1929, 189, 324—326).—Sheets 0.1 mm. thick of aluminium, electrolytic copper, nickel, and iron, and "Armco" nitrated iron, previously distilled or melted in a vacuum of 0.02—0.002 mm., yielded appreciable quantities of hydrogen and carbon monoxide when reheated at a temperature below the m.p. The amount obtained increased with increase in temperature, and, in the case of hydrogen at least, corresponded with the solubility of the gas in the neighbourhood of the m.p. at 760 mm. pressure.

J. GRANT.

Compression and drawing tests on hot metals and alloys. A. PORTEVIN and F. LE CHATELIER (Compt. rend., 1929, 189, 248—250).—Theoretical.

Properties of materials at high temperatures. Strength at elevated temperatures of low-carbon steels for boiler construction. R. G. BATSON and H. J. TAPSELL (Dept. Sci. Ind. Res. Eng. Res., 1929, Spec. Rept. No. 14, 41 pp.).—Comparative creep tests of steels with 0.17% and 0.1% C, from boiler and super-heater tubes and from steel drums, showed curves of two general types merging into one another. There was a decrease in the rate of strain resulting from strain hardening of the material, and in the second type the absence of such decrease was indicative of lack of strain hardening. Any stress therefore (at 647°) which gave rise to initial creep would eventually result in fracture. Some evidence of intercrystalline cracking was noted which would limit the use of such steels at higher temperatures. Corrosion of the steel may exert considerable influence on the "creep" due to the reduced section, and coatings of nickel and aluminium were effective in protecting the steel from corrosion without affecting the creep properties.

C. A. KING.

Occurrence of fractures in brazing steel. W. RIEDE (Stahl u. Eisen, 1929, 49, 1161—1162).—In joining two steel tubes together fractures sometimes occur around the joint after brazing, especially if the tubes are hammer-welded together previous to dipping the joint in the molten brass and if the temperature of the latter is allowed to exceed 1100°. These fractures appear to be due to local recrystallisation during hammer-welding setting up, in the parts adjacent to the recrystallised zones, internal stress which exceeds the

tensile strength at 1100°. The intercrystalline penetration of brass in steel may be detected by etching for 3 min. in ammonia, which colours the brass black; subsequent etching in a 2% alcoholic solution of nitric acid reveals the grain boundaries without affecting the black lines due to brass.

A. R. POWELL.

Determination of iron oxides in acid steel-furnace slags. G. T. DOUGHERTY (Ind. Eng. Chem. [Anal.], 1929, 1, 163).—For the determination of ferrous oxide 0.5 g. of the finely-powdered slag, freed from metallic iron by means of a magnet, is dissolved by heating with 10 c.c. of 1:1 sulphuric acid and 5 c.c. of 48% hydrofluoric acid in a lead test-tube through which a rapid current of carbon dioxide is passed. After cooling, the contents of the tube are poured into 250 c.c. of cold water, to which have been added 25 c.c. of 5% boric acid solution, and the solution is titrated with permanganate. For the determination of total iron, the sample is decomposed as before, and the solution poured into 50 c.c. of water and 15 c.c. of 1:1 sulphuric acid; 3 g. of 20-mesh zinc are added and, when all is dissolved, the solution is filtered and reduced again for a few min. with a zinc rod. The rod is removed and rinsed, and the iron titrated with permanganate after addition of 25 c.c. of 5% boric acid solution.

A. R. POWELL.

Acid testing in [iron]-pickling baths. F. EISENKOLB (Stahl u. Eisen, 1929, 49, 1162—1164).—The advantages and disadvantages of hydrochloric and sulphuric acid pickling baths for cleaning iron and steel are enumerated, and a simple, portable apparatus is described and illustrated for determining the free acid content of used baths by measuring the volume of carbon dioxide evolved when a known volume of the acid liquor is shaken with an excess of powdered limestone.

A. R. POWELL.

Influence of oxygen on corrosion fatigue [of steel]. A. M. BINNIE (Engineering, 1929, 128, 190—191).—The fatigue limit of a 0.9% carbon acid steel was reduced from a normal value of 17 tons/in.² to 7.5 tons by dropping a concentrated solution of sodium chloride on the point of maximum stress. No rust formed on the turned surface, but the areas in which cracks developed turned black. When tested in hydrogen an intermediate value (9.1 tons) was obtained. Similar tests on 0.33% carbon steel showed 18.3 tons/in.² in air, 9.2 tons under a salt drip, and 11.8 tons under a salt drip in an atmosphere of hydrogen.

C. A. KING.

Resistance of iron-nickel-chromium alloys to corrosion by acids. N. B. PILLING and D. E. ACKERMAN (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 174, 3—33).—The metals and alloys contained Ni 0—100, Cr 0—30, Fe 0—100%. In non-oxidising acids the rate of dissolution of alloys containing less than 13% Ni is increased by chromium and decreased by nickel; alloys containing more than 13% Ni corrode at constant rates independently of the chromium or iron content. The rates, but not the critical nickel content, depend on the p_H and the acid employed. The critical nickel content is independent of microstructure or lattice structure, but heat treatment affects the location of the limit of attack. In nitric acid the points at which passivity is attained depend on the chromium content

only, this being a function of the concentration of acid. The solubility in sulphurous acid is influenced by the presence of dissolved salts. The results are incompatible with Tammann's theory relating corrosion resistance to lattice structure. **CHEMICAL ABSTRACTS.**

Resistance of copper-silicon-manganese alloys to corrosion by acids. H. A. BEDWORTH (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 189, 14 pp.).—Addition of silicon (up to 3%) and manganese (up to 1%) increases the resistance of copper to corrosion by dilute hydrochloric or sulphuric acid. Hard-drawn wires containing more than 2% Si are more resistant than annealed wires, which, however, approach equality at 0.5% Si. **CHEMICAL ABSTRACTS.**

Acid-resistant alloys. B. E. FIELD (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 191, 12 pp.).—Nickel-molybdenum alloys containing more than 15% Mo are resistant to 10% hydrochloric acid at 70°. In nickel alloys containing 20% Mo and 0–40% Fe, the corrodibility is maximal at 10% Fe, and is high above 25% Fe. Carbon decreases resistance towards acid. An alloy containing nickel, molybdenum, and iron is resistant to all cold mineral acids, except nitric acid; it is attacked by chlorine solution and other oxidising agents. Nickel-silicon (more than 10%) and -aluminium (more than 20%) alloys are resistant to acids, but brittle; alloys containing 10% Si and 5% Al are strong and resistant to acids. Copper (2.5–3%) gave finer-grained and stronger alloys. **CHEMICAL ABSTRACTS.**

Corrosion of metals as affected by stress, time, and number of cycles. D. J. McADAM, JUN. (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 175, 57 pp.).—The relationship between the variables is shown graphically. Local strengthening of the metal at the bottoms of corrosion pits is indicated. **CHEMICAL ABSTRACTS.**

Heat treatment of high-tensile aluminium alloys. A. R. PAGE (Chem. News, 1929, 139, 102–104).—The properties of forgeable aluminium alloys containing comparatively small quantities of copper, silicon, manganese, iron, etc. are improved greatly by suitable heat treatment. Quenching from a forging temperature does not appreciably influence the mechanical properties, which are only improved by subsequent tempering. Alloys of a certain type, of which duralumin was the first, harden spontaneously after quenching and attain a maximum hardness in about 7 days, due probably to the gradual precipitation of magnesium silicide, but other types with copper as an essential constituent require a temperature of about 150° to cause the copper-aluminium compound to precipitate. The temperature ranges are very narrow, the quenching temperature being 520–530° and the tempering range 149–156°. Automatically controlled electric furnaces are necessary for such treatment. **C. A. KING.**

Duralumin. E. SCHMID and G. WASSERMANN (Metall.-Wirts., 1928, 7, 1329–1335; Chem. Zentr., 1929, i, 796).—X-Ray examination shows that artificial ageing of duralumin is accompanied by separation of crystals of the compound CuAl_2 . **A. A. ELDRIDGE.**

Corrosion phenomena on aluminium sheet. R. WESENBERG (Chem. Fabr., 1929, 208).—Pinhole

perforations in corroded aluminium sheet containing 99.4% Al and 0.28% Si are attributed to the presence of aluminium suboxide, which appears blue on etching with alcoholic nitric acid. **A. R. POWELL.**

Corrosion of light and ultra-light alloys. "Cementation" [plating] of light and ultra-light alloys as a protection against sea-water corrosion. J. COURNOT and E. PEROT (Rev. Mét., 1929, 26, 367–374; cf. B., 1929, 721–2).—When copper is electro-deposited from an acid bath on aluminium, the current density, within wide limits, has no influence on the depth of penetration; deposition in a salt bath (a mixture of equal parts of sodium nitrite and potassium nitrate) causes deeper penetration than cementation in air, but the presence of salt in the superficial layers is very injurious to the adherence of the copper. "Cementation" with copper at a high temperature (900°) causes the interior of the metal to become brittle and easily powdered. Ferro-chrome alloys are ineffective for the cementation of aluminium, a notable absorption of iron taking place. With duralumin a eutectic constituent is precipitated in the interstices of the crystals which form the solid solution throughout the whole mass of the test-piece; at the same time crystals in the neighbourhood of the surface become disintegrated, and the metal becomes softer. The surfaces of test-pieces coated with a primary deposit of copper, followed by a secondary deposit of nickel, chromium, cobalt, or cadmium, and then heated in air in an electric furnace are generally much deformed, the more so at higher cementation temperatures; disintegration of the crystals takes place near the surface. Only a very imperfect deposit of copper could be obtained on a super-light alloy consisting of 94% Mg and 6% Al, the metal being insufficiently protected to resist oxidation at a low temperature. **M. E. NOTTAGE.**

Protection of aluminium and light alloys against corrosion by sea-water by electrolytic deposits of cadmium. J. COURNOT (Rev. Mét., 1929, 26, 374–376).—A deposit of pure cadmium is generally superior to binary cadmium alloys. On both aluminium and duralumin excellent films of cadmium may be obtained from a bath containing (per litre) cadmium carbonate 11.6, potassium cyanide 35, sodium phosphate 5, potassium ferrocyanide 7.5, and gelatin 2.5 g.; these films only crack with the metal support and give very satisfactory corrosion tests. Similar satisfactory results are obtained if a coating of chromium be superposed on a light cadmium coating. **M. E. NOTTAGE.**

p_H Values in the study of sea-water corrosion of light alloys. A. PETIT (Rev. Mét., 1929, 26, 285–286).—The change in the p_H value of sea-water after aluminium or one of its alloys has been immersed in it for periods of 15, 30, 45, and 75 days has been measured, but the results give no indication of the relative corrodibility of the alloys, the position of the various alloys in the series changing with the time of immersion. **A. R. POWELL.**

Micrographic study of the corrosion of some light alloys in sea-water. R. CAZAUD (Rev. Mét., 1929, 26, 274–281).—Micrographic examination of aluminium and its alloys after 6–24 hrs. immersion in

sea-water affords an idea of the relative rates of corrosion. Manganese and silicon appear to retard the corrosion, whereas magnesium accelerates it. Annealed alloys corrode more readily than alloys that have been age-hardened.

A. R. POWELL.

Rate of corrosion of magnesium and ultra-light alloys. A. PORTEVIN and E. PRETET (Rev. Mét., 1929, 26, 259—272; cf. Waché and Chaudron, B., 1929, 477).—The rate of dissolution of magnesium and its light alloys in 0.1N-hydrochloric acid and in 0.1N-citric acid solutions has been determined by measuring the rate of evolution of hydrogen. In the latter solution there is a distinct period of induction which is almost completely absent in hydrochloric acid solutions. The alloys tested may be classified as follows as regards rate of dissolution in hydrochloric acid: (a) magnesium and 4% aluminium-magnesium alloys, (b) magnesium containing up to 4% Zn, Cu, Pb, or Cd, and (c) alloys with 13% Cu or 5% Ni; in citric acid the classification is (a) magnesium and the 5% nickel alloy, (b) alloys containing 4% Al, Pb, or Cu, and (c) alloys with 13% Cu or with 4% Zn or Cd, where (a) indicates slowly soluble, (b) more soluble, and (c) rapidly soluble. Similar results are obtained by the thermal method of Mylius.

A. R. POWELL.

Influence of occluded gas on the corrosion of copper-aluminium alloys by hydrochloric acid. R. CAZAUD and A. PETIT (Rev. Mét., 1929, 26, 281—285).—Removal of the occluded gas from copper-aluminium alloys by melting in a vacuum decreases appreciably the rate of corrosion by dilute hydrochloric acid. The gas evolved from an alloy with 4.5% Cu and 1% Zn cast in sand and remelted at 800° consisted of 15% H, 6.5% O, 27% N, 16.1% CO, 22% CO₂, 2% CH₄, 2.6% C₂H₄, and 1% C₂H₂.

A. R. POWELL.

Colloid theory of rusting. G. SCHIKORV (Korrosion u. Metallschutz, 1928, 4, 242—245; Chem. Zentr., 1929, i, 800).—The theory is criticised.

A. A. ELDRIDGE.

Value of X-ray examination in discovering fissures or inclusions in light alloys. J. COURNOT and A. ROUX (Rev. Mét., 1929, 26, 272—274).—Tests on various light alloys show that X-ray examination is of value only for the detection of included particles of heavy metals and not for the detection of small fissures or intercrystalline cracks.

A. R. POWELL.

Zinc from sulphide ores. V. P. ILINSKI and A. F. SAGAIACHNUI (J. Appl. Chem. Russia, 1928, 1, 109—112).—After calcination of zinc sulphide ores containing iron at 650—750°, 95% of the zinc is dissolved by treatment with concentrated sulphuric or hydrochloric acid, about 40% of sulphur being separated. 80% of zinc is obtained from calcined ores after treatment with alkali hydroxide, the solution being free from copper and iron.

CHEMICAL ABSTRACTS.

Nickel as a constituent of bearing metals. C. L. ACKERMANN (Metall-Wirtsch., 1928, 7, 752—753; Chem. Zentr., 1929, i, 1266).—In high-lead bearing metals addition of nickel markedly raises the resistance to wear in semi-fluid friction, and in high-zinc white metals it causes increased hardness and resistance to compression and corrosion.

A. A. ELDRIDGE.

Nickel [plating] solutions. G. A. HOGABOOM (Metal. Ind., N.Y., 1929, 27, 172—175).—An addendum giving a high-anode and low-cathode efficiency is of little value. Chlorides are beneficial. The behaviour of the electrodes on addition to the solution of sodium fluoride, sodium sulphate, or boric acid is described.

CHEMICAL ABSTRACTS.

Cadmium plating. C. M. HOFF (Metal. Ind., N.Y., 1928, 26, 475—476). O. H. LOVEN (*Ibid.*, 1928, 26, 476—477).—Polemical.

A. A. ELDRIDGE.

Iron in antiquity. T. A. RICKARD (Iron & Steel Inst., Sept., 1929. Advance copy. 20 pp.)

See also A., Aug., 874, **Distribution of austenite in quenched carbon steels** (HONDA and OSAWA). 883, **Aluminium-copper-nickel system** (NISHIMURA). 884, **Iron-molybdenum system** (TAKEI and MURAKAMI). **Aluminium-antimony-silicon and aluminium-antimony-copper systems** (MATSUKAWA). 896, **Purification of mercury** (ROLLER).

Waste-heat recovery. GREGSON.—See II. **Silica brick from basic open-hearth furnaces.** CLARK and ANDERSON.—See VIII. **Corrosiveness of soils.** HOLLER.—See XVI. **Metals in dairy equipment.** HUNZIKER and others.—See XIX.

PATENTS.

Smelting furnaces. P. MARX (B.P. 300,559, 14.11.28, and 301,739, 3.12.28. Ger., [A] 14.11. and [B] 3.12.27).—(A) In a cupola furnace the lower part is surrounded with a refining chamber communicating at its lower end with the hearth of the cupola and at its upper end with the space just above the molten metal in the well of the cupola. Air and fuel gas are supplied to the refining chamber from a series of burners radially disposed below a refractory hood in the centre of the lower part of the cupola shaft in such a position that the flames pass through the upper openings into the refining chamber, whilst the hood supports the mass of unmelted material in the shaft. (B) In a modification of the above the heat to the refining chamber is supplied by oil burners arranged outside the furnace so that the hot gases pass from the chamber into the shaft of the cupola, and the hood inside the latter is provided with a capping piece having radial roof-shaped arms adapted to discharge the molten material in thin streams through the hot gases.

A. R. POWELL.

Method of making ingot moulds. E. GATHMANN (U.S.P. 1,719,544, 2.7.29. Appl., 15.8.26).—The upper end of the core of a mould is treated with a metal which will alloy with iron and is resistant to the cutting action of liquid steel.

C. A. KING.

High-temperature casting compound. C. A. OVERMIRE, ASSR. to WESTERN GOLD & PLATINUM WORKS (U.S.P. 1,719,276, 2.7.29. Appl., 23.5.28).—A mixture suitable for the casting of metals and alloys of high m.p. is composed of calcium sulphate with its own weight or more of magnesium oxide.

C. A. KING.

Forming briquettes from iron ore concentrates. P. GREDT (B.P. 285,040, 9.2.28. Ger., 9.2.27).—A portion of the concentrate is reduced to the metallic form, this is mixed with about 4 times its weight of

raw ore and with sufficient small coke or coal for reduction, and the mixture is moistened with dilute sulphuric or hydrochloric acid or with a dilute solution of magnesium chloride and formed into briquettes which harden within 48 hrs. owing to the oxidation of the reduced ore.
A. R. POWELL.

Iron alloy for milling tools. L. KLÜGER, and ÖESTERR. SCHMIDTSTAHLWERKE A.-G. (B.P. 315,502, 27.4.28).—An alloy for milling tools contains about 2% C, 10.7–25% (16%) Cr, 1.3–3% (2%) Co, 2–4.7% (3%) W, and 1–2.3% (1.5%) Mo, with or without up to 1% V as a deoxidiser, the balance being iron.
C. A. KING.

Heat-resisting [iron] alloys. L. KLÜGER, and ÖESTERR. SCHMIDTSTAHLWERKE A.-G. (B.P. 315,503, 27.4.28).—An iron alloy contains 1–1.9% C, 40–50% Ni, and 27–33% Cr.
C. A. KING.

Rendering iron-chromium-aluminium alloys ductile. V. B. BROWNE (B.P. 302,640, 10.4.28. U.S., 19.12.27).—Alloys containing 5–30% Cr, 3–10% Al, and the remainder iron are rendered ductile by a series of hot-rollings commencing at 1100° and finishing at 650°, followed or not by cold-rolling and annealing below 880°.
A. R. POWELL.

Deoxidation of oxidised metal surfaces and protection of metal surfaces against oxidation. "KOLLOIDCHEMIE" STUDIENGES. M.B.H., J. B. CARPZOW, M. MARCH, R. LENZMANN, and H. SANDERS (B.P. 315,531, 30.5.28).—Metals are treated with a mud containing unsaturated silicon compounds or allied colloids (e.g., a fresh-water mud), and a binding agent. Before use the mud is preferably allowed to develop anaerobic bacteria culture.
C. A. KING.

Proofing of iron and steel against rust. W. H. COLE (U.S.P. 1,719,464, 2.7.29. Appl., 11.3.29. U.K., 29.1.29).—Solutions of the phosphates of iron, zinc, aluminium, and chromium are prepared each having d 1.33 and mixed in the proportion of 5:5:1:1 $\frac{1}{2}$, to form a proofing solution.
C. A. KING.

Production of resistant silver surfaces. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,460, 13.4.28).—To increase their resistance to chemical reagents, articles of silver are treated, at above 500°, with a metal less electropositive than silver, other than zinc, preferably in the form of powder. Suitable metals are copper, tin, cadmium, antimony, aluminium, thorium, magnesium, and in particular manganese and manganese-aluminium alloys, the process being preferably conducted in an inert atmosphere.
C. A. KING.

Apparatus for generating mercury vapour or other vapour having a low heat conductivity. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. J. NERAD (B.P. 298,905, 15.10.28. U.S., 14.10.27).—A vaporising apparatus consists of a battery of parallel tubes contained in an outer casing. Mercury is circulated through the inner tubes, which may be connected to a drum at either end to aid the circulation, and a heating fluid is circulated around the tubes. The whole may be vertical or inclined.
C. A. KING.

Soldering composition. K. GEISEL, Assr. to ALUMINUM SOLDER CORP. (U.S.P. 1,721,814, 23.7.29. Appl., 21.9.28).—A mixture with a soldering temperature about 260° consists of zinc 200, aluminium 13, and tin 87 pts. by wt.

Welding rod. J. B. GREEN (U.S.P. 1,720,039, 9.7.29. Appl., 10.12.25).—An iron rod or wire in which the sulphur content is more than 0.025% in the centre and less than 0.025% in the surface portion is claimed.
C. A. KING.

Metal packing. G. VERDICKT (U.S.P. 1,720,065, 9.7.29. Appl., 22.10.27).—An alloy for joint packings is composed of lead (5 pts.), antimony (1 pt.), and zinc ($\frac{1}{4}$ pt.).
T. A. SMITH.

Working-up solutions obtained by leaching ores which have been subjected to chlorination roasting. H. NEWMARK (U.S.P. 1,720,138, 9.7.29. Appl., 9.11.27).—Copper is removed from the solution by treatment with metallic iron, and sulphate is removed as gypsum by treatment with calcium chloride solution. Zinc is precipitated from the resulting solution of chlorides by means of gas containing hydrogen sulphide.
T. A. SMITH.

Flotation agent. B. R. TUNISON (U.S.P. 1,709,447, 16.4.29. Appl., 9.2.24).—Petroleum naphtha containing sulphur compounds is washed with dilute sodium hydroxide, and the aqueous solution neutralised to liberate the oily flotation agent.
R. BRIGHTMAN.

Apparatus for the recovery of metals by electrolysis. F. COLLINGRIDGE (B.P. 315,481, 18.4.28).—Scrap metal coated with zinc, tin, or lead is electrolysed in perforated containers which are supported upon an insulated grid which slides in a horizontal direction to and fro in the tank so as to keep the electrolyte continuously circulating through the scrap.
A. R. POWELL.

Anode for chromium plating. F. M. BECKET (U.S.P. 1,720,312–3, 9.7.29. Appl., 14.10.26).—(A) A cast chromium anode containing 0.5–7% C, or (B) a substantially carbon-free chromium anode containing 0.25–35% Si with an aqueous solution of chromic acid as electrolyte is claimed.
C. A. KING.

Electrodeposition of chromium. K. W. SCHWARTZ, Assr. to CHROMIUM CORP. OF AMERICA (U.S.P. 1,720,354, 9.7.29. Appl., 30.4.27).—For depositing chromium on hollow cathode articles an elongated anode having angularly placed parts is used, so that gaseous products released opposite one portion of the anode will not interfere with the deposition in the active area of adjacent portions.
C. A. KING.

Manufacture of commercially pure iron alloys. L. F. REINARTZ and J. H. NEAD (B.P. 315,444, 14.2.28).—See U.S.P. 1,665,146; B., 1923, 372.

Vanadium alloys. VANADIUM CORP. OF AMERICA, Assees. of B. D. SAKLATWALLA (B.P. 305,201, 20.12.28. U.S., 2.2.28).—See U.S.P. 1,715,867; B., 1929, 604.

Hard-metal composition. K. SCHRÖTER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,721,416, 16.7.29. Appl., 26.4.26. Ger., 7.5.25).—See B.P. 251,929; B., 1926, 635.

Tuyères for blast furnaces or the like. S. P. BOWEN (B.P. 316,244, 26.4.28).

[Burner for] heat treatment of metallic wire or strip. J. P. HALEY and J. H. PINDER (B.P. 316,720, 24.5.28).

[Apparatus for] electroplating [with chromium]. J. Q. MACDONALD and W. McL. SCOTT (B.P. 299,298, 20.3.28. U.S., 22.10.27).

Calcium carbide and pig iron (U.S.P. 1,719,970).—See VII.

XI.—ELECTROTECHNICS.

Production of emission from oxide-coated filaments: a process phenomenon. V. C. MACNABB (J. Opt. Soc. Amer., 1929, 19, 33—41).—The production of oxide filaments of barium and strontium in an emitting condition was investigated to determine the advantages of several commercial methods of producing this type of cathode, but no fundamental difference was found, the basis of each method being that the filament to become emissive must undergo a gaseous bombardment, probably causing a breaking down of the higher oxides or compounds such as the carbonate to the lower oxides or pure metal, caused or accompanied by a gaseous discharge to produce an active filament. The gas is most suitably obtained from the filament, and produced from a carbonate not reduced until put *in vacuo*, and is probably carbon dioxide. In every case the results favour filaments containing most uncombined coating.

N. M. BLIGH.

Inductor coils for the high-frequency furnace. C. N. SCHUETTE (Ind. Eng. Chem. [Anal.], 1929, 1, 141—144).

See also A., Aug., 886, Electrolytic oxidation of organic substances (MARIE and LEJEUNE). 891, Synthesis of ammonia in the glow discharge (BREWER and WESTHAVER). Electrolytic preparation of ammonium permanganate (RAPIN). 892, Precipitation of manganese dioxide by electrolysis (ROLLET). 896, Purification of mercury (ROLLER).

Electrostatic determination of fusain. DAVIS and YOUNKINS. Air-gas ratio apparatus. ROSECRANS. Production of hydrocarbons from methane. STANLEY and NASH.—See II. Electrolysis of molten zinc chloride. THRELFALL.—See VII. Protection of light alloys. COURNOT and PEROT; COURNOT. Cadmium plating. HOFF; LOVEN. Nickel-plating solutions. HOGABOOM.—See X.

PATENTS.

Electric furnaces. WILD-BARFIELD ELECTRIC FURNACES, LTD., and L. W. WILD (B.P. 315,246, 10.4.28).—Instead of regulating the rate of heating by means of a variable resistance, a fixed ballast resistance, equal to 50—100% of the furnace resistance, is placed in series with the bars. For furnaces using alternating current an impedance (a non-variable choke coil) is substituted. The carborundum bars are made with enlarged ends around which the contact wire is wound.

H. T. S. BRITTON.

Electric furnace. C. L. IPSEN and A. N. OTIS, ASSRS. to GEN. ELECTRIC Co. (U.S.P. 1,719,888, 9.7.29. Appl., 10.10.25).—Across the top of an electric furnace horizontal bars of refractory insulating material carry flat sinuous resistors; the bars are so shaped that the

resistors are in contact with them to a relatively small extent.

H. T. S. BRITTON.

Electric arc furnace. O. L. MILLS, ASSR. to MILLS ALLOYS, INC. (U.S.P. 1,719,558, 2.7.29. Appl., 5.3.29).—Two electrodes, one of which is of carbon of large cross-section, move relatively to one another. At the lower end of the carbon electrode is a projection of smaller cross-section, constructed so as to provide an annular surface from which heat rays are reflected and are thereby confined to the space between the electrodes.

H. T. S. BRITTON.

Semi-solid electric conductor. H. N. MILLER, ASSR. to FANSTEEL PRODUCTS Co., INC. (U.S.P. 1,720,371, 9.7.29. Appl., 29.8.27).—The conductor is composed of gelatin and an electrolyte, and hardened by formaldehyde; when employed in electrical apparatus, it is surrounded by a hardened conducting gel that is not liquefied by heat.

H. T. S. BRITTON.

Electrodes for contact rectifiers. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 304,748, 15.8.28. Ger., 27.1.28).—Rectifiers, of lower resistance and of increased efficiency, are prepared by compressing superposed layers of powdered metal and powdered sulphide, *e.g.*, copper and copper sulphide, in thicknesses in the respective ratio of 2:1 to the extent of several thousand kg. per sq. cm., and then applying the counter electrode, of either aluminium or magnesium, by means of a pressure substantially less than one tenth of the previous pressure. The area of the rectifier should be less than 1 sq. cm. Perfectly dry copper sulphide is obtained by mixing the precipitated sulphide with powdered sulphur, and subliming the free sulphur at 250°.

H. T. S. BRITTON.

Storage battery. H. B. TEFFT, ASSR. to H. C. McNAMER (U.S.P. 1,719,204, 2.7.29. Appl., 23.12.22).—Water, for use after mixing with sulphuric acid as electrolyte and in the preparation of pastes of red lead and litharge, is prepared by electrolysis at a high voltage a suspension of a metallic ore in undistilled water.

H. T. S. BRITTON.

[Electric] battery plate. W. H. GRINDITCH, ASSR. to PHILADELPHIA STORAGE BATTERY Co. (U.S.P. 1,718,139, 18.6.29. Appl., 1.12.21).—Recently prepared negative battery plates are treated with a sugar, preferably sucrose, before being rapidly dried, in order to prevent oxidation of the active metal. The plates are immersed in an aqueous bath of the sugar, or the latter is sprinkled upon them.

F. G. CLARKE.

Electric discharge tubes. INTERNAT. GEN. ELECTRIC Co., INC., ASSCES. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 287,138, 12.3.28. Ger., 16.3.27).—One or more of the following metals: chromium, manganese, titanium, zirconium, hafnium, thorium, silicon, aluminium, and rare-earth metals (the last may be used in the form of misch-metal) may be advantageously included to the extent of 0.5—1.0% in alloys of tungsten or molybdenum to be used as refractory alloys in the internal metal parts, including anodes but not cathodes, of discharge tubes. Without such additions, the two metals tend to retain occluded gas and to liberate it while in use. The essential property of the added metals is that they must be at least

as easily vaporisable as is molybdenum. They may be incorporated either by melting together, or by mixing the ingredients in powder form, pressing, and sintering.

H. T. S. BRITTON.

Photo-electric cell. S. WIEN, Assr. to RADIO PATENTS CORP. (U.S.P. 1,720,654, 9.7.29. Appl., 13.5.25).—The metallic hydride, which constitutes the light-sensitive coating of an electrode, is formed by subjecting the electrode and a metallic salt mixed with an organic reducing agent to an electric brush discharge in the presence of hydrogen.

H. T. S. BRITTON.

Diaphragms for electrolytic cells. K. WOLINSKI (B.P. 315,551, 20.6.28).—Stronger and more durable diaphragms, particularly for use in the electrolysis of water, may be prepared from a fabric of asbestos interwoven with metal threads, preferably of non-corrosive metal. Composite threads of asbestos and metal may also be used. The increased strength afforded by the metal permits the use of lower-grade asbestos.

H. T. S. BRITTON.

Gas accumulator. R. OPPENHEIM, Assr. to SOC. LE CARBONE (U.S.P. 1,717,244, 11.6.29. Appl., 6.6.25).—One or both of the electrodes, which are unattacked by the electrolyte, are surrounded by a porous vessel containing a mixture of a porous, finely-divided conductor, *e.g.*, charcoal, a concentrated solution of the electrolyte, *e.g.*, ammonium or zinc chloride, and colloidal starch or similar colloid which will immobilise the electrolyte and render the finely-divided conductor impermeable to liquids while preserving its porosity to gases.

F. G. CLARKE.

Vibration- and sag-resistant filament. J. H. RAMAGE, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,720,000, 9.7.29. Appl., 28.7.26).—A non-sagging filament is made from a solid-solution alloy of tungsten and tantalum that has been heated so as to oxidise the latter. The rigidity is caused by the large interlocking action of the metal crystals between which tantalum oxide is evenly distributed.

H. T. S. BRITTON.

Magnetisable element. C. L. SEYMOUR (U.S.P. 1,719,564, 2.7.29. Appl., 4.4.27).—In order to reduce the effect of heat on the soft iron element of an electromagnet, it is heated to red heat and cooled in contact with turpentine.

H. T. S. BRITTON.

High-frequency induction furnace. W. ESMARCH, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,721,073, 16.7.29. Appl., 12.10.27. Ger., 10.6.27).—See B.P. 291,774; B., 1929, 101.

Manufacture of oxide cathodes. K. M. VAN GESSEL, Assr. to RADIO CORP. OF AMERICA (U.S.P. 1,721,169, 16.7.29. Appl., 6.4.27. Holl., 6.4.26).—See B.P. 274,233; B., 1927, 705.

Manufacture of oxide cathodes. G. L. HERTZ, Assr. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,720,675, 16.7.29. Appl., 25.7.24. Holl., 18.2.24).—See B.P. 229,622; B., 1925, 663.

[Movable] tapping device for electrical furnaces. SIEMENS & HALSKE A.-G. (B.P. 302,305, 11.8.28. Ger., 14.12.27).

Electric accumulators. CHLORIDE ELECTRICAL STORAGE CO., LTD., and B. HEAP (B.P. 315,556, 22.6.28).

Electric accumulators. H. DEAN, and CHLORIDE ELECTRICAL STORAGE CO., LTD. (B.P. 315,571, 17.7.28).

Positive electrode for electrical accumulators, particularly for use in miners' lamps. O. SCHNEIDER (B.P. 313,553, 25.7.28. Ger., 14.6.28).

Selenium and like cells. H. J. KÜCHENMEISTER (B.P. 294,108, 12.7.28. Ger., 16.7.27).

Photo-electric cell systems. GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 315,598, 16.8.28).

Gaseous glow lamps. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of A. W. HULL (B.P. 298,906, 15.10.28. U.S., 15.10.27).

[Corrugated] anodes for electrolytic baths [for cleaning silver ware]. G. MONAGHAN (B.P. 316,446, 9.8.28).

Recovery of metals by electrolysis (B.P. 315,481). **Electrodeposition of chromium.** (U.S.P. 1,720,354). **Anodes for chromium plating** (U.S.P. 1,720,312—3).—See X.

XII.—FATS; OILS; WAXES.

Effect of various driers on linseed oil films during ageing. P. E. MARLING (Ind. Eng. Chem., 1929, 21, 594—596).—Films of linseed oil containing two concentrations of each of lead, manganese, or cobalt acetate, and subjected to indoor ageing, showed increased acid value and decreased iodine value. The films containing manganese and cobalt were more plastic at the end of the ageing period than were those containing lead. The aged films containing the lower percentages of drier were less soluble in a toluene-alcohol mixture than were those containing the higher percentage. Linseed oil films with and without the addition of cobalt drier were exposed to the mercury-vapour lamp, and the relative effect of the drier showed concordance with the normally aged films.

S. S. WOOLF.

Mechanism of the wrinkling of wood oil. A. V. BLOM (Chem. Umschau, 1929, 36, 229—235).—The theory developed by Eibner and Rossmann (*cf.* B., 1928, 934) of preliminary cracking of the drying tung oil film followed by wrinkling due to expansion is adversely criticised. From experiment and theoretical reasoning it appears that the surface (haptogen) membrane is under pressure rather than tension, and that contraction is very improbable; moreover, a decreasing volume of the liquid oil could only cause a wrinkling of the relatively inelastic skin. The drying of thin droplets of wood oil was studied under the microscope; fan-like groups of wrinkles are formed at the edges of the drop, which radiate until they meet and interfere, causing ridges; these interference zones spread backwards in straight lines, giving the appearance termed "preliminary cracking" by Eibner and Rossmann. By gently pulling with a dissecting needle, the skin can be pulled out leaving no trace of these cell-like markings, which therefore can only be (introverted) folds and not cracks. A similar appearance can be produced artificially on the smooth-skinned film obtained by allowing wood oil to dry in darkness. The formation of crystals was found to depend entirely on the conditions of exposure, and was greatest in direct sunlight, and only very slight or

absent after 8 days' drying in darkness; no crystals were observed after 12 hrs.' irradiation by ultra-violet light (screened by a nickel oxide-glass filter), so that the change from α - to β -elaeostearic glyceride apparently depends on the longer wave-lengths. The crystals always appeared in the liquid phase underlying the skin, and the author contends that the drying and characteristic wrinkling proceed independently of, and undisturbed by, crystal formation. The first signs of change in the drying film, which precedes the formation of any detectable film, is shown by the production of irregular patches when the film is breathed upon; it is suggested that this may be caused by orientation in the surface of less soluble polar molecules. Further ageing of a dry film is regarded as the squeezing out of the solvate sheaths (cf. Blom, B., 1927, 371) and a facilitation of condensation processes by the concentration of active colloid complexes in the pressure zones of the haptogen membrane; these changes cause strains which are evidenced experimentally by the typical (strain) double refraction which can be observed in a wood oil film after 2–3 weeks.

E. LEWKOWITSCH.

Olive oil analytical method. II. Use of ultra-violet ray in detection of refined in "virgin" olive oil. S. MUSER and C. E. WILLOUGHBY (Oil & Fat Ind., 1929, 6, [8], 15–16; cf. B., 1929, 137).—As little as 5% of refined olive oil in virgin oil could be detected by the fluorescence in ultra-violet light; below this limit the results were doubtful (cf. Stratta and Mangini, B., 1928, 646) and it was difficult to determine the composition of a mixture containing over 65% of refined oil without a spectrophotometer. Crude cottonseed oil showed a yellow fluorescence comparable with that of virgin olive oil, but all refined olive and other vegetable oils gave a bluish-violet colour, which is attributed to a change in the chlorophyll constituents due to the refining process; a virgin oil which had been heated at 300° for $\frac{1}{2}$ hr. showed a fluorescence equivalent to that of a mixture containing 5–10% of refined oil.

E. LEWKOWITSCH.

Theory unifying drying and heat-polymerisation of fatty oils. B. SCHEFFLE (Z. angew. Chem., 1929, 42, 787–790).—Theories of gel-formation in drying oils are discussed. The tendency of unsaturated compounds to polymerise increases with the number of double linkings and the closeness of their grouping; hence, wood oil, owing to the three conjugated double linkings of elaeostearic acid, has a greater tendency to polymerisation, and consequently depends less on oxygen absorption for the formation of a film than does linseed oil. The small effect of driers in the drying of wood oil is thus explained.

E. LEWKOWITSCH.

Oil from grape kernels. N. KOZIN (Oil Fat Ind. Russia, 1928, No. 8, 24–26).—The kernels of white muscat grapes contained moisture 8.59, fat 13.58, crude protein 11.08, epidermis 35.25, and ash 2.92%. The oil (cold-pressed) had d_{20}^{20} 0.9220, n_D^{20} 1.4678, acid value 3.33, saponif. value 192.57, iodine value 131.24, unsaponifiable matter 0.92%. The oil, which has poor drying qualities, is edible.

CHEMICAL ABSTRACTS.

Piqui-a fruit oils. C. D. V. GEORGI (Malayan Agric. J., 1929, 17, 166–170).—Piqui-a fruit (*Caryocar villosum*)

yields two oils: (a) from the inner layer of the pericarp—a reddish-orange oil which resembles palm oil, has an agreeable odour, and is easily bleached (6.2% on fresh fruit), and (b) kernel oil (0.8% on the fresh fruit, 61.4% of dry kernel), which is yellowish in colour and semi-solid at ordinary temperatures and resembles Sawarri fat (from *Caryocar tomentosum*). The extracted oils had, respectively d_{20}^{20} 0.8622, 0.8617; m.p. 27–28°, 31–32°; saponif. value 204.9, 202.9; iodine value (Wijs) 46.6, 52; acidity 1.1, 0.2%; unsaponif. matter 0.7, 1.3%. The fatty acids of the pericarp oil had titer value 48°, mean mol. wt. 274, iodine value (Wijs) 48.5. Both oils are suitable for edible purposes, but in order to obtain oils of high quality it is necessary to treat the ripe fruits without delay. The outer layer of the pericarp contains 9% of a tannin of a pyrogallol type.

E. LEWKOWITSCH.

Oil from *Hydnocarpus anthelmintica*. C. D. V. GEORGI and G. L. TEIK (Malayan Agric. J., 1929, 17, 171–174).—The fresh seeds contained 31.8% of kernel (26.5% of moisture), which yielded on expression 13.5% (calc. on fresh seed) of a pale yellow, pleasant-smelling oil having d_{20}^{20} 0.9429, n_D^{20} 1.4726, saponif. value 206.4, iodine value (Wijs) 81.5, acid value 1.0, $[\alpha]_D^{25}$ +47.9° in chloroform; the fatty acids had titer value 39.1°, mean mol. wt. 260.5, neutralisation value 215.3, iodine value (Wijs) 84.8, $[\alpha]_D^{25}$ +49.7° in chloroform.

E. LEWKOWITSCH.

Benzene and alcohol as solvents for castor oil. A. SLASHCHIEV (Masloboino Zhir. Delo, 1928, 15, No. 7).—Benzene is a satisfactory solvent for the extraction (12 hrs. at 30–55°) of castor oil, giving the same yields as benzene and carbon disulphide. Oil extracted with ethyl alcohol is impure. Addition to the benzene of 2.5–10% of benzene is advantageous.

CHEMICAL ABSTRACTS.

Spontaneous decomposition of butter fat. E. MUNDINGER (Milchwirt. Forsch., 1929, 7, 292–331; Chem. Zentr., 1929, i, 1162).—Bacterial decomposition of butter fat affords free acids, chiefly of high mol. wt. Such acids are formed in unimportant quantities by chemical decomposition, apparently through the influence of light and air. Small quantities of copper and iron have a similar effect. Rancid butter contains substances which give a precipitate with 2:4-dinitrophenylhydrazine.

A. A. ELDRIDGE.

American reindeer fat. W. F. BAUGHMAN, G. S. JAMIESON, and R. S. MCKINNEY (Oil & Fat Ind., 1929, 6, [8], 11–12).—Five samples of fat from various parts of the carcass of reindeer produced under the usual Alaskan conditions were examined; the characteristics are given having the following limiting values: softening point 40–41°, m.p. 45.8–48.6°, d_{20}^{20} 0.8981–0.8993, n_D^{20} 1.4510, acid value 2–8.6, saponif. value 197.3–199.2 (194.3, kidney fat), unsaponif. matter 0.4%, iodine value (Hanus) 33.7–39.4, acetyl value 5–8, Reichert–Meissl value 0–0.3, Polenske value 0.3–0.5, saturated acids 53.6–59.9%, unsaturated acids 35.2–41.4% (iodine value 90). The saturated fatty acids, separated by the lead salt-ether method and analysed by fractionating their methyl esters, showed the fat to contain the glycerides of the following acids: oleic 36.8, myristic 6.7, palmitic 35.0, stearic 20.5,

arachidic 0.7%, and unsaponifiable matter 0.4%. The presence of arachidic acid and the large amount of myristic acid are noteworthy. E. LEWKOWITSCH.

Naturally occurring saturated fatty acids of high mol. wt. I. So-called arachidic acid and other [higher] saturated acids of arachis oil. D. HOLDE, W. BLEYBERG, and I. RABINOWICZ (Chem. Umschau, 1929, 36, 245—253).—The acids obtainable from the "crude arachidic acid" fraction of an arachis acid oil were rigorously examined and purified by distillation in high vacuum of the esters, and especially of the acids themselves, by fractional crystallisation from solvents, and by fractional precipitation of the lithium salts. A C_{22} -acid, m.p. 75.5—76°, corresponding to the acid described by Ehrenstein and Stuewer (B., 1923, 1031A) as "isobehenic acid," was found to be impure; on very painstaking distillation in a high vacuum the m.p. of the product rose to 79—80°, without change of mol. wt., and the acid was identified (mixed m.p.) as *n*-behenic acid. The "isohexacosic acid" recorded by Holde and Godbole (A., 1926, 268) was also found to be susceptible to fractionation, a product of m.p. 78—79° being obtained, the composition of which approximated to $C_{25}H_{50}O_2$, but which is considered to be probably a mixture of hexacosic and *n*-tetracosic acids. No C_{20} -acid was isolated, but indications of its presence were obtained in the fractions of lowest mol. wt. and in the mother-liquors from the separation of "crude arachidic acid." E. LEWKOWITSCH.

Standard method for the analysis of soap. ANON. (Ann. Falsif., 1929, 22, 325—333).—Instructions are given in detail for sampling, and the determination of moisture, fatty acids, total and free alkali, chloride, glycerol, resin, and foreign (insoluble) matter in "72% soap" (28% moisture content). The methods were drawn up at the request of the Chambre Syndicale of soap manufacturers of Marseilles, and are in accordance with common practice. E. B. HUGHES.

Gelatinising power of soaps. J. HERITES (Z. phys.-chem. Seifenforsch., 1928, 1, 70—72; Chem. Zentr., 1929, i, 1063).—Lüers' pectinometer was employed in comparative determinations, and variations in the results according to the conditions are recorded. A. A. ELDRIDGE.

See also A., Aug., 952, Variation of characters of animal oils (MARGAILLAN). 961, Influence of malting on fat of barley (TÄUFEL and RUSCH). Bonducella nut oil (GODBOLE and others).

Adulteration of butter and ghee. SANYAL.—See XIX.

PATENTS.

Soap paste. J. M. WEISS and C. R. DOWNS, Assrs. to WEISS & DOWNS, INC. (U.S.P. 1,709,294, 16.4.29. Appl., 27.8.27).—An alkyl ester of a dibasic organic acid, e.g., phthalic, maleic, or fumaric acid, is mixed with soap and water, a colloidal clay, and a highly absorptive mineral filler. R. BRIGHTMAN.

Extracting the non-saponifiable and difficultly saponifiable matter from fatty material. J. K.

MARCUS (B.P. 289,798, 30.4.28).—See U.S.P. 1,960,091; B., 1929, 529.

Soap [cakes etc. with antiseptic cores]. A. M. LOW (B.P. 315,997, 17.7.28).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Reflection factors of white paint. F. H. RHODES and J. V. STARR (Ind. Eng. Chem., 1929, 21, 596—599).

—The effects of the addition of small amounts of dark pigments on the brightness and hiding power of typical white paints were examined quantitatively. Brightness of film was determined directly by a Taylor reflectometer, and "ultimate brightness" obtained by extrapolation. As criterion of hiding power is taken the amount of paint per unit area to bring the brightness to within 2% of the "ultimate brightness" for the paint. Small amounts of carbon black or Prussian blue increase the hiding power markedly with only slight decrease in brightness, but on increasing the amount of dark pigment the decrease in brightness becomes more pronounced, particularly so with carbon black. The extent to which Prussian blue can be used is, however, limited by its effect on the tint of the paint. The use of aluminium powder in this connexion, whilst greatly increasing the hiding power, lowers the brightness excessively and in addition gives a "flecked" film; it is therefore unsuitable for use in finishing coats, but should be of advantage in white paint undercoats. S. S. WOOLF.

Torsion viscosimeter for paints. W. BECK (Farben-Ztg., 1929, 34, 2515—2518).—A modified Couette concentric-cylinder torsion viscosimeter is described, and its application to paint material is discussed. S. S. WOOLF.

Oil absorption and particle size [of pigments]. H. WAGNER and H. PFANNER (Farben-Ztg., 1929, 34, 2513—2514).—Contrary to the views of Klumpp (B., 1929, 609), increase of surface involved in grinding a pigment leads to increase in oil absorption. Apparent anomalies are explained by the production, in intensive grinding, of agglomerates or "secondary particles" (illustrated by photomicrographs). It is suggested that the three stages, wetting, smearing, and flowing, be recognised in the determination of oil absorption. S. S. WOOLF.

Influence of light on "toning down" [of colours]. O. MERZ (Farben-Ztg., 1929, 34, 1117—1119).—The dependence of colour on illumination is discussed with special reference to variation in colour shades when viewed by direct and diffused sunlight and by the light of a new sunlight-lamp ("Naturlicht-lampe"). The use of the latter for colour-matching purposes shows satisfactory agreement with midday sunlight, but marked divergence from diffused sunlight. S. S. WOOLF.

Synthetic amyl products as lacquer solvents. M. M. WILSON and F. J. WORSTER (Ind. Eng. Chem., 1929, 21, 592—594).—The properties of synthetic amyl alcohol and acetate ("Pentamol" and "Pentacetate") indicate their suitability for use in nitrocellulose lacquers. Data on distillation and evaporation rates, dilution ratios, viscosity of nitrocellulose solutions, and resin

solvent powers are tabulated. Types of apparatus for determining rate of evaporation of solvents and resistance to "blush" of lacquer films are described.

S. S. WOOLF.

Influence of plasticisers on viscosity and susceptibility to light of nitrocellulose lacquers. H. WOLFF and B. ROSEN (*Farben-Ztg.*, 1929, 34, 2564—2566).—The effect of adding tolyl phosphate, butyl phthalate, "Sipalin MOM," and "Sipalin AOM" to nitrocellulose solutions was examined. With one or two exceptions, progressive decrease in viscosity and increase in dilution ratio (toluene and light petroleum being the diluents used) occur with increase in plasticiser content. Addition of plasticiser is also found to increase progressively the tendency of a nitrocellulose solution to yellow under the influence of actinic rays, tolyl phosphate being the most harmful in this respect, but in all cases plasticised solutions showed better general film properties than a plasticiser-free solution.

S. S. WOOLF.

Viscosity of nitrocellulose solutions. III. O. MERZ (*Farben-Ztg.*, 1929, 34, 2566—2570; cf. B., 1927, 810).—Difficulties arising out of variation in nitrocellulose content of supplies of alcohol-damped nitrocellulose are indicated. The following equation correlating dry nitrocellulose content of a solution of 60 pts. of damp nitrocellulose in 40 pts. of butyl acetate with viscosity of the solution as measured either by efflux or by falling-sphere methods is developed: $t = K_A \cdot K_L^{(0.5p-10)}$, where t is time, K_A and K_L are constants for apparatus and solution respectively, and p is concentration of dry nitrocellulose in the solution. The results of the application of this equation to 120 samples of nitrocellulose are tabulated. The presence of more than 1% of water in the solution is shown to exert a marked influence on the viscosity.

S. S. WOOLF.

Viscous liquid [rosin solution] mixed and dried in 80% less time. R. D. JESSUP (*Chem. Met. Eng.*, 1929, 36, 413—414).—A rosin insulating compound was being prepared by melting lump rosin in rosin oil and heating to remove moisture, the two operations requiring 24—36 hrs. This procedure was improved by arranging continuous circulation between two tanks (into one of which the solid rosin was fed) by an automatic air lift. Melting then required only 2—3 hrs. Dehydration was accelerated by applying a gradually increasing vacuum, and the whole treatment was complete in 5—6 hrs.

C. IRWIN.

Turpentine from sulphate-cellulose. POSTOVSKI and PLUSNIN.—See V. **Water content of turpentine.** NICOL. See XX.

PATENTS.

Nitrocellulose coating composition. M. V. HITT, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,710,453, 23.4.29. Appl., 13,10.23).—Pyroxylin coating composition having a viscosity of 100—300 centipoises at 28° and containing 7—9% or more of nitrocellulose and above 15% of total solids, excluding pigment, is prepared by mixing 6 pts. of nitrocellulose of viscosity below 1500 centipoises in ethyl acetate at 28° with about 1—4 pts. of acetyl-laurin, benzyl butyl phthalate,

butyl phthalate, or other solvent softener, less than 3 pts. of resin, about 6 pts. of a low-boiling alcohol, 6—18 pts. of a high-boiling ketone or ester, and 3—5 pts. of a pigment.

R. BRIGHTMAN.

Drying method and device, particularly for drying lacquered articles [by ozonised air]. HUNDT & WEBER GES.M.B.H. (B.P. 303,875, 12.7.28. Ger., 12.1.28. Addn. to B.P. 297,411, 21.9.27).

Manufacture of linoleum or like material. LINOLEUM MANUF. Co., LTD., and A. A. GODFREY (B.P. 316,646, 2.5.28).

Manufacture of plates from resins etc. [reinforced by metal sheets]. "HEROLD" A.-G. (B.P. 303,103, 27.9.28, Ger., 28.12.27).

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanisation [of rubber] without sulphur. L. ECK (*Gummi-Ztg.*, 1929, 43, 2481—2482).—A critical review of the claims made by Ostromyslenski as to the possibility of satisfactory vulcanisation without the use of sulphur (cf. B., 1916, 59). D. F. TWISS.

Weighing. PARTRIDGE. **Plastometer.** KARRER.—See I.

PATENTS.

Impregnation of permeable substances with rubber latex. P. H. HEAD (B.P. 316,335, 29.3.28).

Surface coatings of ebonite, hard rubber, or the like. H. F. W. MENZEL (B.P. 316,353, 1.5.28).

Paper-like material (U.S.P. 1,716,654).—See V.

XV.—LEATHER; GLUE.

Chrome tanning. G. A. BRAVO (*Boll. Uff. Staz. Sperim. Ind. Pelli*, 1929, 7, 166—181, 192—205).—Experiment shows that the insolubilisation of skins by the action of sunlight or ultra-violet light after treatment with potassium dichromate is similar to that of dichromate-gelatin, although, as would be expected from the different chemical and physical structural characteristics of gelatin and collagen, the two phenomena follow different quantitative courses. T. H. POPE.

Dextrin from corn. LINK.—See XIX.

XVI.—AGRICULTURE.

Microbiological investigation on the virgin and arable volcanic soils from Sakura-jima, Japan. A. ITANO and S. ARAKAWA (*Ber. Ohara Inst. Landw. Forsch.*, 1929, 4, 27—33).—Results of bacteriological investigations made of the various soils from a volcanic island off Kyushu, Japan, are given. E. A. LUNT.

Soils in rice fields. II. General microbiological investigation. III. Microbiological analyses in rice fields and dry farm soils. A. ITANO and S. ARAKAWA (*Ber. Ohara Inst. landw. Forsch.*, 1929, 4, 35—54, 55—66; cf. B., 1927, 611).—II. Quantitative determinations have been made on the soil taken from a typical Japanese rice field, which is flooded for certain portions of the year, with reference to the variation with season of the following: total number of fungi and of bacteria, and of each of the following bacteria classi-

fied functionally: ammonifying, nitrifying, denitrifying, nitrogen-fixing, and cellulose-fermenting.

III. The soil from a Japanese rice field was found to have a higher C:N ratio than that from a typical unflooded farm soil, and to contain less bacteria capable of reducing nitrates to nitrites. The rice field contained some indole-producing strains, whilst the dry soil contained none. Details are given of the numbers of the various groups in all the strata examined. E. A. LUNT.

Corrosiveness of soils with respect to iron and steel. H. D. HOLLER (Ind. Eng. Chem., 1929, 21, 750—755).—It is theoretically probable that the rate of corrosion of iron in soil is related to the rate at which hydrogen is produced at, and removed from, the surface of the iron. The only factors affecting the production of hydrogen and capable of separate control are acidity and salt content, it having been found that particle size is unimportant. Attempts to establish a relationship between these two factors and corrosiveness on 47 samples of soil from all over the United States lead to no very clear result, but there is a general tendency towards increased corrosion with increased acidity. The acidity in turn tends to vary directly with the annual rainfall. A low p_H value will not necessarily mean long-continued corrosion unless there is sufficient colloidal matter present to "buffer" it at its low value. A method of determining total acidity by titrating the soil in a 5% solution of potassium chloride with a hydrogen electrode was worked out. Consistent results are only obtained with the addition of potassium chloride. This in many cases lowers the p_H value through base exchange, and the results so obtained are considered to be a fair measure of total acidity. The end-point of the titration is taken as p_H 9.4, at which point iron ceases to corrode. A general relation is also shown to exist between amount of hydrogen evolved in contact with iron and acidity determined as above.

C. IRWIN.

Buffering, acid density, soil zones, and a single-value unit describing soil conditions. IV. S. GOY, P. MÜLLER, and O. ROOS (Z. Pflanz. Düng., 1929, 14A, 220—240. Cf. B., 1928, 796; 1929, 335).—Buffer values of soils increase considerably as the neutral point is approached. To clarify the meaning the term "general acid density" is introduced and is defined as the ratio of buffer value to base equivalent. The value is independent of the nature of the base used in the titration. To eliminate the possible effect of changes of acidity on the nature and condition of the soil colloids the term "specific acid density" is utilised to express the above relationship for a definite range of p_H values, and as a means of expressing acidity conditions in soils. The general classification of soils and the significance of the electrometric measurements previously described are discussed in the light of the above values.

A. G. POLLARD.

Elution and decomposition of synthetic urea in the soil with reference to climatic conditions. R. BUS (Fortschr. Landw., 1929, 4, 1—4; Chem. Zentr., 1929, i, 789).—Urea (0.33, 1.66, or 3.33%) was added to a sandy or loamy soil in pots exposed to natural conditions. With small applications, the nitrogen was completely washed out of the sandy soil

in 6 weeks; with loam the large applications were most quickly removed. The nitrate ion was, in both soils, more completely although more slowly removed than the ammonium ion. In sandy soils the amounts of urea applied are parallel to the production of nitrate. Hence nitrification, but not ammonification, and elution are parallel. A. A. ELDRIDGE.

Changes in the solubility of the phosphoric acid of soils in different biological conditions. L. VON KREYBIG (Z. Pflanz. Düng., 1929, 14A, 240—251).—The phosphate manuring of acid soils without appropriate liming is seldom profitable. The Neubauer values for phosphorus in weakly acid or non-acid soils are high and their true significance depends on a consideration of the biological conditions of the soil. A. G. POLLARD.

The phosphate question [in soils]. II. Phosphate analysis. O. ARRHENIUS (Z. Pflanz. Düng., 1929, 14A, 185—194).—The molybdenum-blue method for the determination of phosphates, involving the reduction with sodium sulphite, is examined, and the following details are prescribed for its satisfactory working:—The portion of soil extract (usually citric extract) for analysis is diluted with water to about 80 c.c., and to it are added 10 c.c. of molybdate solution, 1 c.c. of sulphite solution, and 1 c.c. of quinol solution. The volume of the mixture is adjusted to 100 c.c. and the whole placed in a water-bath at 55° for 6 hrs. After keeping at ordinary temperature overnight the colour is compared with those of standard solutions similarly prepared. The latter should contain phosphate in quantities of not more than 0.03 mg. P_2O_5 difference in consecutive members of the series. Solutions containing more than 0.6 mg. are not easily compared by this means. The adverse effect of citric acid on the production of the blue colour is eliminated by heating to 55°. The ammonium molybdate solution contains 25 g. dissolved in 825 c.c. of water, followed by 175 c.c. of sulphuric acid added slowly, with cooling. The sodium sulphite solution contains 20 g. in 80 c.c. of water. The quinol solution (0.5%) is acidified with one drop of sulphuric acid. (Cf. B., 1929, 756.) A. G. POLLARD.

Favourable action of sulphur [on soils]. Colloidal sulphur. M. J. BOREAS (Ann. Sci. agron. franç., 1929, 45, 128; Bied. Zentr., 1929, 58, 357—358).—Crop increases are recorded following the application to soil of colloidal sulphur obtained by the treatment of solutions of sulphur in sulphur compounds with weak acids, e.g., carbon dioxide from the air.

A. G. POLLARD.

Mechanical analysis of soils. IV. M. KÖHN (Z. Pflanz. Düng., 1929, 14A, 268—280).—The criticisms of Köttgen and Heuser (B., 1929, 369) are answered. The use of glycerin and glycerin-water mixtures by these authors in the pipette method of mechanical analysis is condemned as unsound in theory, cumbersome, and tedious.

A. G. POLLARD.

Improved form of Schulze apparatus for [soil] elutriation. E. RAUTERBERG (Z. Pflanz. Düng., 1929, 14A, 261—268).—The Schulze-Harkort elutriator (cf. B., 1928, 506) is adapted for use in soil analysis. Details of soil preparation and comparative results are recorded.

A. G. POLLARD.

Determination of the fertiliser requirement of soil. J. HIRSCH (Fortschr. Landw., 1928, 3, 1118—1121; Chem. Zentr., 1929, i, 1043).—A comparative study. Neither the chemical nor Neubauer's method gave useful results, although these were attained by Mitscherlich's pot experiments. A. A. ELDRIDGE.

Determination of the phosphoric acid requirement of soil. F. SCHEFFER (Fortschr. Landw., 1929, 4, 37—40; Chem. Zentr., 1929, i, 1043—1044).—Niklas' *Azotobacter* method is modified; a simpler method consists in shaking the soil with calcium hydrogen carbonate solution and determining colorimetrically the phosphate in the filtrate. A. A. ELDRIDGE.

Exchangeable potassium [in soils]. K. BAMBERG (Z. Pflanz. Düng., 1929, 14A, 177—184).—In peat soils saturated with potassium, the latter is more easily displaced by calcium and magnesium than by sodium or ammonium. Ammonium chloride displaces more potassium than does ammonium acetate. The reaction of the soil plays little part in this displacement. In the same soils after the destruction of organic matter the potassium is most easily displaced by ammonium, followed in order by calcium, magnesium, and sodium. The greater part of the potassium in natural peat soils is held by the clay complex. The result of varying the concentration and nature of the leaching solution in the determination of the displaceable potash is examined. Comparison is made of the displaceable and assimilable potash as determined by Hissink's method. A. G. POLLARD.

Potash-iron antagonism in plants as the basis of a method for the determination of the potash requirement of soils. O. ECKSTEIN and A. JACOB (Z. Pflanz. Düng., 1929, 14A, 205—220).—Examination is made of Hoffer's reaction (Purdue Univ. Bull., 1926) in which the inverse relationship of the iron and potash contents of the nodes of maize stems is utilised to determine the potassium requirement of soils. Trials are described with varying soil types and fertiliser treatment, and results are compared with those obtained by the Neubauer method. In general, Hoffer's work is confirmed but some exceptional cases remain unexplained. Similar phenomena occur with other plants. The antagonism between certain ions in plant nutrition is not an isolated phenomenon, but is interrelated with the numerous other factors concerned. A. G. POLLARD.

Determination of total replaceable bases in soils. R. H. BRAY and F. M. WILLHITE (Ind. Eng. Chem. [Anal.], 1929, 1, 144).—The soil is leached with a solution of ammonium acetate and the filtrate evaporated to dryness. After ignition to convert acetates into carbonates or oxides an excess of standard acid is added and the excess titrated with standard alkali. The method is satisfactory for sodium, potassium, calcium, magnesium, barium, and manganese. Iron and aluminium give no titration values. J. S. CARTER.

Determination of the unsaturation and lime requirement of soils on the basis of their hydrolytic acidity. J. VON CSIKY (Z. Pflanz. Düng., 1929, 14A, 281—294).—The numerous recent processes for

determining the lime requirement of soils are discussed. Comparison with field trials shows that the lime required to produce the maximum yield frequently exceeds that calculated from the hydrolytic acidity. The extent to which this occurs varies to some extent with the crop. More satisfactory results are obtained by increasing the proportion of N-calcium acetate solution used in determining the hydrolytic acidity to the order of 1:60, and revising the empirical factor introduced into the calculation. Even with this modification agreement between field trial and laboratory calculation is not entirely satisfactory. A. G. POLLARD.

Preparation of humus extracts with neutral agents. K. SIMON (Z. Pflanz. Düng., 1929, 14A, 252—257).—Humus may be extracted from soil without decomposition by treatment with ten times its weight of cold 1% sodium fluoride solution. On evaporation the dark brown, water-soluble alkali humate is obtained. The humic acid obtained by the acidification of the above extract differs in certain points from acidum huminum (Merck). It may be used with advantage as a standard material for comparative soil examination. A. G. POLLARD.

Respiration of sorghum grains. D. A. COLEMAN, B. E. ROTHGEB, and H. C. FELLOWS (U.S. Dept. Agric. Tech. Bull., 1928, No. 100, 1—16).—At high humidities damaged kernels respire more vigorously than do normal kernels. Storage of the grains is discussed. CHEMICAL ABSTRACTS.

Saltpetre [fertiliser] question. K. NEHRING (Fortschr. Landw., 1929, 4, 40—42; Chem. Zentr., 1929, i, 1042).—Spraying with potassium iodide is injurious to oats. Relative grain yields were: German sodium nitrate 100, Chile saltpetre 98, Leuna saltpetre 96, "Kalksalpeter" 94, ammonium sulphate 95, sodium nitrate (0.5 g. N) with potassium iodide (5 mg.) 104. A. A. ELDRIDGE.

Method for comparing the value of ammoniacal nitrogen and nitrate-nitrogen [for plants]. W. G. FRIEDEMANN (Science, 1929, 70, 43—44).—Details are given for the preparation of water culture media suitable for the comparison of the nutrient values for plants of ammoniacal and nitrate-nitrogen. Adjustments in the p_H values of the solutions are easily made. A. G. POLLARD.

Effect of dark and light periods on the nutrition of plant roots. M. K. DOMONTOVITSCH and A. J. GROSCHEKOV (Z. Pflanz. Düng., 1929, 14A, 194—205).—The growth of plants in water cultures was compared under controlled conditions of light and darkness and in solutions alternately lacking and containing one of the principal plant nutrients. Such "differential" nutrition in the case of phosphate had no detrimental effect on the dry matter production of plants. Nutrition was increased by a small but definite extent by light. The effects of differential feeding with potassium were slight, and light was practically without influence on the assimilation of this element. During the differential feeding of the plants with calcium growth practically ceased. The assimilation of inorganic radicals was affected by light in the order calcium (greatest), magnesium, nitrate, sulphate, ammonium, phosphate,

potassium. Light increased the absorption of water by plants. The transpiration process and the absorption of inorganic matter are not closely related.

A. G. POLLARD.

Analysis of insecticides containing fluorine compounds. L. HART (Ind. Eng. Chem. [Anal.], 1929, 1, 133—135).—Methods are described for the analysis of preparations containing (a) alkali silicofluorides and boric acid; (b) fluorides with arsenic trioxide or soluble arsenic compounds; (c) fluorides, bifluorides, and silicofluorides. (a) Total acidity with respect to 0.2N-sodium hydroxide solution is first determined and then acidity due to boric acid after removal of silicofluoride by precipitation as the potassium salt in the presence of 1:2 alcohol (cf. Katz, B., 1904, 562; Dinwiddie, B., 1916, 1216). (b) Arsenic is precipitated by 10% silver nitrate solution and the precipitate distilled with hydrazine sulphate, followed by titration of the distillate with 0.05N-iodine solution. After removal of arsenic, fluorine is determined by precipitation with 10% calcium chloride solution, ignition of the calcium fluoride, and confirmation by converting into calcium sulphate. (c) Total acidity due to bifluorides and silicofluoride is determined by titration with 0.2N-sodium hydroxide solution, and bifluoride acidity by titration at 0° with 0.2N-sodium hydroxide in the presence of potassium chloride and 1:1 alcohol. The difference is the silicofluoride equivalent. Total fluorine is determined by precipitation, as the calcium salt, and hence the sodium fluoride content is found by deducting the fluorine equivalent of the bifluoride and silicofluoride present (cf. Flisik, B., 1925, 353).

H. J. DOWDEN.

Competition between cultivated plants and soil micro-organisms for mineral food; action of dried blood on phosphate fertiliser. D. CHOUGHAK (Compt. rend., 1929, 189, 262—264).—In confirmation of earlier work (*ibid.*, 1927, 185, 82) in which it was suggested that the effect of micro-organisms in the soil in decreasing the yield of crops was due to the removal of phosphates by these organisms, the effect of the addition of varying doses of organic nitrogen in the form of dried blood on soils to which different amounts of disodium phosphate had been added, has been investigated. With small, insufficient doses of phosphate a large amount of dried blood destroys the crop. By successive exhaustions of soils, in atmospheres of different humidity (35% and 25%), to which the same quantity of phosphate but different amounts of dried blood were added, it was shown that in the first series (humidity 35%) addition of large quantities of dried blood completely removes the phosphate, whilst in the second series (25% humidity, good aeration) the phosphorus is found almost entirely in an organic form in which it is less readily assimilated by the plant. Addition of traces of chloroform destroys the micro-organisms and the phosphate content then diminishes slowly and regularly, the ratio of mineral to organic phosphorus remaining constant.

J. W. BAKER.

Effect of fluorine in dairy cattle ration. G. E. TAYLOR (Mich. Agric. Exp. Sta. Quart. Bull., 1929, 11, 101—104).—Fluorine (0.125 % of ration) is detrimental; raw rock phosphate (containing 3% F) was inferior to steamed bone meal.

CHEMICAL ABSTRACTS.

See also A., Aug., 876, Wetting power [of insecticides] (GREEN). 899, Colorimetric determination of phosphoric acid (LEVITZKI). 958, Fungicidal action of sulphur (MARSH). 960, Micro-detection of germinable seeds (NIETHAMMER). Translocation of potassium in tomato plants (JANSSEN and BARTHOLOMEW). Determination of inorganic nitrogen in plant extracts (SESSIONS and SHIVE). Moisture in wheat grown under constant conditions (VAN DE SANDE-BAKHUYZEN). 961, Catalase and oxidase of tomatoes and the soil reaction (HABER). 962, Occurrence of chlorates in tomato soil (OWEN).

Plant nutrient content of mud from beet-sugar factories. NOVÁK.—See XVII. Nitrogen in tobacco. VICKERY and PUCHER.—See XX.

PATENT.

Recovery of products [fertilisers] from spent sulphite liquors. G. A. RICHTER, ASST. to BROWN Co. (U.S.P. 1,710,272, 23.4.29. Appl., 6.1.27).—Spent sulphite liquors containing ammonium salts are neutralised with ground basic calcium phosphate rock, evaporated to 5% concentration, dried, and used as fertiliser. A potassium salt may be added to the spent lye or to the original digesting solution to increase the fertilising value.

R. BRIGHTMAN.

XVII.—SUGARS; STARCHES; GUMS.

Drying of beet slices and its influence on sugar content. B. ZIMMERMANN (Z. Zuckerind. Czechoslov., 1929, 53, 554—558).—On exposure to the air for 2 hrs. piles of fresh beet slices and beet pulp, weighing 500 g. each, lost 1.3—4.0% of their weight by evaporation of moisture. Evaporation during the pulping of slices in a Keystone pulping apparatus did not exceed 0.5%. It is concluded that, in factory control, errors of 0.1% in the sugar content of beet slices may easily arise owing to evaporation before analysis.

J. H. LANE.

Heating of [sugar] thin juice under pressure. E. THIELEPAPE and P. MEIER (Z. Ver. deut. Zucker-Ind., 1929, 79, 316—326).—The practice of boiling-up thin juice under pressure, e.g., for 10 min. at 116—120°, before evaporation (cf. B., 1928, 831) does not cause any appreciable decomposition of sugar. Its chief advantage is that it avoids evolution of carbon dioxide in the evaporating plant, the condensate from which thus becomes much less liable to cause corrosion when used as boiler feed water than under present conditions. Experiments indicate that about 0.1—0.3 g. of carbon dioxide may be evolved per kg. of thin juice. By far the greater part of this is derived from organic non-sugars. Under practical conditions the boiling of the thin juice lowers the alkalinity very slightly, and may also produce a slight precipitate containing calcium carbonate which can be removed by a pressure filter. Satisfactory factory trials have been made using two old evaporator units for the pressure boiling.

J. H. LANE.

Microscopical investigations on the carbonatation process. T. BREDT (Z. Ver. deut. Zucker-Ind., 1929, 79, 285—315).—On the lines of an earlier study by Claassen (B., 1920, 554 A) the precipitate produced in

beet juices by carbonatation under various conditions and at different stages was examined, a special solution of fuchsin being used as a stain for coagulable colloids. The precipitate contained flocks of colloidal impurities, besides soft, porous spherules, 1–16 μ in diam., consisting of crystal aggregates more or less optically anisotropic and interpenetrated with colloids, and also individual crystals. With the progress of carbonatation the colloidal matters showed increasing intensity of staining, indicating progressive dehydration and coagulation. This process appears a more important subject for further study than the mechanism of precipitation of calcium carbonate. The ease of filtration of carbonatated juice is not always correctly indicated by the rate of deposition of the precipitate in a test cylinder. Colloidal matters in a highly hydrated form may remain invisible in the supernatant juice and yet be capable of dehydration and coagulation under suitable conditions, e.g., by liming and carbonatating at higher temperatures.

J. H. LANE.

Behaviour of phosphoric acid during liming and carbonatation in relation to the natural alkalinity [of beet juices]. O. SPENGLER and A. TRÄGEL (Z. Ver. deut. Zucker-Ind., 1929, 79, 457–462).—Experiments with diffusion juice, and with pure sugar solutions of like contents of sucrose and phosphoric acid treated with varying quantities of sodium hydroxide to produce different natural alkalinities (B., 1928, 421), showed that, given natural alkalinities of 0.004–0.1%, phosphoric acid is almost completely eliminated from beet juice by carbonatation. The presence of large proportions of phosphate in incrustations from the first evaporator unit, which has been observed, is probably attributable to incomplete decomposition of calcium glycerophosphate during liming, this compound then remaining in solution after carbonatation and undergoing decomposition in the evaporator. Given a sufficient natural alkalinity of the juice, however, the liberated phosphoric acid should remain in solution as sodium phosphate and not be deposited as calcium salt.

J. H. LANE.

Clarification of diffusion juice in relation to the possible inversion of sucrose and influence of micro-organisms. V. MAREŠ (Z. Zuckerind. Czechoslov., 1929, 53, 728–732).—Comparative laboratory experiments with a diffusion juice of normal composition showed that if, instead of heating the juice before addition of lime, $\frac{1}{2}$ of the total quantity of lime is added before heating and the rest afterwards, in order to reduce risk of inversion of sucrose, the purification effected by carbonatation may be somewhat impaired; e.g., in the present case, the final thin juice contained 0.067% instead of 0.057% of total nitrogen. In cases of infection of the raw juice with yeasts or moulds, however, it is advisable to add about $\frac{1}{2}$ of the total quantity of lime to the juice in the measuring vessels, as very considerable inversion may otherwise occur.

J. H. LANE.

Sugar-factory filter cloths. IV. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1929, 53, 569–576; cf. B., 1929, 572).—Laboratory tests were made on the effect of varying pressures on the strength of different types of filter-cloth as used in beet-sugar factories. A pressure of

55 kg./cm.² distinctly diminished the strength on new jute cloth, whilst that of used cloth of the same material was considerably weakened. Cotton cloth commenced to weaken after having been submitted to pressures of 110 kg. Cotton cloth which had already been used in filter-presses retained its strength fairly well after pressure in a single layer, but folded pieces easily broke. Contact with a rusting surface greatly diminishes the strength of all types of cloth. Resistance to the blows of a hammer is best borne by new cotton, jute and linen cloths being, in general, much weakened. Permeability in all types of cloth was much diminished by the presence of incrustation (artificially produced), most of all in the case of jute, but also to a marked extent in that of a mixture of cotton and linen. Cotton cloth, on the other hand, showed a relatively good rate of flow through incrustated cloth under the experimental conditions (in which solutions of molasses of 15° and 26° Bg. were filtered).

J. P. OGILVIE.

Determination and nature of the alkalinity of raw [beet] sugars. V. PREININGER (Z. Zuckerind. Czechoslov., 1929, 53, 617–629).—Conductometric titration and chemical analysis of raw beet sugars indicate that their alkalinity is due mainly to basic substances of the buffer class, carbonates accounting for about $\frac{1}{3}$ – $\frac{1}{2}$. The suitability of raw sugars for storage is better ascertained by determinations of p_H value than by titration, sugars having p_H 7–8 being unsuitable for long storage, whilst those having p_H < 7 are specially liable to inversion. The p_H value can be determined with sufficient accuracy (to within 0.3 of the electrometric value) by colorimetric methods, two of which are described. The first consists in comparing the colour produced by 10 drops of a 0.5% solution of phenolphthalein or a 0.04% solution of bromothymol-blue, in a solution of 8 g. of sample in 20 c.c. of water (complete dissolution of the sugar is not essential), with that produced in standard borate buffer solutions, by means of the Walpole comparator. The second method is based on the use of Šandera's photocolormeter (B., 1928, 344).

J. H. LANE.

Denaturing of raw sugar. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1929, 53, 561–567).—Raw beet sugars can now be used (in Czechoslovakia) as fodder, free from tax, if denatured with 2% of salt and 1% of spent vegetable char or 3% of spent animal char in the case of first runnings, or 0.75% of vegetable or 2% of animal char in the case of second runnings. The admixed carbon is not injurious to the animals.

J. H. LANE.

Speed of crystallisation of sucrose from beet products. M. I. NAKHMANOVICH and I. F. ZELIKMAN (Nauch. Zapiski, 1928, 6, 109–122).—For materials of high (but not low) purity the rate of crystallisation increased rapidly with increasing supersaturation; increase in the concentration of non-sugars lowered the speed of crystallisation of sucrose. Differences in laboratory and factory results are attributed to insufficient diffusion in the crystallisers.

CHEMICAL ABSTRACTS.

Cane molasses and solid sugar-cane juice. L. ROSE (Z. Spiritusind., 1929, 52, 254–255).—The polarisation, inversion, and Fehling's solution, and fermenta-

tion methods of evaluating these raw materials for alcohol production are compared. The usually employed inversion-copper reduction method gives 2–3% higher results than the fermentation method with molasses, but only slightly higher results with dried juices. Exceptions occur when the juices have been overheated, as shown by browning. With molasses the polarisation + invert figures are slightly higher still, probably owing to over-determination of the invert. For the latter determination an unclarified solution is preferred. With solid juices the double polarisation figure is low, owing to the formation of inactive but fermentable sugars during drying. The differences between all three methods increase as the quality of the products examined deteriorates. The results of duplicate determinations of five typical examples of each are tabulated, the alcohol yields from the molasses being 31.9–37.9 litres/100 kg., and from dried juices 52.6–56.3 litres/100 kg. The technical process employed is described, and the yield for each raw material is given as molasses (60.2% of sugar) 32 litres, dried juice (89% of sugar) 50 litres of alcohol per 100 kg. over a typical period. F. E. DAY.

Relation of the amides to alkalinity decrease during beet-sugar manufacture. J. VONDRÁK (Z. Zuckerind. Czechoslov., 1929, 53, 537–542).—Beets grown under dry conditions yield juice the alkalinity of which decreases during manufacture, particularly in evaporation. Such juice contains more total nitrogenous substances including amides than that obtained from normal roots. The proportion of glutamin, however, is smaller. J. P. OGILVIE.

Plant nutrient content of mud from clarifying basins of [beet-]sugar factories. J. NOVÁK (Z. Zuckerind. Czechoslov., 1929, 53, 717–718).—Analysis of the mud from the effluent clarifying basins gave: total nitrogen 0.03, phosphoric acid 0.28, potash 0.14, calcium oxide 1.06, water 14.4%. Pot cultures by Mitscherlich's method showed that of the total plant nutrient present in the mud the following percentages were taken up by oats during their growth: total nitrogen 13, potash 33%, phosphoric acid none.

J. P. OGILVIE.

Graphical method for calculating the composition of a sugar mixture [sucrose, dextrose, lævulose]. J. DUBAQUIÉ (Ann. Falsif., 1929, 22, 352–353).—Instructions are given for the drawing of curves and the preparation of a movable triangle from which the amounts of sucrose, dextrose, and lævulose in a mixture of these are readily calculated. The analytical determinations necessary are polarisation before and after inversion, and the total reducing sugars (Fehling) after inversion. E. B. HUGHES.

Determination of the solubility of white dextrins and soluble starches. O. WOLFF (Z. Spiritusind., 1929, 52, 248–249).—Though the solubility of dextrins should strictly be determined under the actual conditions of their employment, this is often impossible owing to difficulties of dissolution, filtration, etc. For comparative purposes a solution containing 2% by wt., prepared by stirring for 30 min. with water at exactly 20°, is recommended. The temperature is important, and in an example a rise of 2° raised the solubility from 53.8%

to 57% on a 1-hr. extraction. An aliquot part of the filtered extract is carefully evaporated and the residue weighed. For rapid working a refractometric or pycnometric method might be calibrated from a series of such determinations. For the characterisation of a dextrin a viscosity determination on a warm aqueous extract is also necessary. F. E. DAY.

New starch tables. SPROCKHOFF (Z. Spiritusind., 1929, 52, 238).—An extension of the author's previous tables (*ibid.*, 1922). The amount of starch per 100 litres and per 100 kg. of suspension, and the volume and weight of suspensions containing 100 kg. of starch, are given for each degree to 24° B. The figures are given for dry starch and for wet starch containing 20 and 50% of water. F. E. DAY.

See also A., Aug., 914, **Potato starch** (HESS and SMITH). 948, **Iodometric determination of dextrose** (TOSCANI). 957, **Alcoholic fermentation of amino-acids** (PARISI and others).

Adsorptive characters of charcoals. LANDT and BHARAVA.—See II. **Cane-molasses fermentations.** HILDEBRANDT. **Dextrin-fermenting yeasts.** STAIGER and GLAUBITZ.—See XVIII.

PATENTS.

Increasing the efficiency of mash tubs for massecuite and the like. A. RÖLZ (B.P. 297,054, 10.9.28. Ger., 13.9.27).—A mixing vessel for massecuite, provided with stirring blades attached to a rotating horizontal shaft, has a fixed perforated pipe extending along its lower part, through which a liquid can be extruded into the massecuite intermittently, the valve controlling the flow of the liquid being actuated by a cam on the rotating shaft. J. H. LANE.

Crystallisers for sugar manufacture. DUNCAN, STEWART & Co., LTD. From J. E. R. HERRISON (B.P. 313,290, 21.5.28 and 1.1.29).—In a crystalliser for massecuite, with stirring arms mounted on a horizontal rotating shaft extending longitudinally through the apparatus, horizontal tubes through which cooling liquid flows are mounted on the shaft in radial sets, thus serving as means for stirring and cooling the massecuite at the same time. J. H. LANE.

Decolorisation and purification of saccharine materials. H. B. WOOLDRIDGE and P. G. CLARK (B.P. 312,705, 6.3.28).—To improve the quality of low-grade cane or beet syrups or molasses, for yeast manufacture, for use in sugar refining, or for alimentary purposes, they are diluted to about *d* 1.07, passed through a filter press the cloths of which have previously been coated with a filter aid such as Celite, and then allowed to percolate through filters containing some form of bauxite or precipitated alumina, or mixed therewith and filtered. The original syrup may first be defecated by heating with phosphoric acid, neutralising with lime, and removing the precipitated sludge.

J. H. LANE.

Revivification of used kieselguhr [from sugar refineries]. R. CALVERT (U.S.P. 1,717,661, 18.6.29. Appl., 25.10.24).—The used kieselguhr, in the form of

wet filter cake, is disintegrated by means of a blower in a current of hot gas containing oxygen, and then blown into a chamber at a higher temperature, *e.g.*, 400°; after exhaustion from this chamber the dry dust is separated from the gas.

J. H. LANE.

XVIII.—FERMENTATION INDUSTRIES.

Sinker test in malt analysis. L. C. WILSON (J. Inst. Brew., 1929, 35, 414—415).—If the percentage of sinking corns is high, the malt will be irregular in growth and show many badly modified and hard-ended corns. Analyses of the sinking corns, which have been separated from the bulk of the malt, give values for the extract, cold-water extract, and diastatic power which are considerably lower than those obtained with the original malt. It is considered that the sinking corns are responsible for the variations in the extract values returned by different chemists. C. RANKEN.

Formation of buffer substances in malting. R. H. HOPKINS and H. E. KELLY (J. Inst. Brew., 1929, 35, 402—409).—About one half of the buffer substances preformed in barley are removed during steeping, with a resultant fall in the p_H value. During the first few days of germination a rapid increase both in phosphate and protein buffers occurs along with a pronounced rise in the p_H . Comparatively little change in the content of buffer substances occurs during the subsequent days on the floor, but the p_H almost invariably falls during kilning. The amount of buffer substances is not affected by "stewing" the withered green malt at 32° and 46° for 48 hrs. There appears to be no relationship between the p_H or the buffer content of the malt on the one hand and the ordinary analytical results on the other. The precise condition of growing may affect the p_H more readily than the buffer content.

C. RANKEN.

Staining of bottom-fermentation beer yeasts with methylene-blue. H. HAEHN and M. GLAUBITZ (Woch. Brau., 1929, 46, 315—320).—Not only dead cells, but those which, though weak, are capable of reproduction, are stained by methylene-blue and other dyes. By treatment for about 24 hrs. with 0.5% lactic or 0.1% sulphuric acid, preparations can be made in which all the cells stain, though fermentation and reproduction occur on adding the yeast to wort. The proportion of stained cells in normal yeasts, which may have been weakened by storage in an ice chest, depends on the concentration of the staining solution and the duration of its action. Methylene-blue of 0.05% concentration gives in most cases nearly twice as many stained cells as a 0.01% solution, and the latter concentration gives approximately the same number of deeply stained (dead) cells as are judged to be dead from examination of an unstained sample. The lightly stained cells are to be regarded as weakened. The stained preparations should be examined without delay, since a 15-min. contact with the dye increases the number of stained cells by a large but irregular amount. Sampling is important, as in one case different parts of the mass gave from 7% to 52% of stained cells. The decrease of fermentative power during storage is greater than the proportional increase of weak cells as shown by

staining; thus, 3.18 g. of a yeast containing 26.5% of stainable cells produced only 3.9 c.c. of carbon dioxide in 2 hrs., whereas 2.5 g. of the original yeast produced 14.8 c.c. Though the proportion of stainable cells is not directly related to fermentative power, it is a useful guide to the fitness of yeast. F. E. DAY.

Dextrin-fermenting yeasts. STAIGER and GLAUBITZ (Z. Spiritusind., 1929, 52, 243).—The yeasts Logos, Race 12, and *Schizosaccharomyces Pombe* were compared as regards their fermentative powers on dextrin, dextrinous wort, and wort containing diastase. All fermented at similar rates and to similar alcohol yields, *S. Pombe* being slightly the weakest, but none fermented dextrin except in presence of diastase. The authors' previous conclusion (B., 1925, 936) that Logos and Pombe yeasts are not capable of fermenting or hastening the fermentation of dextrin is confirmed.

F. E. DAY.

Determination of fermentative power of pressed yeasts from the point of view of bread-making. E. and L. ELION (Bull. Soc. Chim. biol., 1929, 11, 724—730).—The best method of comparing yeasts as to suitability for bread-making is by measurement of the carbon dioxide evolved by a flour cake made with the yeast in question.

J. H. BIRKINSHAW.

Sediment yeast and pumping off in the fermenting cellar. F. WINDISCH (Woch. Brau., 1929, 46, 308—310).—The superiority of the sediment yeast over that in suspension during primary fermentation is more marked when weak yeast (*e.g.*, deposit from the secondary fermentation) is used for pitching. Dead and weak cells tend to remain in suspension to the end of the primary fermentation, and when weak pitching yeasts are used are present in an excessive proportion in the raw beer. Pumping off of the fermenting wort is considered to be of value in some cases, but is to be avoided where weak yeasts are used. F. E. DAY.

Boiling of wort in relation to the primary and secondary fermentation. R. HORCH and SCHULTEIS (Woch. Brau., 1929, 46, 295—298).—An auxiliary boiling apparatus, consisting of a cylindrical casing, open below and containing steam-heated tubes, covered with a loose cap carrying three outlet tubes, was fitted in a steam-heated copper. By its vigorous boiling in the casing the wort is driven through the outlet tubes, which are so arranged that a rotation is imparted to the cap. The wort so ejected maintains an active agitation of the contents of the copper and breaks down any excessive froth. By its use the authors find that the time of boiling can be reduced from 2 to 1½ hrs., and that the evaporation rate is increased from 4—5% to 8—9% per hr. On the cooler 10—20% more sludge was collected, and this was denser and less voluminous than when the apparatus was not used. On reboiling the worts, the former yielded 0.81 g., the latter 0.93 g., of further coagulam per kg. of extract. The difference appears to be mainly due to increased precipitation of calcium phosphate, but the reaction and buffering of the worts during boiling and fermentation were not appreciably affected. The chief effect on the primary fermentation was that the yeast deposited during the first four days was coated with a brownish slime, and

had to be washed in an apparatus which removed this by mechanical attrition before a good press-cake was obtainable. The yeast which separated during the secondary fermentation was also darker than usual. The beers finished paler, had a fuller, cleaner flavour, and were in no way inferior to those boiled without the apparatus. It was found necessary to use 2–3% more hops, and to add these after the wort had boiled for 30 min. No comparisons have been made with wort boiled in fire-heated coppers, but it is suggested that the possibly well-founded objection of many brewers to steam-heated coppers could be overcome by designing them to permit of more vigorous boiling. F. E. DAY.

Adjustment of acidity of cane-molasses fermentations for maximum alcohol yields. F. M. HILDEBRANDT (Ind. Eng. Chem., 1929, 21, 779–781).—Maximum yields are obtained when the initial acidity is such that during fermentation the p_H shows no shift towards the acid side. This is generally, but not invariably, at about p_H 5, and may be attained by suitable addition of sulphuric acid. F. E. DAY.

Colorimetric determination of acidity in wort and beer. P. KOLBACH (Woch. Brau, 1929, 46, 305–308).—The usual colour-matching difficulties due to change of turbidity and colour with change of reaction and colloid and salt effects are eliminated by using a standard buffer solution which has the desired reaction when diluted to three times its volume, and diluting 10 c.c. of this with 20 c.c. of the wort or beer. To another 20 c.c. of wort or beer 10 c.c. of water are added, then a suitable indicator. The two mixtures are examined in a colour comparator, and standard alkali or acid is added to the unbuffered liquid until the indicator colours match. To the buffered portion an equal volume of alkali or acid is added, plus 10% to allow approximately for the extra amount which the wort or beer will require to obtain a match to the slightly altered tint. The wort is again adjusted by the further addition of reagent, and to a fresh buffer-wort-indicator mixture a volume equal to that used by the wort titration is added. The buffer can now be considered as being diluted with wort or beer of its own reaction, i.e., its p_H is unaltered, and a fresh portion of 20 c.c. of wort with indicator and 10 c.c. of water is titrated to a colour match. For example, in the titration of the alkalinity of a wort to p_H 4.27, 10 c.c. of an acetate buffer solution, of that reaction when three times diluted, with 20 c.c. of wort and 2 c.c. of 0.15% chlorobromophenol-blue was compared against 20 c.c. of wort with 10 c.c. of water and 2 c.c. of indicator solution. The latter required 1.10 c.c. of 0.1N-hydrochloric acid, hence 1.21 c.c. of standard acid were added to the buffer mixture; the wort then required a further 0.13 c.c. of acid. To a fresh buffer mixture 1.23 c.c. of 0.1N-acid were added, and a fresh portion of 20 c.c. of wort required 1.22 c.c. of acid, equivalent to 6.10 c.c. per 100 c.c. of wort. The method is particularly applicable to the titration of worts to p_H 4.27, since the salt error due to the buffer compensates the dilution error of the wort. In the case of beers, the error introduced by dilution is less, and, if allowance is made for the salt error, the method is accurate to 0.05 p_H unit. Details

of the titration of wort acidity have yet to be worked out. F. E. DAY.

Clarification of wine by potassium ferrocyanide. E. COUERBE (Ann. Falsif., 1929, 22, 354–362).—It is claimed that potassium ferrocyanide can safely be used in the clarification of wine (removal of iron), there being no danger of formation of hydrocyanic acid if the correct quantity is employed. Excess of the ferrocyanide is readily detectable by taste, and produces blue precipitation and colour alteration in the wine after filtration. It is recommended that this process should be legalised in France, as it is in Germany, in place of the present method of treating with oxygen and then with tannin. E. B. HUGHES.

Raisin wine. L. CHAUVEAU and A. VASSEUR (Ann. Falsif., 1929, 22, 340–351).—The authors prefer the term “raisin piquette” rather than “raisin wine.” The chemical characteristics of raisin wine are discussed, and it is shown that for carefully-prepared raisin wines these may not differ, in general, from those of ordinary wine. The main distinctions to be relied on are odour and taste of the wine and its distillate, and the luminescence in Wood's light. E. B. HUGHES.

Determination of the p_H of wines. A. QUARTAROLI (Annali Chim. Appl., 1929, 19, 253–254).—A question of precedence. T. II. POPE.

Detection of ethyl phthalate in spirits. H. SZANCER (Pharm. Zentr., 1929, 70, 502–503).—A modification of the method of Utz (B., 1924, 687) is described. A few drops of the brown reaction mixture (sulphuric acid, resorcinol, and sample) are mixed with about 500 c.c. of water and then a few drops of ammonia are added. The fluorescence, if any, is very distinct, and, by comparison with the colour of the lower part of the liquid, easily recognised. In a similar way, if phenol instead of resorcinol and potassium hydroxide instead of ammonia be used, the violet-red colour due to the formation of phenolphthalein is easily distinguished. E. II. SHARPLES.

Determination of sulphurous acid in apple juice and cider. WARCOLLIER and LE MOAL (Ann. Falsif., 1929, 22, 333–340).—The methods of Haas and of Ripper are discussed. The former gives correct results. The latter gives high results with high percentages of sulphurous acid, and low results with low percentage. Ripper's method will, however, give satisfactory results, for rapid work, if the liquid after addition of caustic potash is allowed to remain alkaline for only 5 min. instead of 15 min. Caustic potash oxidises sulphur dioxide in presence of the tannins of the juice or cider. This modified method should apply equally well to wine. E. B. HUGHES.

See also A., Aug., 956, **Preparation of diastase** (WINKLER and KÖCK). 957, **Proteolytic enzymes in green malt** (MILL and LINDERSTRÖM-LANG). **Alcoholic fermentation of amino-acids** (PARISI and others). 959, **Vitamin-B from brewer's yeast** (SEIDELL).

PATENTS.

[**Butyl alcohol-acetone**] **fermentation.** E. R. WEXER (U.S.P. 1,696,022, 18.12.28. Appl., 4.3.27).—

Higher yields are obtained in the fermentation of carbohydrate mash by treating the mash during or after sterilisation with an antiseptic, such as resorcinol butyl or phenyl butyl ether, which is non-toxic towards the butyl alcohol-acetone-producing cultures but toxic to the contaminating organisms. R. BRIGHTMAN.

Manufacture of yeast. E. KLEIN, Assr. to FLEISCHMANN Co. (U.S.P. 1,722,803, 30.7.29. Appl., 17.8.23. Austr., 29.8.22).—See B.P. 205,813; B., 1924, 273.

XIX.—FOODS.

Composition of corn (*Zea mays*) seedlings.
I. Isolation of xylan and cellulose from cell walls. **II. Isolation of a dextrin similar to the trihexosan obtained by thermal depolymerisation of potato starch.** K. P. LINK (J. Amer. Chem. Soc., 1929, 51, 2506—2516, 2516—2522).—I. The seedlings are produced at 12° and 24° and the sprouts (radicle and plumule) killed with alcohol. Extraction of the finely-divided tissue with 90 and 99% alcohol and light petroleum (removal of fats etc.), subsequent digestion with 1% ammonia, and repeated treatment with chlorine dioxide and sodium sulphite (removal of proteins, pectin, lignin, etc.) affords an amorphous powder containing 0.33% of ash and 0.67% N. Extraction of this powder with 5% sodium hydroxide solution and treatment of the extract with alcohol precipitates xylan-A (8.5—9% of dry tissue), purified by reprecipitation from ammoniacal copper hydroxide solution. This is soluble in hot water, giving when cooled a thin gel, which swells when treated with alcohol. Hydrolysis first with 72% sulphuric acid at 15° and then with boiling 2% acid (by dilution) affords xylose. The residue after removal of xylan-A is extracted with 10% sodium hydroxide solution at 60°, whereby xylan-B is obtained (3—4% of dry tissue). The residue from this after extraction with 15% sodium hydroxide solution at 30° (to remove substances, not true pentosans, which give furfuraldehyde when distilled with 12% hydrochloric acid) consists of cellulose (14—18% of dry seedlings). This resembles cotton cellulose; it gives a triacetate, $[\alpha]_D^{25} -22.1^\circ$ in chloroform, simultaneous acetylation and hydrolysis affords 46—48% of α -cellobiose octa-acetate, and dextrose is the only recognisable sugar produced by hydrolysis with sulphuric acid. There is a quantitative difference in the distribution of the xylans in the seedlings grown at 12° and at 24°, xylan-A predominating at the lower temperature. The total amounts of xylans are approximately the same in each case.

II. The sugar- and fat-free, dried tissue is extracted with 20% alcohol. The alcohol content of the extract is increased to 40%, when a dextrin (colour with iodine) is obtained. At a subsequent alcohol concentration of 85% a second dextrin (no colour with iodine), decomp. 220—230° without melting, $[\alpha]_D^{25} +163.6^\circ$ in water, is isolated. This is similar to the trihexosan described by Pictet and Jahn (A., 1922, i, 987), yields a nona-acetyl derivative, m.p. 152—153°, $[\alpha]_D^{25} +125.9^\circ$ in chloroform, and when hydrolysed with 2.5% sulphuric acid affords 92% of dextrose. Hydrolysis of its monomethyl derivative with 5% hydrochloric acid gives 6-methylglucose. The mol. wt. of the dextrin varies from 510 to 639 in water. H. BURTON.

Colour of wheat flour. A. C. HARDY, P. I. COLE, and C. W. RICKER, JUN. (Ind. Eng. Chem. [Anal.], 1929, 1, 151—152).—Spectrophotometric observations have been made on a number of grades of bleached and unbleached flour and their reflecting powers, referred to magnesium carbonate as standard, are represented graphically in an attempt to correlate colour with other known properties. Bleaching was found to raise the reflecting power in the blue end of the spectrum. To simplify the procedure measurements were made of brilliance at 556 m μ in the green, and of yellowness at 440 m μ in the blue region of the spectrum. It was observed that brilliance tends to decrease, and yellowness to increase, with increase of ash content. H. J. DOWDEN.

Effects of wheat drying on milling and baking properties. R. C. SHERWOOD (Bull. State Dept. Agric., Minnesota, No. 66, 1929, 35 pp.).—Comparison is made of direct and indirect methods for wheat drying, in which the air current is heated by means of coke furnaces and by steam radiators, respectively. In neither process was the milling or baking value of the wheat affected by the drying. Processes involving a continuous flow of grain are preferable to drying in batches since temperature control is more certain. In the indirect heating process the utilisation of air from the cooling section did not damage the wheat. The use of coke containing 0.56% S in the direct process did not lead to the absorption of appreciable amounts of sulphur dioxide by the grain. A. G. POLLARD.

Application of the bromate differential test in the estimation of baking quality of Canadian hard red spring-wheat flour. R. K. LARMOUR and A. G. MACLEOD (Sci. Agric., 1929, 9, 477—490).—The addition of potassium bromate (0.5 mg. per 100-g. loaf), to afford stimulation approximately proportional to the protein content, is preferred to the "basic standard baking test." Results with wheat containing 11.2—17.4% of crude protein are recorded.

CHEMICAL ABSTRACTS.

Effect of [feeding with] lupin-fish meal on the quality of milk and butter fat. O. MROZEK, H. SCHLAG, and A. EICHSTÄDT (Milchwirt. Forsch., 1929, 7, 495—501; Chem. Zentr., 1929, i, 1162).—The proportion of large fat particles is reduced. The iodine value of the fat tends to rise and the Polenske value to fall, whilst the saponification and Reichert-Meissl values remain unchanged. A. A. ELDRIDGE.

Adulteration of butter and ghee with animal fat and vegetable ghee, and its detection. P. SANYAL (Mem. Dept. Agric. India, 1929, 10, 143—155).—One gram of the melted filtered fat is dissolved in 3 c.c. of dry ethyl acetate, warmed to 30°, 4 c.c. of 93% alcohol at 30° are added, with mixing, and the liquid is kept at 30° for 30 min. With genuine butter and ghee no precipitate forms, but if foreign fat is present (5% upwards of animal fat, and 12% upwards of vegetable fat) a precipitate proportional to the amount of adulterant (up to 25%) separates. Butter or ghee from buffaloes fed on cotton-seed cakes gives a precipitate, but none in a similar test using 4 c.c. of ethyl acetate and 3 c.c. of 93% alcohol, though with reagents in

these proportions the test is less sensitive, indicating not less than 12% of foreign animal fat.

E. B. HUGHES.

Blue milk. W. STOCKER (Milchwirt. Forsch., 1929, 7, 332—339; Chem. Zentr., 1929, i, 1161).—Conditions leading to the production of blue milk (due to the presence of Beijerinck's *Acinomyces*) are described.

A. A. ELDRIDGE.

Determination of f.p. of milk. J. KRENN (Milchwirt. Forsch., 1929, 7, 436—445; Chem. Zentr., 1929, i, 1163—1164).—Of 188 samples tested, none had $\Delta \times 10^2$ less than 51.5. For milk of individual cows, those having $\Delta \times 10^2$ above 53 are genuine, 53.0—50.0 probably, and under 50 certainly diluted.

A. A. ELDRIDGE.

Optical detection of watering of milk. E. MUNDINGER (Milchwirt. Forsch., 1929, 7, 280—291; Chem. Zentr., 1929, i, 1163).—The refractivity of a drop of milk, determined by Löwe's method, bears a fairly constant relation to the total solids. Shaking and small variations in acidity affect the result but slightly.

A. A. ELDRIDGE.

Sensitiveness of the "thybromol" test in comparison with other methods for detecting pathological changes in milk. G. ROEDER (Milchwirt. Forsch., 1929, 7, 365—435; Chem. Zentr., 1929, i, 1164).—The "thybromol" test is preferred.

A. A. ELDRIDGE.

Water content of cheese. K. TEICHERT and H. SCHLAG (Milchwirt. Forsch., 1929, 7, 259—270; Chem. Zentr., 1929, i, 1162).—Average values for cheese containing 40—50 (or 20—30)% of fat are: Emmentaler 35, Dutch 43 (50), Tilsit 43 (52), Limburg 50 (60), Camembert 55 (62). In young cheese higher values are permissible.

A. A. ELDRIDGE.

Changes produced in eggs by micro-organisms. G. L. PAVARINO (Annali Chim. Appl., 1929, 19, 266—272).—Altered eggs from store showed spots due to moulds on the interior of the shell, and the yolk was sometimes liquefied and the white turbid and watery and often coloured superficially by a green fluorescent pigment. These changes were due mainly to *B. fluorescens-liquefaciens*, *Micrococcus roseus*, and *Staphylococcus aureus*.

T. H. POPE.

Viscosity of egg albumin and the changes it undergoes in fresh and preserved eggs. D. CORTESE (Annali Chim. Appl., 1929, 19, 260—265).—In eggs kept at the ordinary temperature, the viscosity of the albumin diminishes rapidly and fairly regularly, in spite of the evaporation of water which occurs. At lower temperatures, fluidification also proceeds regularly but is far more slow. In accord with the known dissolving and liquefying action of bacteria, the diminution in viscosity is much more rapid in altered eggs.

T. H. POPE.

Influence of p_H of the white on keeping quality of hen eggs. P. F. SHARP (Science, 1929, 69, 278—280).—Control of the p_H value is essential in the storage of eggs. Freshly-laid eggs containing few bacteria keep well if the p_H value is maintained at about 7.6, the normal figure; a rise in the p_H value favours the factors which make for deterioration. The loss of carbon

dioxide by stored eggs causes the p_H to rise to about 9.7 and the white to become fluid; the addition of small percentages of carbon dioxide to the storage atmosphere hinders this change. L. S. THEOBALD.

Effect of certain salts on the physical properties of ice cream mixes. J. C. HENING and A. C. DAHLBERG (J. Dairy Sci., 1929, 12, 129—139).—Addition of sodium citrate, potassium oxalate, or sodium hydrogen phosphate to ice cream before pasteurisation and homogenisation reduces the viscosity, diminishes the size of the fat-globule clumps, and causes easier whipping. The size of the clumps is probably related to the viscosity. Calcium lactate increased both, causing more difficult whipping; lactic or citric acid had a similar effect. Difficult whipping can be corrected by addition of alkali. The relative amounts of sodium and calcium salts in ice cream made from normal dairy products probably varies sufficiently to affect the whipping properties.

CHEMICAL ABSTRACTS.

Metals in dairy equipment; metallic corrosion in milk products and its effect on flavour. O. F. HUNZIKER, W. A. CORDES, and B. H. NISSEN (J. Dairy Sci., 1929, 12, 140—181).—A study of the effect of various metals, coated metals, and alloys on dilute solutions of acids and on milk products.

CHEMICAL ABSTRACTS.

Production of caffeine-free coffee. F. IHLOW (Chem.-Ztg., 1929, 65, 629—630).—Plant for the purpose is described.

See also A., Aug., 947, Irradiated proteins (SPIEGEL-ADOLF and KRUMPEL). 953, Inorganic constituents of milk (WRIGHT and PAPISH). 959, Antineuritic and water-soluble B-vitamins in beef and pork (HOAGLAND). 960, Effect of drying and of sulphur dioxide on antiscorbutic property of fruits (MORGAN and FIELD). 962, Determination of chlorine in foods (BIRNER).

Spray drying of dairy products. NYROP.—See I. Spontaneous decomposition of butter fat. MUNDINGER.—See XII. Yeasts for bread-making. E. and L. ELION. Sulphurous acid in apple juice. WARCOLLIER and LE MOAL.—See XVIII.

PATENTS.

Apparatus for manufacture of margarine. H. BORGES and G. W. WADSWORTH (B.P. 316,481, 25.10.28).

Food product [in cheese envelope]. E. GALBANI (B.P. 316,850, 22.1.29).

Method and apparatus for use in preparation of malted foodstuffs. J. SLEEMAN (B.P. 315,329, 10.1.28).

Food products [from honey]. O. A. SIPPEL (B.P. 312,036, 30.10.28. U.S., 18.5.28).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of formaldehyde in a pharmaceutical preparation. O. HEIM (Ind. Eng. Chem. [Anal.], 1929, 1, 128).—To a 10-c.c. sample (about 0.2% of formaldehyde) 2 c.c. of hydrochloric acid and 10 c.c. of *N*-silver nitrate are added, and, after shaking, 4 c.c. of 50% sodium hydroxide. After 15—20 min.

with occasional shaking the blackened precipitate is washed and the reduced silver is dissolved in nitric acid and determined in the usual way as silver chloride ($2\text{AgCl} = 1\text{CH}_2\text{O}$).

C. HOLLINS.

Acids of tobacco. I. A. SCHNUCK (U.S.S.R. State Inst. Tobacco Invest., 1929, Bull. 50, 10 pp.).—By extracting fermented Trebizond tobacco with dilute hydrochloric acid, and extracting the concentrated filtrate with ether, a solution was obtained containing malic, succinic, fumaric, acetic, and formic acids, together with other acids not yet identified.

T. H. POPE.

Acids of tobacco as a qualitative indication of its value. M. PIATINSKI (U.S.S.R. State Inst. Tobacco Invest., 1929, Bull. 51, 11 pp.).—The proportion of total acids present in tobacco, determined by extracting the acidified tobacco with ether and titrating the extracted matter with standard sodium hydroxide solution, is lower in good than in poor tobaccos.

T. H. POPE.

Determination of nitrate-nitrogen in tobacco. H. B. VICKERY and G. W. PUCHER (Ind. Eng. Chem. [Anal.], 1929, 1, 121–123).—To avoid large blank determinations, nicotine and pre-existing ammonia are removed by steam distillation of a suspension of the tobacco product in alkaline solution. Two 5-g. samples of the material with 30 c.c. of water and 5 c.c. of 50% sodium hydroxide solution are steam-distilled into 0.1N-hydrochloric acid until 800 c.c. have been collected. Titration of the distillate gives total volatile bases, nicotine being determined subsequently by the silicotungstic acid method. Nitrate-nitrogen is determined in one of the residues by reduction with sulphuric acid and iron powder (cf. Jones, B., 1927, 262). The second residue is used for a blank determination, the difference in the titration values of the two residues being the nitrate-nitrogen in the original sample. Wide variations in nicotine and nitrate-nitrogen were observed.

H. J. DOWDEN.

Determination of ammonia and amide-nitrogen in tobacco. H. B. VICKERY and G. W. PUCHER (J. Biol. Chem., 1929, 83, 1–10).—Ammonia can be determined in presence of other volatile bases by absorption with permittit followed by liberation from the latter with alkali and determination by Nessler's reagent. Extract of tobacco is distilled over magnesium oxide, and the ammonia in the distillate determined by the above method; similar treatment of another sample of extract after hydrolysis with hydrochloric acid gives the sum of the ammonia- and amide-nitrogen.

C. R. HARRINGTON.

Nicotine content of fresh tobacco leaves. S. NISHIYAMA (Bul. Sci. Fak. Terkultura, 1929, 3, 10–15).—The nicotine content increases from the lower to the higher leaves.

CHEMICAL ABSTRACTS.

Determination of nicotine in tobacco. M. SAMEJIMA and K. KATAI (Bul. Sci. Fak. Terkultura, 1929, 3, 112–116).—Kissling's, Chapin's, and Okuda's methods give similar results. Toth's method gives high, and Fodor and Reifenberg's method low, results.

CHEMICAL ABSTRACTS.

Determination of morphine in opium, opium extract, and tinctures. J. BOEHM (Apoth.-Ztg., 1929,

44, 88–91; Chem. Zentr., 1929, i, 1244).—The official (D.A.B. VI) method gives high and variable results, whilst Hairs' method gives concordant, although somewhat low, results. A new procedure is described.

A. A. ELDRIDGE.

An impurity in commercial narceine, which gives the colour reaction with sodium nitroprusside. J. J. L. ZWIKKER (Pharm. Weekblad, 1929, 66, 445–449).—The earlier observation (cf. A., 1929, 832) that narceine gives a coloration with nitroprusside is found to be erroneous; the colour is due to the presence of a small quantity of methylnarceine, formed in methylation during the synthesis from narcotine. Pure narceine gives no colour, but the presence of 1 mg. of the methyl derivative in 50 mg. of the alkaloid is enough to give a positive reaction. The m.p. of the hydrochloride of the methyl compound is 234° , and not 243° , as given by Tambach and Jäger (cf. A., 1906, i, 879). S. I. LEVY.

Preparation of emodin. C. ROULIER and R. DUBREUIL (Bull. Soc. Pharm. Bordeaux, 1928, 66, 145–152; Chem. Zentr., 1929, i, 888).—The bark of *Rhamnus frangula* is heated for $\frac{1}{2}$ hr. at 75° with five times its weight of 97% alcohol and 1 g. of hydrogen chloride per 5 litres of liquid. After cooling and separation by pressure the liquid is evaporated, the residue dried in a vacuum at the ordinary temperature, and the powdered material is treated in a mortar with 5% ammonia solution until a red coloration no longer appears. The solution is precipitated in presence of ether with excess of hydrochloric acid, and exhaustively extracted with several litres of ether. The ether is evaporated; the residue is hardened by cooling or keeping, dissolved in the minimal quantity of 5% ammonia solution, filtered, precipitated with hydrochloric acid, centrifuged, and washed twice with distilled water, these operations being repeated, and the precipitate dried over sulphuric acid for several days. It is then redissolved in ether and extracted with 5% ammonia solution; the ether is completely removed and the hot liquid precipitated dropwise with hydrochloric acid. The mixture is cooled slowly, centrifuged, decanted, and the precipitate washed with distilled water and dried over sulphuric acid, being finally extracted 3–4 times with 97% alcohol, which is then slowly evaporated.

A. A. ELDRIDGE.

Commercial lecithins and specialities with lecithin as base. I. J. SONOL (Rev. fac. cienc. quim. Univ. La Plata, 1927, 4, No. 2, 95–110).—The purest commercial specimens of lecithin contain cholesterol and other substances. The following method of preparation is preferred: The tissue is dried over acetone, and extracted for 12–24 hrs. three times at 50° with absolute ethyl alcohol, which is incompletely removed in a vacuum. The yellow liquid is decanted from the lower red liquid, concentrated in a vacuum, and treated with a little ether. The lecithin is precipitated three times with acetone and dried to remove acetone.

CHEMICAL ABSTRACTS.

Assay of belladonna. J. NOLLE (Arch. exp. Path. Pharm., 1929, 143, 184–191).—A unit is defined as that amount of a preparation which after 10 minutes' action renders the frog's gastrocnemius insensible to acetylcholine (1/100,000). The contents of a number

of commercial extracts expressed in physiological units are tabulated and show marked deviation from the alkaloid content as determined chemically.

P. W. CLUTTERBUCK.

Water content of essential oils and turpentine. H. NICOL (Compt. rend., 1929, 189, 289—292).—The absorption of radiant heat by the vapours of essential oils, observed by Tyndall, is believed to be due chiefly to the water which they contain, and attention is drawn to the fact that these oils are distilled in the presence of water. A simple conductivity method for measuring the relative water content has been devised; the results are in agreement with those obtained by a qualitative electrostatic method and by the turbidity produced when they are mixed with turpentine. Oils from xerophilous plants and from peels of citrus fruits are anhydrous and even hydrofuge, whilst those from the *Cymbopogon* may have a relatively high water content. From these facts conclusions concerning the botanical history of the oils have been drawn. Of all the products of plant physiology examined, only turpentine gives a turbidity with every oil containing water.

A. A. GOLDBERG.

Oil of *Asarum Europeanum*. S. GERÖ (Riechstoffind., 1928, 3, 176—177, 195—196, 214—216, 232; Chem. Zentr., 1929, i, 946—947).—The composition of the essential oil of the root of *Asarum Europeanum* depends on soil and climatic conditions. The oil contains asaraldehyde, $C_{10}H_{12}O_4$, m.p. 114° (oxime, m.p. 138°), asarone (dibromide, m.p. 86°), diasarone ($C_{12}H_{16}O_3$)₂ (oxidised to asaronic acid, m.p. 144°), an etheral substance, $C_{11}H_{18}O$, a sesquiterpene, and a sesquiterpene alcohol.

A. A. ELDRIDGE.

Hungarian essential oils. M. JANICSEK (Riechstoffind., 1928, 3, 211—213; Chem. Zentr., 1929, i, 946).—The following data are recorded for the corresponding essential oils: *Thymus vulgaris*: d^{15}_D 0.9051, α_D $-2^\circ 80'$, n^{20}_D 1.4922; *Thymus serpyllum*: d^{15}_D 0.8944, α_D -10.93° , n_D 1.4859; *Salvia officinalis*: d^{15}_D 0.9195, α_D $+13^\circ 32'$, n^{20}_D 1.4631 (German seed), and d^{15}_D 0.9165, α_D -1.60° , n^{20}_D 1.4623 (French seed); *Mentha crispata*: d^{15}_D 1.4793; *Chenopodium ambrosioides* var. *anthelminticum*: d^{15}_D 0.9852, α_D $-8^\circ 39'$, n^{20}_D 1.4760; *Lavandula vera*: d^{15}_D 0.8885, α_D $-7^\circ 37'$, n^{20}_D 1.4632; *Juniperus communis*: d^{15}_D 0.8686, α_D $-5^\circ 61'$, n^{20}_D 1.4802, and data for rectified and residual oil; *Mentha piperita*: data for various fractions.

A. A. ELDRIDGE.

Hungarian essential oils. M. FÖLSCH (Riechstoffind., 1928, 3, 181, 197—198, 217—218, 233—234; Chem. Zentr., 1929, i, 945—946).—The following data are recorded for the corresponding essential oils: peppermint: d^{15}_D 0.901—0.905, α_D -26.38° , n^{20}_D 1.4605, total menthol 59.6%; *Tanacetum vulgare*, L.: d^{15}_D 0.926; *Salvia officinalis*, L.: d^{15}_D 0.926; *Salvia sclarea*, L.: d^{15}_D 0.918, α_D $+4^\circ$, n^{20}_D 1.4603, acid value 1.25, ester value 21.2 (after acetylation 69); *Achillea millefolium*: d^{15}_D 0.935; *Thymus vulgaris*: d^{15}_D 0.916; *Juniperus communis*, L.: d^{15}_D 0.876, α_D $-7^\circ 12'$, n^{20}_D 1.4793; wine lees oil: d^{15}_D 0.8771, α_D $+0.5^\circ$, n^{20}_D 1.4296, acid value 54, ester value 208; *Artemisia absinthium*: d^{15}_D 0.937.

A. A. ELDRIDGE.

See also A., Aug., 916, Synthesis of ephedrine and

its homologues (MANSKE and JOHNSON). 932, Cedrene (RUZICKA and VAN MELSEN). 933, Rotenic and tubaic acids from *Derris* root (TAKEI and others). 934 Tea catechin from green tea (TSUJIMURA). 939, Copper compounds of diethylbarbituric acid (ROMANOWA). 943, 4-*mp*-Dihydroxyphenylthiazoles (JOHNSON and GATEWOOD). 944, Nicotine tetrachloriodide (CHATTAWAY and PARKES). Calycanthine from *Meratia praecox* (MANSKE). *Strychnos* alkaloids (LEUCHS and HOFFMANN). Disinomenine and ψ -disinomenine (GOTO and SUDZUKI). 948, Microdetermination of sulphur, phosphorus, and arsenic in organic compounds (HELLER). 949, Determination of cocaine in physiological material (SADOLIN). 959, Standardisation of thyroid preparations (MORCH). Vitamin-B from brewer's yeast (SEIDELL). Antineuritic and water-soluble B vitamins in beef and pork (HOAGLAND). 961, *Phytolacca* (JENKINS). Lobeline-like substance from roots of *Lobelia sessilifolia* (KUBOTA and others). *isoSakuranetin* (HATTORI).

PATENTS.

Antiseptic anaesthetic salts. E. H. VOLWILER and D. L. TABERN, ASSRS. to ABBOTT LABS. (U.S.P. 1,708,712, 9.4.29. Appl., 25.7.27).—Compounds of *n*-butyl *p*-aminobenzoate and a halogenated phenolsulphonic acid have both antiseptic and anaesthetic action. The *n*-butyl *p*-aminobenzoates of 2:6-di-iodo-, m.p. 200° (decomp.), and 2:6-dibromo-phenol-4-sulphonic acid, m.p. 216 — 218° , and of 7-iodo-8-hydroxyquinoline-5-sulphonic acid, m.p. 235 — 240° , are described. R. BRIGHTMAN.

Chloro-*n*-amylmalonamide. A. W. DOX, ASSR. to PARKE, DAVIS, & CO. (U.S.P. 1,713,822, 21.5.29. Appl., 28.5.28).—Ethyl *n*-amylmalonate, b.p. 134 — $136^\circ/14$ mm., is converted into *n*-amylmalonamide, m.p. 206° , which, when chlorinated in acetic acid, yields chloro-*n*-amylmalonamide, $Me \cdot [CH_2]_4 \cdot CCl(CO \cdot NH_2)_2$, m.p. 134 — 135° , claimed as a sweetening agent. R. BRIGHTMAN.

Production of camphene from pinene hydrochloride. H. GAMMAY (U.S.P. 1,721,990, 23.7.29. Appl., 28.1.26).—See B.P. 263,311; B., 1927, 156.

Production of menthol. H. JORDAN, W. SCHOELLER, and R. CLERC, ASSRS. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,706,784, 26.3.29. Appl., 15.8.27. Ger., 22.2.27).—See B.P. 285,833; B., 1929, 699.

Increasing the yield in civetone, starting from civet. L. RUZICKA, ASSR. to M. NAEF & CO. (U.S.P. 1,720,748, 16.7.29. Appl., 25.1.27. Switz., 17.3.26).—See B.P. 267,893; B., 1927, 892.

[Recessed] surgical dressings. A. E. M. HUNN and C. R. GRAY (B.P. 316,112, 23.3., 13.6., and 11.7.28).

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Theory of hypersensitisation. K. JACOBSON (Z. wiss. Phot., 1929, 27, 19—23).—The hypersensitisation treatment previously described (B., 1928, 211) has a much greater effect on emulsions prepared by the heating method than on those prepared with ammonia. This is probably largely due to ammonia being necessary

for the formation of silver sulphide nuclei by the allylthiocarbimide in the gelatin (cf. Sheppard and Hudson, B., 1927, 765).

R. CUTHILL.

Distribution of silver iodide in silver bromide. LÜPPO-CRAMER (Z. wiss. Phot., 1929, 27, 9—19).—An ammoniacal silver bromide-iodide emulsion in the preparation of which a considerable excess of alkali bromide has been used shows a pronounced tendency to solarisation and the nuclei are remarkably sensitive to the action of chromic acid. These effects do not appear, however, if no iodide is present. In presence of iodide the grains are more or less of a uniform size, whereas in absence of iodide some are smaller and others are larger. Nevertheless there does not seem to be any connexion between variability in size of grain on the one hand, and liability to solarisation and sensitivity to denucleation on the other. It is suggested that the iodide acts by forming a surface layer on the bromide grains, for iodide is known to solarise particularly readily, and acid would displace adsorbed iodide from the surface of the ripening nuclei more easily than adsorbed bromide. Addition of the iodide as silver iodide to the bromide emulsion before mixing has the same effect as adding it as alkali iodide, which would agree with the observations of Baldisiefen, Sease, and Renwick (B., 1926, 466) on the peptisation and inclusion of silver iodide by freshly-formed silver bromide. Addition of silver iodide after the bromide emulsion has been mixed, however, has no effect of any kind.

R. CUTHILL.

See also A., Aug., 871, **Photodichroism and photoanisotropy** (WEIGERT). 893, **Nature of sensitivity and latent image** (GERMANN and SHEN). 894, **Photochemistry of silver halides** (SCHMIDT and PRETSCHNER). **Primary process in formation of latent image** (SHEPPARD). **Measurement of induced photodichroism** (WEIGERT). 902, **Magnesium light for photographic sensitometry** (EDER).

PATENTS.

Combined [photographic] desensitiser and developer. H. MEYER, Assr. to AGFA ANSCO CORP. (U.S.P. 1,713,613, 21.5.29. Appl., 8.9.27. Ger., 13.11.26).—Cyanides of heavy metals are claimed as desensitisers; e.g., 0.3 g. of mercuric cyanide is added to 1 litre of a metol-quinol developer.

R. BRIGHTMAN.

Photographic silver halide emulsion. O. MATTHIES and W. DIETERLE, Assrs. to AGFA ANSCO CORP. (U.S.P. 1,719,711, 2.9.29. Appl., 6.8.26. Ger., 14.9.25).—See B.P. 258,237; B., 1927, 861.

Film for lenticular-screen colour photography. R. BERTHON, Assr. to SOC. CIVILE POUR L'ÉTUDE DE LA PHOTOGRAPHIE ET DE LA CINÉMATOGRAPHIE AU COULEURS (U.S.P. 1,721,244, 16.7.29. Appl., 17.6.29. Fr., 20.7.26).—See B.P. 274,837; B., 1928, 286.

XXII.—EXPLOSIVES; MATCHES.

See also A., Aug., 940, **New group of hydrazomethylenes** (BUSCH and others).

PATENTS.

Manufacture of nitrocellulose smokeless powder. A. S. HAWKESWORTH (U.S.P. 1,713,505, 14.5.29. Appl.,

11.6.28).—Nitrocellulose is simultaneously dried, dissolved, and stabilised by means of mononitrotoluene.

H. ROYAL-DAWSON.

Manufacture and packing of matches. A. OVTSCHINNIKOFF (B.P. 316,592, 27.1.28).

XXIII.—SANITATION; WATER PURIFICATION.

Sodium peroxide respirators. G. STAMPE and E. HORN (Z. angew. Chem., 1929, 42, 776—779).—In 12 experiments the period of service of a sodium peroxide respirator averaged 61 min., with a mean deviation of 3.5 min. The addition of 0.1% of various "catalysts" (manganese dioxide, copper oxide, etc.), in general, raised the period of service, but produced greater fluctuations from the mean value in series of repeat experiments. The period of service can be somewhat lengthened by the introduction of a drying agent (calcium chloride) into the lower part of the respirator. The activity of the sodium peroxide falls off on keeping. The material used, "pyroxylit," had a water content of 6%, which appears to be about the optimum for use in respirators.

A. B. MANNING.

Biochemical oxygen demand of certain substances. G. E. SYMONS and A. M. BUSWELL (Ind. Eng. Chem. [Anal.], 1929, 1, 161—162).—An attempt has been made to establish a relationship between the biochemical oxygen demand and the theoretical oxygen demand of lactose, starch, cellulose, sodium palmitate, peptone, and urea. The demands of a filtered sewage as control and of the control plus the pure substance have been studied over a period of 30 days, the difference being the oxygen demand of the pure substance. Difficulty was experienced at the 15th day, when nitrification began in the control, but the procedure was again applicable when nitrification also set in in the other series. Urea undergoes nitrification from the start and does not present this difficulty. It was found that with carbonaceous substances the oxidation follows an equation of the first order, but the biochemical oxygen demand is only 70—85% of the theoretical demand. Urea shows only second-stage oxidation, and the biochemical oxygen demand is equal to the calculated demand.

H. J. DOWDEN.

See also A., Aug., 904, **Trinidad well waters** (PARKER and SOUTHWELL).

Insecticides. HART.—See XVI.

PATENTS.

Removal of dissolved silicic acids from liquids, especially water for domestic or industrial uses. A. ROSENHEIM (B.P. 291,435, 1.5.28. Ger., 4.6.27).—The water is filtered through beds comprising dried gels capable of forming insoluble or only slightly soluble silicic acid adsorption compounds, polysilicic acid compounds, or double silicates, e.g., oxides or hydroxides of iron, chromium, or the elements of group III, or mixtures of these. The process can be combined with softening by using gels containing alkalis. The gels are regenerated by treatment with alkalis etc.

L. A. COLES.

Apparatus for treating refuse. F. C. EVANS (B.P. 315,458, 13.4.28).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCT. 11, 1929.

I.—GENERAL; PLANT; MACHINERY.

Use of carbon tetrachloride in fire extinguishers.

F. WIRTH (Chem.-Ztg., 1929, 53, 651—652).—Experiments are described in which artificial fires were produced by burning oil, benzene, and acetylene, and were extinguished with carbon tetrachloride. A series of gas tests for phosgene was made. Positive reactions were obtained in nearly all instances, the principal factor in phosgene production being time of contact. It is considered that the use of carbon tetrachloride is dangerous in confined spaces or when a fire has got a firm hold and cannot be immediately extinguished. The danger can, of course, be obviated by wearing a gas mask. No substance has yet been found the addition of which to the carbon tetrachloride will prevent phosgene formation.

C. IRWIN.

Clarification of liquids by means of centrifuges with filter drums. A. SCHMÜCKING and K. DIEHL (Chem. Fabr., 1929, 287—288, 298—300).—An illustrated description of a new Krupp clarifying centrifuge fitted with a filter drum and automatic delivery and overflow is given.

A. R. POWELL.

Fractional distillation analysis. W. J. PODBIELNIAK (Refiner Nat. Gas. Mfr., 1929, 8, No. 3, 55).—A simplified form of Leslie's apparatus for gas analysis by liquefaction and fractional distillation is described.

CHEMICAL ABSTRACTS.

Bimetals. ROHN.—See X. **Boiler corrosion.** SPLITTGERBER.—See XXIII.

PATENTS.

Furnace. L. DE FLOREZ, Assr. to TEXAS CO. (U.S.P. 1,717,334, 11.6.29. Appl., 2.7.26).—A furnace for heating fluid is arranged in two parts, in which the fluid (in tubes) is heated by radiation and convection, respectively. The first part is constructed of a number of vertical tubes arranged in a circle, and forming the wall of an elongated combustion space; burning fluid fuel is caused to pass axially upwards as a flame of small diameter, which meets with no obstruction, and is, therefore, substantially out of contact with the tubes but heats them by radiation. The tubes are suspended from their upper ends, and may be easily removed. From the radiation device the products of combustion may pass downwards through a heater of known type with cross tubes, which constitutes the "convection" member.

B. M. VENABLES.

Regenerative furnace. G. E. ROSE (U.S.P. 1,721,885, 23.7.29. Appl., 9.9.22).—Vertical fixed flues, the discharge ends of which can be restricted, are provided on opposite sides of the furnace, and two air- and fuel-port units having restricted air and fuel passages can

be moved so that they register with the furnace end of the flues. When used in alternating operations, the discharge end of the flue is restricted so that the flue acts as an uptake, while the other flue is unrestricted and acts as a downtake.

F. G. CLARKE.

Tunnel kilns. A. HEIMSOOTH, and HEIMSOOTH & VOLLMER GES.M.B.H. (B.P. 315,540, 6.6.28).—A cooling zone is provided for a tunnel kiln comprising a number of individual passages in the roof and side walls. Cooling fluid is supplied to the roof passages at the centre or crown, and leaves at the sides or springing of the arch through individual regulators upwards into a pair of common flues provided with dampers. Other portions of cooling fluid are admitted to the upper parts of the wall passages, leaving at the bottom and passing into a pair of lower flues provided with dampers.

B. M. VENABLES.

Production and repair of refractory linings for hearths of metallurgical and other furnaces. VACUUMSCHMELZE GES.M.B.H. and W. ROHN, Assees. of HIRSCH KUPFER- u. MESSINGWERKE A.-G. (B.P. 303,417, 1.1.29. Ger., 2.1.28. Addn. to B.P. 226,891; B., 1925, 850).—Quartzite of the cement-quartzite type is used as the refractory material.

C. A. KING.

Chemical apparatus for reactions on heated solid material. T. GRISWOLD, JUN., Assr. to DOW CHEM. CO. (U.S.P. 1,719,509, 2.7.29. Appl., 1.2.27).—Apparatus suitable, e.g., for the manufacture of carbon disulphide comprises a removable, vertical, cylindrical reaction chamber set in a common flue system, with a stack of metallic vaporising pots. Molten sulphur supplied to the top pot passes to the bottom pot by means of external conduits, and finally enters the refuse chamber, beneath the reaction chamber, in the vaporous condition. The refuse chamber being below the bottom sulphur pot, the latter remains uncontaminated. Several reaction chambers, with their sulphur pots, may be assembled in one system of flues.

F. G. CLARKE.

Catalytic apparatus. SELDEN CO., Assees. of A. O. JAEGER (B.P. 306,442, 27.4.28. U.S., 20.2.23).—An apparatus embodying the heat-exchange, double countercurrent principle described in B.P. 306,884 (B., 1929, 672) is figured.

C. HOLLINS.

Heat interchanger. E. B. MCCABE and G. E. CHAMBERLAIN, Assrs. to CARBONDALE MACHINE CO. (U.S.P. 1,720,912, 16.7.29. Appl., 1.8.27).—A cylindrical shell has a collar at one end presenting inwardly and outwardly extending flanges. A clamping ring engages the outer face of the inner flange, and a tube sheet, which fits the inside of the shell, engages the inner face of the flange, the parts in these positions being

clamped together. A tube sheet is also detachably secured at the other end of the shell. F. G. CLARKE.

[Refractory] heat exchanger. R. L. FRINK (U.S.P. 1,721,442, 16.7.29. Appl., 2.8.27).—A concrete mix containing a refractory material, low-temperature cement, and glass-forming materials is cast, allowed to set, and heated to produce a glass-like bond for the refractory material. F. G. CLARKE.

Melting ladle for materials of low m.p. I. GONYK (B.P. 316,034, 21.8.28).—The apparatus resembles a soldering iron, but has a combined ladle proper and melting chamber instead of the copper "bit."

B. M. VENABLES.

Fireproof composition. T. SHIGA, Assr. to T. KAWATA (U.S.P. 1,720,926, 16.7.29. Appl., 17.10.27. Jap., 9.3.27).—An aqueous solution of ammonium phosphate and sulphate is mixed with a clear solution of magnesium borate in boric acid.

H. ROYAL-DAWSON.

Drying apparatus. W. TRETOW (B.P. 297,774, 25.9.28. Ger., 27.9.27).—A twin row of drying compartments is divided by a central series of compartments containing fans and air-heating devices. The drying air is caused to pass through the whole device in a zig-zag path, leaving at the point where the goods enter. The goods are preferably drawn through the side compartments on conveyors.

B. M. VENABLES.

Fillings for cooling towers. A. E. SIMMONDS, J. H. SIMMONDS, J. H. TODD, and REUNERT & LENZ, LTD. (B.P. 316,393, 25.6.28).—A number of perforated plates are bent and laid with convexity or ridge upwards in staggered rows within the cooling tower.

B. M. VENABLES.

Crushing mills. E. C. LOESCHE (B.P. 315,781, 14.4.28).—The apparatus described in B.P. 313,690 (B., 1929, 669) is provided with a casing, and provision is made for swinging the rollers out through holes in the casing, the holes being provided with lids.

B. M. VENABLES.

Pulverising mill. HARTSTOFF-METALL A.-G. (HAME-TAG) (B.P. 304,152, 14.1.29. Ger., 14.1.28).—Beaters are rotated in a casing which is wide at the top and converges to the bottom. The top is provided with two openings out of the path of the beaters, and also, if desired, with a third in the plane of the beaters, these openings being used for inlet and outlet of material and air. Deflectors, preferably comprising square plates with a diagonal in the plane of the beater, are also provided.

B. M. VENABLES.

Grinding or mixing mill. F. B. REED, Assr. to CLEARFIELD MACHINE Co. (U.S.P. 1,718,004, 18.6.29. Appl., 17.3.28).—A rotary pan with edge runners is also provided with rotating discs of which the axes are adjustable and which serve to scrape the material in the pan either into the path of the edge runner or out of the pan according to the position of their axes.

B. M. VENABLES.

Mixing, emulsifying, homogenising, comminuting, etc. substances of all kinds. H. HILDEBRANDT (B.P. 284,354, 28.1.28. Ger., 28.1.27).—A rotating member provided with blades, teeth, or the like runs at

an adjustable distance from a fixed member (or they may be pressed together by springs), and between the fixed member and the casing a space is left forming a return passage for the material back to the emulsifying zone.

B. M. VENABLES.

Mixing machine. HOBART MANUF. CO., Assees. of H. L. JOHNSTON (B.P. 291,732, 1.6.28. U.S., 6.6.27).—A bowl for the material (such as dough) is raised into the operating position by means of a hand wheel, worm gear, and rack. The stirrer is driven by an electric motor through a four-speed gear provided with a friction clutch and brake.

B. M. VENABLES.

Fractional separation, grading, and sizing of solid materials in the form of lumps, granular particles, and powders, and the separation of the components of a mixture of different solid materials. B. MOORE (B.P. 315,245, 10.4.28).—The stream of fluid (gas, vapours, or liquid) conveying the comminuted materials in suspension is subjected to a number of alternate abrupt contractions and expansions, separate grades being removed from the duct at points between the constrictions.

B. M. VENABLES.

Removing oil, grease, and like foreign matter from the interior surfaces of steam condensers. S. B. FREEMAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 315,930, 4.5. and 28.7.28).—The condenser, or other apparatus not easily accessible, is filled with the vapour of a volatile solvent such as trichloroethylene, the vapour being produced either in an external apparatus or by means of a removable heating coil in the lower part of the condenser shell. The process of filling with vapour (and displacement of air) can usually be followed by feeling with the hand the temperature of the casing. The tubes will usually be cold enough to cause condensation of liquid solvent (hence removal of grease by dissolution), but, if necessary, cooling water may be passed through the tubes for that purpose, and should be used in any case at the finish to reduce the vapour pressure of the solvent so that there is a negligible quantity left in at the end of the cleaning process. In some apparatus it may be necessary to provide a removable cooling coil.

B. M. VENABLES.

Centrifugal separator. E. S. ECCLESTON (U.S.P. 1,718,547, 25.6.29. Appl., 1.6.26).—A slightly conical bowl is provided with a number of orifices arranged in horizontal rings; the pulp is supplied to the inner surface at points above the orifices, clings to the interior, and works downwardly to the orifices; the outlet of material through the orifices is controlled by angle rings attached to the outside of the bowl, the upstanding flanges obstructing the orifices, and the separated material flows over the flanges into stationary collecting channels.

B. M. VENABLES.

Centrifugal machines. SULZER FRÈRES SOC. ANON. (B.P. 316,503, 20.12.28. Switz., 6.9.28).—When a number of impellers or other elements are mounted on a long shaft of different material and the apparatus is subjected to changes of temperature with consequent differential expansion, an elastic collar is inserted at some point between the impellers to allow for expansion. In the case of a high-pressure pump for circu-

lating hot water, this member may be hollow and kept distended by the pressure of the fluid.

B. M. VENABLES.

Non-rotative centrifugal separator. E. C. R. MARKS. From CENTRIFIX CORP. (B.P. 316,716, 19.5.28).—A vortical type of separator, suspended in a steam boiler at the outlet to ensure delivery of dry steam, is described.

B. M. VENABLES.

Centrifugal apparatus for extracting juice from juicy materials. M. LINARES (B.P. 301,822, 19.10.28. Fr., 6.12.27).—The material is caused to traverse downwards through a conical, perforated basket by means of a worm driven at a speed different from that of the basket. The liquid is caught in a gutter just above the lower edge of the basket, and the basket terminates some distance above the bottom of the worm, so that space is left for the compacted material to be flung out against obstructions which break it up and from which it falls on to a plate rotating comparatively slowly, whence it is removed outside the apparatus. The bearings of the basket and worm are a sufficient distance apart to allow for the above arrangement.

B. M. VENABLES.

Concentration of juices, vegetable extracts, and organic and inorganic solutions generally. G. LA CAUZA (B.P. 316,167, 23.4.28).—Apparatus suitable for concentrating liquids (that might be damaged by heat) by refrigeration is described. A tank of the liquid may be provided with partition walls containing freezing coils upon which walls pure ice is formed, or the liquid contained in specially shaped moulds (e.g., star section) may be placed in the chilled brine bath of an ordinary ice-making plant.

B. M. VENABLES.

Filter. G. H. GREENHALGH, ASSR. to E. J. SWEETLAND (U.S.P. 1,721,250, 16.7.29. Appl., 27.11.23).—The filter is arranged in a casing comprising two similar, counter-part sections, the rims of which are secured together by outwardly projecting flanges. The latter are also engaged by a device for supporting the casing.

F. G. CLARKE.

Filter for fine filtering of fluids, especially fuel oils. T. V. HEMMINGSEN (B.P. 307,003, 26.4.28. Denm., 1.3.28).—The filter described is of the "conical plug and body" type, the surface of the plug being provided with parallel grooves extending alternately to the inlet and outlet ends, the inlet grooves being made larger than the outlet to allow for accumulation of residue, and the outlet grooves being shallow and so shaped that they may be easily cleaned. Two or more filters may be placed inside each other, the largest plug being bored out to receive a smaller one.

B. M. VENABLES.

Film evaporator. COAL OIL EXTRACTION, LTD., ASSEES. of W. RUNGE (B.P. 296,430, 3.8.28. U.S., 2.9.27).—The apparatus is of the type in which a cage comprising substantially horizontal heating tubes rotates in a vessel partly filled with the liquid to be evaporated, so that heat is supplied both to the liquid and to the vapour space above it. Any one tube leading from the rotary inlet header to the rotary outlet header is provided with two return bends so that the heating fluid makes three horizontal zig-zag passes.

B. M. VENABLES.

Vacuum evaporator. G. A. ZEITLER (U.S.P. 1,721,760, 23.7.29. Appl., 8.4.29).—One end of a horizontal cylindrical shell is detachable and the other fixed. A heating unit, which can be inserted through the detachable end, engages steam inlet and exhaust conduits passing through the fixed end of the shell by means of detachable connexions.

F. G. CLARKE.

Production of high vacua. H. S. COOPER, ASSR. to KEMET LABS. CO., INC. (U.S.P. 1,721,544, 23.7.29. Appl., 14.4.27).—The gases in a sealed envelope are converted into solid compounds by means of an alkaline-earth metal, which is liberated in the nascent state by a non-gas-forming exothermic reaction.

F. G. CLARKE.

Production of heavy granular concentrates [from dilute solutions]. A. B. JONES, ASSR. to INDUSTRIAL ASSOCIATES, INC. (U.S.P. 1,721,452, 16.7.29. Appl., 11.3.26).—The solutions are evaporated by spraying, and the light solids so obtained are liquefied and dispersed into an atmosphere sufficiently cool to cause solidification.

F. G. CLARKE.

Reconstruction of liquid mixtures. A. JENSEN (U.S.P. 1,721,121, 16.7.29. Appl., 11.3.26).—A fluid mixture of liquids and normally solid or semi-solid substances is introduced into one end of the narrow space between two concentric, relatively rotating, horizontal cylinders, and its temperature is progressively varied as it passes to the other end, emulsions being thereby obtained.

F. G. CLARKE.

Gas washer. J. C. HAYES, JUN., ASSR. to FREYN ENGINEERING CO. (U.S.P. 1,722,466, 30.7.29. Appl., 26.9.27).—The gas passes upwards first through a number of hurdles and then through a honeycomb of vertical, rectangular passages, each of which is provided with a water spray.

B. M. VENABLES.

Washing of smoke and fumes from furnaces and the like. V. R. CHADWICK (B.P. 314,906, 18.4.28).—The gases are propelled and moistened by jets of water, wet steam, or other fluid blowing through groups of Venturi tubes, which pass through a transverse partition in the flue carrying the gases. The moistening device may be by-passed and access obtained to it for cleaning by means of flap valves or dampers.

B. M. VENABLES.

Separating substances from gases by cooling. KALI-IND. A.-G. (B.P. 306,106, 27.4.28. Fr., 17.2.28).—The gas passes through a chamber within which are rotated at centrifugal speed two or more drums with smooth exterior surfaces which are cooled by an internal fluid. The particles or substances separating on the drums are flung off and collected in the lower part of the casing.

B. M. VENABLES.

Separation of mixed gases by progressive solubility. W. L. DE BAUFRE, ASSR. to S. G. ALLEN (U.S.P. 1,722,458, 30.7.29. Appl., 3.6.24).—The mixed gases enter the lowest zone of a tower-like apparatus and come in contact with a solvent (of different nature from the gases) at a lower pressure. The undissolved gases are then compressed and delivered to an intermediate point of the highest zone of the tower, which is maintained

under pressure and to the extreme top of which the solvent is delivered by a pump drawing from a well at the bottom of the tower. One constituent of the gases (nitrogen in the case of air) is drawn off at the top of the tower, the other (oxygen) being drawn off from a middle section in which the downward-flowing solvent is subjected to the reduced pressure, in the absence of any upward current of gas. B. M. VENABLES.

Apparatus for chemically transforming gases. H. O. C. ISENBERG, ASST. to GEN. CHEM. CO. (U.S.P. 1,719,610, 2.7.29. Appl., 10.10.25).—Apparatus suitable, e.g., for the oxidation of sulphur dioxide comprises a series of annular converters arranged one above the other, with a heat-exchanger in the central space for each converter. The heat-exchangers may each comprise a gas-inlet distributing chamber at the top and a similar gas-outlet chamber at the bottom, the chambers being connected by vertical pipes, which are surrounded by the cooling or heating medium. From the outlet chamber the gas passes through radial pipes to the converter, and then enters the inlet chamber of the next heat exchanger below. F. G. CLARKE.

Preventing contamination of centrifugally purified liquids. W. R. CHADBURN, ASST. to DE LAVAL SEPARATOR CO. (U.S.P. 1,723,329, 6.8.29. Appl., 7.1.26. U.K., 27.1.25).—See B.P. 252,421; B., 1926, 649.

Separation by liquefaction of complex gaseous mixtures. G. CLAUDE, ASST. to L'AIR LIQUIDE SOC. ANON. POUR L'EXPLOIT. DES PROC. G. CLAUDE (U.S.P. 1,724,004, 13.8.29. Appl., 16.7.25. Fr., 7.8.24).—See B.P. 238,174; B., 1925, 872.

Refrigerating apparatus. J. C. SHAW (B.P. 317,598, 6.7.28).

Absorption refrigerating machines. P. VON VÁGÖ and V. MAGYAR (B.P. 317,336, 14.3.28).

Refrigerating machines of the absorption type. G. MAIURI and R. F. BOSSINI (B.P. 317,334, 10.2.28).

Power presses. J. H. ROBERTS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 317,686, 17.11.28).

Welded joints. A. CARPMAEL. From I. G. FARBEN-IND. A.-G. (B.P. 317,140, 15.5.28).

[Pressure-tight joint for bomb] calorimeters. G. E. SCHOLDS (B.P. 315,285, 10.4.28).

Means for indicating the condition of [automotive] filters. A.C. SPARK PLUG CO. (B.P. 291,097, 17.5.28. U.S., 27.5.27).

[Asbestos-bakelite] friction bodies, especially for brakes. KIRCHBACH'SCHE WERKE KIRCHBACH & CO. (B.P. 306,927, 9.5.28. Ger., 28.2.28).

Thickening of pulp (U.S.P. 1,717,604).—See V. Refractory arch for furnaces (U.S.P. 1,719,416).—See VIII. Removal of boiler scale (B.P. 315,287).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Correlation between the adsorptive capacity of coals and peats and their other characteristics. B. P. PENTEGOV (Pub. Far Eastern State Univ. Vladivostok, 1929, No. 12, 3—28).—The degree of fossilisation of coal is characterised by its colloidal state;

advance of fossilisation is associated with decrease in the unsaturated, non-oriented carbon atoms and in the colloidal phase. Hence younger coals contain more hygroscopic moisture. The author classifies coals according to their adsorptive capacity. Weathering decreases the adsorptive power. Similarly the absorptive power of coke is characteristic. The ratio of the value for coal to that for the corresponding coke is usually less than 1 for bituminous coal, approx. 1 for anthracite, less than 1 for peat, and greater than 1 (sometimes greater than 2) for brown coals; the ratio is reduced by weathering, and increases with the depth of the coal stratum. The gas content of coals is also a function of their adsorptive capacity. Spontaneous combustion is apt to follow adsorption of sulphates and their reduction to sulphides. The greater is the adsorption value of a coal, the greater is the yield of ether-soluble hydrogenation products by Fischer's method.

CHEMICAL ABSTRACTS.

Titrimetric determination of carbon-containing gases in exhaust gases of motors. J. HIRSCH (Z. Hyg., 1928, 109, 266—271; Chem. Zentr., 1929, i, 1406).—The significance of the difference between the total carbon and the carbon present as dioxide, as determined by the author's method, is discussed.

A. A. ELDRIDGE.

Accurate determination of the gasoline content of natural gas and analytical separation of natural gas by isothermal fractional distillation. M. SHEPHERD (Bur. Stand. J. Res., 1929, 2, 1145—1199).—“Natural gasoline” is defined as the maximum amount of condensate possessing a fixed vapour pressure (p) at a fixed temperature (T) which can be extracted from the gas, p and T being fixed arbitrarily. It includes all the pentane and hydrocarbons of higher b.p. together with sufficient n -butane to give a saturation pressure p at T° . The determination of gasoline in natural gas involves separation by fractional distillation into three fractions: (1) isobutane and hydrocarbons of lower b.p. together with gases other than hydrocarbons, (2) n -butane, (3) pentane and hydrocarbons of higher b.p., and their re-combination in such a way that the maximum amount of condensate present in the original gas having a vapour pressure p at T° can be determined. Details of apparatus and analyses are given.

C. J. SMITHELLS.

Coumarone in coal tar. ORLOV and BELOPOLSKI.—See III. Minerals and flotation. TAKAHASHI.—See X.

See also A., Sept., 998, Adsorption with active charcoal (ENGEL). 999, Sorption of water vapour by active charcoal (ALLMAND and others). Charcoal as an adsorbent (DUBININ). 1000, Adsorption of weak electrolytes by pure charcoal (PHIELPS and PETERS). 1004, Smoke particles (PATTERSON and others). 1036, Theory of formation of fusain (LIESKE). 1037, Thermochemical investigation of petroleum (ABE and SHOBAYASHI). 1108, Bacterial oxidation of crude oils (TAUSSON).

PATENTS.

Carbonising or distilling material. INTERNAT. COMBUSTION, LTD., and S. McEWEN (B.P. 316,901,

3.4.28).—The material is given a positive and, if necessary, a continuous movement through the retort, and the gases are withdrawn without being decomposed by heat. The bench of retorts (horizontal or vertical) is made up of heating walls alternating with pairs of endless-chain grates provided with projections which engage the material when it is fed into the spaces between the walls and the moving grates. The wall-moving-grate system may constitute the whole or only part of the carbonising chamber. The products of carbonisation are withdrawn (each section separately, if required) through the grates into chambers situated between the two runs of each grate. The retort space may be tapered if necessary. J. A. SUGDEN.

Coking of solid fuel. E. RAFFLOER (U.S.P. 1,723,807, 6.8.29. Appl., 27.5.26).—The material is passed through an externally heated chamber where it is dried and desulphurised and then, while moving through the heating chamber in the opposite direction, is compressed in long, narrow masses, the surfaces being intermittently exposed to allow the free discharge of gases. J. A. SUGDEN.

Production of carbon and hydrogen chloride. R. M. WINTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 317,165, 9. and 29.6.28).—Mixed hydrocarbon vapour or gas and chlorine gas is burnt at a suitable orifice in an atmosphere of air. The flame consists of a highly luminous interior where the chlorine reacts with the hydrocarbons, and an outer mantle where partial oxidation takes place. The gases are led away and the suspended carbon is separated by filtration or electrostatic precipitation etc.; the gases are then washed in water to separate the hydrogen chloride. High yields of hydrogen chloride and a very pure carbon black are obtained. J. A. SUGDEN.

Apparatus for the hydrogenation of coal, oil, or the like. K. GORDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 315,513, 12.5.28).—The reaction vessel is so designed that the walls are protected, by an incoming stream of comparatively cool oil or coal-oil mixture, from the action of hydrogen at high temperatures. Thus, by means of a suitable baffle, the incoming stream, at say 200°, may be made to flow down in contact with the wall of the high-pressure vessel and thence up into the reaction space, which is maintained at the necessary high temperature by introducing the hydrogen at about 500°. A. B. MANNING.

Apparatus for automatically indicating and recording the percentage of carbon dioxide gas contained in the combustion gases of furnace flues and the like. A. and L. LUMB (B.P. 315,510, 7.5.28).—The multiple-way cock described in the original apparatus of Ward (B.P. 23,846 of 1909; B., 1910, 1241) is replaced by two valves of the mushroom type, one to control the supply of gas to the measuring chamber, and the other to control the discharge from the bell-float to the atmosphere. A. B. MANNING.

Distillation of tar. BARRETT CO., ASSEES. OF S. P. MILLER (B.P. 284,703, 28.1.28. U.S., 5.2.27).—In a modification of the processes described in B.P. 282,367 and 282,826 (B., 1929, 507, 633), the gases from the whole battery are collected (without cooling) in one

main where they meet a spray of the tar to be distilled. Deposition of pitch in the main is prevented by continuous flushing with a large amount of hot tar or pitch which is partly withdrawn and partly recirculated. The enriched gases pass on to suitable condensers. The operation may take place in a series of separate small collector mains, and the tar to be distilled may be passed successively from one to the other, thus separating different fractions of the oil. One battery is capable of distilling tar from a large number of batteries. The process may be adapted for dehydrating tars.

J. A. SUGDEN.

Production of pitch. BARRETT CO., ASSEES. OF G. E. McCLOSKEY and W. B. WINGERT (B.P. 289,378, 12.4.28. U.S., 25.4.27).—Coke-oven gases discharged during the "rich" and "lean gas" periods are collected and treated separately whereby two types of pitch (which must ordinarily be obtained from gas-retort and water-gas tars) are obtained, one high and one low in free carbon content. The gases are drawn off through separate mains where they are cooled (by liquor sprays) to a suitable temperature. The pitch separates as a fog and is precipitated electrically. The gases pass on, are cooled and scrubbed, and yield clean oils (free from tarry matter) which may be used without further treatment. J. A. SUGDEN.

Treatment of oils, tars, or pitches derived from coal to modify their viscosities at predetermined temperatures. SOUTH METROPOLITAN GAS CO., H. PICKARD, and H. STANIER (B.P. 316,897, 3.3.28).—To modify the "consistency" of coal tar for road-making or waterproofing purposes without subjecting it to distillation or adding bitumen or asphaltic bituminous substances, the dispersion of not more than 15% of bituminous coal or peat in the tar at 300° is claimed to give a cheaper but equally good product. J. A. SUGDEN.

Apparatus for extracting liquid hydrocarbons from oil shale. A. SCHILLING, R. SACHSE, D. LIAMIN, and T. CALLAERT (U.S.P. 1,721,836, 23.7.29. Appl., 23.11.26).—Shale is introduced at the top of a refractory-lined, vertical, tubular generator which is provided at the top and bottom with fluid-sealed removable closures. Oxygen is introduced above the charge, which rests upon a grate, and just above the latter the gaseous products are withdrawn at the periphery. Liquid products are collected in the lower closure, which can be raised, lowered, and swung laterally.

F. G. CLARKE.

Cracking of hydrocarbon oils. E. W. ISOM, ASST. TO SINCLAIR REFINING CO. (U.S.P. 1,722,147, 23.7.29. Appl., 21.5.27).—Preheated oil is raised to cracking temperature in a heating conduit, and is then discharged into oil in a reservoir maintained at a similar temperature. The resulting gases are subjected to two successive refluxing operations, cooling being effected by means of fresh oil. The heated oil from the first reflux is passed to the reservoir, and that from the second, together with the condensates from both refluxes, is introduced into the heating conduit. F. G. CLARKE.

Treatment of [hydrocarbon] oils. R. T. POLLOCK, ASST. TO UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,721,973,

23.7.29. Appl., 23.3.21. Renewed 29.5.28).—Having passed through a chamber heated by combustion of carbon deposited in a previous cracking operation, a stream of oil enters a second similar chamber in which it is cracked. The oil stream is subsequently introduced into the second chamber, which is heated by combustion of the previously deposited carbon, and is cracked in the first chamber. F. G. CLARKE.

Manufacture of hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 316,422, 13.7.28).—Conversion of mixtures of olefines and acetylene into hydrocarbons of increased mol. wt. is carried out at high temperatures, under elevated, atmospheric, or diminished pressure, and without the deposition of carbon, provided that the heated gases and vapours come into contact only with elements of group IV, *e.g.*, tin, silicon, lead, carbon (as graphite), or with zinc or aluminium or alloys containing substantial amounts of these metals, and, if desired, in the presence of catalysts consisting of anhydrous chlorides of the heavy metals or aluminium chloride. H. S. GARLICK.

Electric arc treatment of liquid hydrocarbons and apparatus therefor. Y. MERCIER (B.P. 316,336 and 316,352, 20.4.28).—(A) In the electrical treatment of liquid hydrocarbons to produce gaseous products the arc is struck while two electrodes are in contact, and these are separated from one another to the maximum extent consistent with the stability of the arc. Alternatively, a high-frequency spark may be made to jump between the electrodes, making a path through the liquid for the normal arc. (B) Oil to be treated enters a container through the central part of one or more stationary, insulated, hollow electrodes, having conical ends to allow spreading of the arc that jumps between them and opposite, insulated, movable electrodes similarly cone-shaped. Oil is pumped between the electrodes, and gases formed are instantaneously cooled by the surrounding oil (thereby favouring the formation of acetylene) and removed from the system by a vacuum pump. [Stat. ref. to (A).] H. S. GARLICK.

Manufacture of unsaturated hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,249, 10.4.28).—Higher olefines and diolefines, mainly of a gaseous character, *e.g.*, propylene and butadiene, are formed by subjecting ethylene to the action of a spark or arc discharge, the gas being passed at a rate of flow higher than that required for the production of acetylene. A. B. MANNING.

Decomposition of liquid hydrocarbons. J. BETHENOD (B.P. 316,905, 4.4.28).—Petroleum or coal-tar oil etc. is decomposed into hydrogen and acetylene (and small quantities of methane, ethylene, and carbon monoxide) by circulating it round an electrically heated conductor at temperatures above 800°. The gaseous mixture as it is evolved is quickly cooled to prevent the decomposition of the acetylene. J. A. SUGDEN.

Refining hydrocarbon distillates. GRAY PROCESSES CORP., Assees. of H. PEASE (B.P. 293,440, 28.2.28. U.S., 7.7.27).—Reactivation and prolongation of the life of solid adsorptive material used for treating fluid hydrocarbon distillates is accomplished by maintaining

superatmospheric pressure on the entering hydrocarbons and periodically reducing the pressure applied to the effluent hydrocarbons. A description of a Cross cracking plant incorporating this process is given.

H. S. GARLICK.

Desulphurisation of hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,439, 14.1.28).—Crude hydrocarbons, *e.g.*, benzene, naphthalene, are freed from sulphur by treating them with hydrogen at elevated temperatures (200—300°) and, if desired, under elevated pressures, in the presence of a metallic sulphide, *e.g.*, molybdenum or nickel sulphide, which is stable under the conditions of working. The metallic sulphide may be mixed with a metallic activator, *e.g.*, chromium or cobalt oxide, and is preferably deposited on a carrier, *e.g.*, activated charcoal. The organic sulphur compounds are converted into hydrogen sulphide. If this is then removed, the mixture of benzene, or other hydrocarbon, with the excess hydrogen, may be passed directly over a hydrogenating catalyst for the production of cyclohexane etc.

A. B. MANNING.

Dewaxing of oils. L. W. NAYLOR, Assr. to CONTINENTAL OIL Co. (U.S.P. 1,722,307, 30.7.29. Appl., 7.6.27).—A long-cut residuum is treated with sufficient diluent to render the wax incompletely soluble at a low temperature, then cooled to such temperature, and the wax precipitated from the solvent mixture in the presence of particles of litharge.

H. S. GARLICK.

Production of montan wax compositions. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,283, 4.4.28. Addn. to B.P. 296,145; B., 1928, 779).—The product described in the main patent, and consisting of salts and esters of bleached montan wax, is improved by the addition of an ester, *e.g.*, the glycol ester, of bleached montan wax, in amount sufficient to reduce the content of salts to less than 25%, preferably to 15—20%, of the mixture. The product may replace carnauba wax, beeswax, etc. in the manufacture of shoe creams, polishes, etc. A. B. MANNING.

Refining crude paraffin etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,117, 3.8.28).—The wax is treated with dilute nitric acid or, in the presence of water, with gaseous mixtures containing small amounts of oxides of nitrogen having an oxidising action, and the product washed with an alkaline agent or with a small quantity of sulphuric acid.

H. S. GARLICK.

Utilisation of petroleum hydrocarbons. A. P. BJERREGAARD, Assr. to DOHERTY RES. Co. (U.S.P. 1,723,008, 6.8.29. Appl., 3.5.24).—The hydrocarbons are vaporised and the vapours cracked at atmospheric pressure; after removal of benzene by condensation any olefines present are esterified.

H. ROYAL-DAWSON.

Removing terpene products from gasoline-terpene mixtures. E. E. REID, Assr. to HERCULES POWDER Co. (U.S.P. 1,722,765, 30.7.29. Appl., 9.8.27).—The terpene products are separated by treating the mixture with liquid sulphur dioxide and removing the lighter hydrocarbon layer.

H. S. GARLICK.

Manufacture of emulsifying and stabilising agents and dispersions obtained therewith. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ, and H. LIMBURG (B.P. 291,393, 7.5.28. Holl., 1.6.27).—Mineral oil fractions, especially those rich in aromatic and hydro-aromatic compounds, are five times treated with 10% of 20% oleum, the acid sludge being separated each time. The sludge from the last two treatments is neutralised and the soluble salts are isolated. The calcium salts are soluble in water and in calcium chloride solutions.

C. HOLLINS.

Flotation oil. R. LUCKENBACH (U.S.P. 1,722,528, 30.7.29. Appl., 23.3.25).—The oil consists of 50–70% of a heavy petroleum residue and 50–30% of a diluent, the latter being a frothing agent containing 30–50% of pine pitch and kerosene.

H. ROYAL-DAWSON.

Manufacture of binder for binding an aggregate [fuel briquettes]. S. W. CARPENTER and G. N. WHITE, ASSRS. to PULP BINDERS DEVELOPMENT CO., LTD. (U.S.P. 1,724,393, 13.8.29. Appl., 9.7.25. U.K., 18.7.24).—See B.P. 244,517; B., 1926, 146.

Oil-gas process. J. E. HACKFORD, ASSR. to HAKOL, LTD. (U.S.P. 1,723,418, 6.8.29. Appl., 22.10.23. U.K., 19.12.22).—See B.P. 217,613; B., 1924, 703.

Treating materials [tars etc.] in liquid state electrochemically. C. LONGHI (U.S.P. 1,720,910, 16.7.29. Appl., 12.12.24. Ital., 17.12.23).—See B.P. 248,830; B., 1926, 525.

Separating congealable solids from oils. J. F. P. SCHÖNFELD, ASSR. to N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (U.S.P. 1,724,732, 13.8.29. Appl., 12.3.25. Holl., 24.3.24).—See Dutch P. 14,653; B., 1928, 836.

Production of lubricants containing water. M. J. HEITMANN (U.S.P. 1,724,653, 13.8.29. Appl., 24.3.26. Ger., 11.4.25).—See B.P. 250,562; B., 1927, 695.

Gas, vaporised-oil, or coal-dust burners for furnaces. F. LILGE (B.P. 317,582, 25.6.28).

Turbulent burners. A. W. BENNIS (B.P. 316,957, 2.4.28).

Coke extractors for vertical retorts for distillation of coal and the like. E. WEST, and WEST'S GAS IMPROVEMENT CO., LTD. (B.P. 317,257, 11.10.28).

Coke extracting mechanism for vertical retorts for carbonisation of coal and the like. F. J. and E. WEST, and WEST'S GAS IMPROVEMENT CO., LTD. (B.P. 317,284, 29.12.26).

Filtering fuel oils (B.P. 307,003).—See I. Treatment of unsaturated carbon compounds (B.P. 289,414–5). Organic acids from paraffins (B.P. 315,813).—See III. Paper from peat (U.S.P. 1,721,974).—See V. Treating oxides with hydrocarbons (B.P. 315,459). Gas purification (B.P. 288,977).—See VII. Bituminous emulsions (B.P. 315,495). Wood preservative (U.S.P. 1,722,323). Fungicide (U.S.P. 1,720,905).—See IX. Blue pigments (B.P. 317,274). Plastic substances (B.P. 302,270).—See XIII. Adhesive rubber (U.S.P. 1,719,948).—See XIV.

III.—ORGANIC INTERMEDIATES.

Extraction of acetic acid from its dilute solutions. S. P. NASAKIN (J. Chem. Ind. Moscow, 1928, 5, 1149–1157, 1369–1376).—As solvent a fraction from crude wood alcohol and coal tar, d_{20}^4 0.905–0.920, b.p. 50–70°, consisting (80–85%) of esters of lower fatty acids and alcohols of low b.p., the remainder being ketones, is employed with good results.

CHEMICAL ABSTRACTS.

Cresyl [tolyl] esters of phenylacetic acid. L. C. RAIFORD and J. G. HILDEBRAND, JUN. (Amer. J. Pharm., 1929, 101, 481–484).—By warming phenylacetyl chloride with *o*-, *m*-, or *p*-cresol the following phenylacetates are prepared: *o*-tolyl, m.p. 44–45°; *m*-tolyl, m.p. 51–52°; *p*-tolyl, m.p. 74–75° (lit. 86°).

R. K. CALLOW.

Manufacture of ethylene chlorohydrin. E. A. SHILOV (J. Chem. Ind. Moscow, 1928, 5, 1273–1276).—Directions for the manufacture of ethylene chlorohydrin by passing ethylene into hypochlorous acid solution are given. The gas obtained by passing ethyl alcohol vapour through a tube containing pumice moistened with phosphoric acid at 250° contains 95% of ethylene. Hypochlorous acid can be obtained in theoretical yield by adding alkali to a cold solution into which chlorine is being passed; the chlorine is maintained in excess, the solution being finally treated with air or calcium carbonate. The velocity of absorption of ethylene in hypochlorous acid solution increases with the rate of stirring, and the formation of the chlorohydrin is catalysed by hydrogen ions. The chlorohydrin, which is obtained by operation in dilute solutions in a yield of 80%, calculated on the hypochlorous acid present, is only slightly oxidised by the hypochlorous acid.

CHEMICAL ABSTRACTS.

Manufacture of ethylene chlorohydrin. M. B. ZAPADINSKI (J. Chem. Ind. Moscow, 1928, 5, 1426–1429).—Directions for the manufacture of ethylene chlorohydrin by passing ethylene and chlorine into water are given. The addition of *s*-dichloroethane or alcohol decreases the yield; operation in presence of petroleum is of doubtful advantage. The ethylene chlorohydrin is separated by distillation, followed by extraction with *s*-dichloroethane and distillation through a dephlegmator.

CHEMICAL ABSTRACTS.

Separation of carbazole from crude anthracene. B. V. MAKOROV (J. Chem. Ind. Moscow, 1929, 6, 41–45. Cf. G.P. 386,597 and 393,693; B., 1924, 590).—The formaldehyde method is satisfactory if the anthracene is first washed with xylene to remove phenanthrene and other impurities. Repetitions of the treatment fail to remove the nitrogen completely. Two or three treatments with a mixture of ethyl alcohol (150 pts.), formalin (15 pts.), and sodium carbonate (3 pts. per 50 pts. of crude anthracene) are necessary; an excess of sodium carbonate with prolonged heating is undesirable. Anthracene previously treated with potassium hydroxide cannot be freed from nitrogen compounds by formaldehyde. Treatment with potassium hydroxide should be carried out without access of air, and at a low temperature for a short time. Anthracene may be treated in a Soxhlet apparatus with a formaldehyde mixture

containing 30–40% aqueous alcohol, using lime as the alkali.

CHEMICAL ABSTRACTS.

Synthesis of thiocarbanilide. M. N. UEDINOV, N. S. DROZDOV, and N. A. STEPANOV (J. Chem. Ind. Moscow, 1929, 6, 37–39).—Thiocarbanilide was prepared in 86–88% yield by the method of B.P. 244,070 (B., 1926, 721). The relative proportions of carbon disulphide and aniline employed greatly influence the yield. When the carbon disulphide is in 10–20% excess the yield increases to 94%; an excess of 20–40% does not further affect the yield, whilst a greater excess decreases it as a result of the reaction: $\text{NH}_2\text{Ph} + \text{CS}_2 = \text{NPh}:\text{CS} + \text{H}_2\text{S}$. An excess of aniline reduces the yield of thiocarbanilide owing to the tendency to form triphenylguanidine. The highest temperature of reaction (77°) is reached when the yield is best, the excess of carbon disulphide being 10–20%.

CHEMICAL ABSTRACTS.

Formation of coumarone in coal tar. N. A. ORLOV and M. A. BELOPOLSKI (Ber., 1929, 62, 1752–1754).—By passing hexahydrodiphenylene oxide through a tinned-iron tube at 700° small amounts of a polymeric methylcoumarone are produced. The bearing of the observation on the presence of coumarones in coal tar is discussed.

H. WREN.

See also A., Sept., 1021, **Electrolytic preparation of iodoform** (VYSKOČIL). 1057, **Catalytic hydrogenation under pressure in presence of nickel salts: α - and β -hydroxy- and amino-anthracenes** (VON BRAUN and BAYER). 1070, **2:4-Dinitrobenzaldehyde as reagent** (BENNETT and PRATT). 1080, **Pyrazolines** (VON AUWERS and CAUER). 1082, **Diphenylmethylpyrazoles** (VON AUWERS and SCHAUM). 1104, **Toxicity of hydrocarbon vapours** (LAZAREV). 1108, **Acetic acid fermentation** (ROSENBLATT and MORDKOVITSCH).

PATENTS.

Manufacture of glacial acetic acid. A. H. MAUDE, ASSR. to RUBBER SERVICE LABS. CO. (U.S.P. 1,722,532, 30.7.29. Appl., 18.9.26).—The dilute acid (10% of water) is heated with 20 vol.-% of benzol and then subjected to single-stage distillation.

F. G. CLARKE.

Manufacture of acetone from acetylation liquors. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 300,548, 2.11.28. Ger., 14.11.27).—Acetylation liquor from the manufacture of cellulose acetate is evaporated and the vapours are led over a ketonising catalyst (ceria on pumice) at 480–500°.

C. HOLLINS.

Hydrogenation of aldehydes. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 316,399, 29.6.28).—Crotonaldehyde vapour is passed with hydrogen (and preferably with steam) at 120° over a mixed copper and/or silver catalyst containing an oxide or hydroxide of iron, cobalt, nickel, zinc, calcium, strontium, barium, magnesium, chromium, or manganese, not completely reduced under the conditions of reaction; copper and/or silver, mixed with iron and/or nickel and oxide of iron and/or nickel, may also be used. Butyl alcohol is obtained in good yield.

C. HOLLINS.

Catalytic hydrogenation of non-nitrogenous organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 306,803, 14.4.28. U.S., 25.2.28).—Vapour-

phase hydrogenation of such compounds is conducted with the aid of a siliceous or non-siliceous base-exchange material carrying or containing the effective catalyst. *E.g.*, mixed solutions of aluminium chloride and nickel nitrate are added to a stirrable paste of sodium silicate solution and kieselguhr, and the precipitated jelly is washed, dried, and reduced with water-gas or hydrogen at 250–350°.

C. HOLLINS.

Catalytic reduction and hydrogenation of organic nitrogen compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 304,640, 19.3.28. U.S. 23.1.28).—Siliceous or non-siliceous base-exchange bodies containing copper, tin, silver, nickel, iron, cobalt, or zinc are used as catalysts for hydrogenation of nitro-compounds, oximes, nitriles, amines, etc.

C. HOLLINS.

Catalytic oxidation of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 295,270 and 300,968, [A] 6.2.28, [B] 21.11.28. U.S., [A] 8.8.27, [B] 21.11.27).—(A) A non-siliceous base-exchange body containing the catalytic material physically associated or chemically combined in exchangeable or non-exchangeable form is used as catalyst in a variety of organic vapour-phase oxidations. Thirty-two examples of such preparations, usually containing vanadium, are described. (B) The heat-exchange, double countercurrent principle described in B.P. 306,884 (B., 1929, 672) is applied to vapour-phase oxidations of organic compounds.

C. HOLLINS.

Manufacture of crotyl [β -butenyl] bromide. I. G. FARBENIND. A.-G. (B.P. 289,777, 27.4.28. Ger., 30.4.27).—Butadiene is passed into a solution of hydrogen bromide in glacial acetic acid.

C. HOLLINS.

Manufacture of butadiene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,595, 11.8.28).— α -Butylene glycol vapour is led over a red phosphorus catalyst at 250–300°. With sodium dihydrogen phosphate or potassium aluminium sulphate mixed with 5% of red phosphorus yields of 98–99% are obtained.

C. HOLLINS.

Transformation of olefines into alkylene chlorohydrins by means of chlorine water. T. GOLDSCHMIDT A.-G. (B.P. 293,754, 3.7.28. Ger., 11.7.27).—Water is circulated from a vessel, *A*, through a pump, *B*, and returns by a pipe-line, *C*, to *A*. Chlorine is introduced between *A* and *B*, and olefine gas, forced into *C* shortly beyond *A*, forms a froth with the chlorine water and is rapidly converted into chlorohydrin. Cold water is added at *A* and solution of chlorohydrin overflows at the same rate into a receiver.

C. HOLLINS.

Treatment of substances containing unsaturated carbon compounds for the purpose of modifying the physical properties thereof. L. AUER (B.P. 289,414–5, 26.4.28. Hung., 26.4.27).—(A) Oils, resins, and other unsaturated compounds, which have been “modified” by treatment above 200° with metals, metal oxides or hydroxides, or organic bases, are sulphurised or vulcanised with sulphur, sulphur chloride, etc., with or without addition of fillers, accelerators, etc. (B) In the process of B.P. 287,943 (B., 1928, 398), especially as applied to unsaturated fatty oils, the modifying agent is used in solution in an organic

solvent; *e.g.*, sodium amyloxide in amyl alcohol for modification of tung oil, sodium chlorate and β -naphthol in alcohol for rubber dissolved in benzene, trichloroacetic acid in benzene for asphalt. The products may be sulphurised or vulcanised, or emulsified. C. HOLLINS.

Manufacture of monocarboxylic acids. I. G. FARBENIND. A.-G. (B.P. 315,892, 17.2.28. Addn. to B.P. 262,101; B., 1928, 596).—In the process of the prior patent the life of the catalyst is prolonged by removing sulphur compounds from the entrant vapours by means of a countercurrent of silica gel, pumice, or active carbon at about 360°. C. HOLLINS.

Manufacture of hydroxy-aliphatic acids [and derivatives] or salts thereof. H. DREYFUS (B.P. 316,158—9, 24.3. and 18.4.28).—(A) Monoalkyl esters or (B) acid salts of dibasic acids (oxalic, malonic, succinic) are hydrogenated at 15—100° and at 100—150 atm. in presence of reduced nickel etc. C. HOLLINS.

Recovery of organic acids from oxidation products of paraffin hydrocarbons, waxes, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,813, 16.3. and 22.10.28).—The acid oxidation products are stirred with lime or magnesia at raised temperature (135—160°) in presence or absence of water; the dried soap is extracted with benzene and decomposed with mineral acid. C. HOLLINS.

Manufacture of quaternary ammonium compounds. SOC. CHEM. IND. IN BASLE (B.P. 294,582, 26.7.28. Switz., 26.7.27. Addn. to B.P. 219,304; B., 1925, 899).—The soap-like products of the prior patent are converted by alkylating agents into corresponding quaternary ammonium salts (*e.g.*, the methiodide of *N*-oleyl-*N'*-diethylethylenediamine) which are not precipitated by alkali. C. HOLLINS.

Obtaining from seaweed a stable form of alginic acid and its compounds. O. REYNARD (B.P. 316,119, 23.4.28).—Well-leached seaweed is boiled for not more than 1 hr. with dilute aqueous sodium carbonate, the pulp is ground, diluted, filtered in not more than 4 hrs. from the commencement, and calcium alginate is precipitated by addition of calcium chloride, compressed into thin films, dried, and treated with concentrated hydrochloric acid or converted into the sodium salt. C. HOLLINS.

Manufacture of dinitrophenol. D. B. BRADNER and F. H. BEALL (U.S.P. 1,723,761, 6.8.29. Appl., 29.5.23).—The reaction between nitric acid and benzene in the presence of mercury is accelerated by the addition of a fatty acid as catalyst, which increases the solubility of the benzene in the acid. E. LEWKOWITSCH.

Manufacture of 4-(*p*-oxyethylamino)-1-oxybenzene [*p*- β -hydroxyethylaminophenol]. I. G. FARBENIND. A.-G. (B.P. 301,808, 5.12.28. Ger., 5.12.27).—Quinol is boiled with β -aminoethyl alcohol, and after removal of excess quinol *p*- β -hydroxyethylaminophenol is extracted from sodium carbonate solution with ethyl acetate or precipitated as sulphate from alcoholic solution. C. HOLLINS.

Preparation of aminophenol ethers. E. F. GREYER, Assr. to Dow CHEM. Co. (U.S.P. 1,722,417, 30.7.29. Appl., 2.6.26).—A phenol is coupled with a

diazo compound, and the product is alkylated and finally reduced with iron and an iron salt in presence of water. C. HOLLINS.

Production of salts of aromatic hydroxyaldehydes [vanillin and bourbonal]. GRAESSER-MONSANTO CHEM. WORKS, LTD., and D. P. HUDSON (B.P. 316,444, 4.8.28).—Vanillin and bourbonal yield "acid" sodium salts of the type, R·ONa, R·OH, which are sparingly soluble and may be used for isolation of the hydroxyaldehydes. Acid potassium salts may be prepared in alcohol. C. HOLLINS.

Separation, isolation, and purification of aromatic hydroxy-acids. GRAESSER-MONSANTO CHEM. WORKS, LTD., and D. P. HUDSON (B.P. 316,703, 12.5.28).—Salicylic acid is separated from *p*-hydroxybenzoic acid by fractional acidification of a solution of the sodium salts, or by fractional dissolution of the acids in alkali. The process is generally applicable to aromatic hydroxy-acids. C. HOLLINS.

Manufacture of derivatives of the benzene, naphthalene, and acenaphthene series. L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 315,200, 3.1.28).—Sulphonated amines of the naphthalene, acenaphthene, or benzene series (*e.g.*, α -naphthylamine-3:6:8-trisulphonic acid or its alkaline fusion products) are condensed with a nitroaryl chloride and reduced, the process being repeated at least once, and finally the product is condensed with an aliphatic or aromatic acid chloride containing no nitro-group. C. HOLLINS.

Manufacture of a condensation product of α -naphthylamine and acetaldehyde, and application thereof in the manufacture of vulcanised rubber. CLAYTON ANILINE Co., LTD., and R. ROBINSON (B.P. 316,761, 20.7.28).—Condensation of α -naphthylamine (1 mol.) with acetaldehyde (2 mols.) preferably in the presence of an indifferent solvent such as alcohol yields a product which possesses valuable anti-ageing properties for rubber. D. F. TWISS.

Manufacture of aminoalkylarylcarbinols or *N*-alkylaminoalkylarylcarbinols. SOC. CHEM. IND. IN BASLE (B.P. 296,006, 22.8.28. Ger., 22.8.27).—The corresponding ketones, *e.g.*, *N*-methyl-3:4-dihydroxyphenylamine, are hydrogenated in alkaline solution in presence of a nickel catalyst. C. HOLLINS.

Manufacture of ureas and thioureas of the naphthalene series. G. M. DYSON and A. RENSHAW (B.P. 314,909, 1.2.28).—Carboxylated carbamides or thiocarbamides containing at least one aminobenzamido-residue are converted into the corresponding acid chlorides, which are then condensed with α -naphthylamine- or acenaphthene-sulphonic acids. The process provides an alternative route to compounds of the Bayer 205 type. C. HOLLINS.

Manufacture of sulphur-containing hydroxyquinones. I. G. FARBENIND. A.-G. (B.P. 296,761, 6.9.28. Ger., 7.9.27).—A thionaphthen-2:3-dicarboxylic anhydride is condensed with quinol (or other phenol with unsubstituted 2:3-positions) in one operation to give a hydroxylated phthaloylthiophen-2:3-(3':6'-Dihydroxyphthaloyl)thionaphthen, m.p. 253—
h 2

254°, and its 5-chloro-7-methyl (m.p. 291—293°) and 2'(or 3')-hydroxy- (m.p. 241—242°) derivatives are described.

C. HOLLINS.

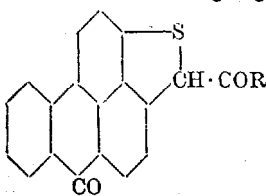
Manufacture of 1-methyl-5-chlorobenzene-2-carboxamino-3-thioglycollic acid [5-chloro-2-carbamyl-*m*-tolylthioglycollic acid]. E. HOFFA, J. and F. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,703,145, 26.2.29. Appl., 9.8.27. Ger., 16.4.27).—5-Chloro-2-cyano-*m*-tolylthioglycollic acid is hydrolysed to the amide, m.p. 173—174°, with 90—98% sulphuric acid.

C. HOLLINS.

Manufacture of hydroxythionaphthens [thioindoxyls]. I. G. FARBENIND. A.-G. (B.P. 288,986, 17.4.28. Ger., 16.4.27).—*o*-Carbamylarylthioglycollic acids, $\text{NH}_2\cdot\text{CO}\cdot\text{Ar}\cdot\text{S}\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, lose ammonia when heated with aqueous alkali, giving thioindoxyls and thioindoxylic acids. Thus 5-chloro-2-carbamyl-*m*-tolylthioglycollic acid yields 6-chloro-4-methylthioindoxyl, which on oxidation is converted into the thioindigo.

C. HOLLINS.

Manufacture of condensation products and vat dyes of the benzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 316,172, 24.3. and 5.9.28).—Compounds of the type 3-benzanthronyl acetyl (or phenacyl) sulphide are converted by alkaline condensing agents (potassium hydroxide in



pyridine, alcoholic potassium hydroxide, anhydrous sodium sulphide) at 35—60° into cyclic compounds (annexed formula), which on removal of the acyl group by alkaline or acid hydrolysis followed by oxidation yield *peri*-thioindigos of the benzanthrone series. These are blue-green vat dyes.

C. HOLLINS.

Manufacture of 1-aminoanthraquinone-2-sulphonic acid. I. G. FARBENIND. A.-G. (B.P. 289,097, 23.4.28. Ger., 23.4.27).—1-Aminoanthraquinone is sulphonated with oleum at 60—120° in presence of an equal weight of anhydrous sodium or potassium sulphate, which prevents formation of the 4-sulphonic acid and also oxidation.

C. HOLLINS.

Manufacture of condensation products of the anthraquinone series. A. CARMAEL. From I. G. FARBENIND. A.-G. (B.P. 315,911, 20.4.28).—A primary amino-derivative of anthraquinone is treated below 100° with alcoholic alkali, preferably in presence of an oxidant (e.g., a current of air). New compounds of unknown constitution are thus obtained from 1- or 2-aminoanthraquinone and 1-aminoanthraquinone-2-sulphonic acid.

C. HOLLINS.

Preparation of tetrazoles. A. BOEHRINGER (B.P. 287,924, 16.3.28. Ger., 30.3.27. Addn. to B.P. 280,529; B., 1929, 350).—Tetrazoles are obtained by the action of sodium azide on imido-chlorides prepared by treating monosubstituted acid amides with phosphoryl chloride, benzenesulphonyl chloride, etc. ϵ -Leucine lactam thus gives pentamethylenetetrazole.

C. HOLLINS.

Chemical apparatus (U.S.P. 1,719,509). **Catalytic apparatus** (B.P. 306,442).—See I. **Hydrocarbons**

(B.P. 316,422). **Unsaturated hydrocarbons** (B.P. 315,249). **Emulsifying and stabilising agents** (B.P. 291,393).—See II. **Wetting-out agent** (B.P. 316,356).—See VI. **Catalysts** (B.P. 316,133).—See VII. **Use of dyes in isolation of organisms.** PROUTY.—See XVI.

IV.—DYESTUFFS.

See A., Sept., 1064, **Reaction of resorcinol and new coloured indicator** (BEY and FAILLEBIN). 1067, **Formulation of dye salts** (DINKLAGE). 1069, **Synthesis of substantive dyes of dicinnamoylmethane group** (LAMPE). 1077, **Carotinoid pigment from maize** (KARRER and others). 1084, **Vat dyes from dibenzodithiazinequinone** (SHIBATA and others). 1084, **Determination of methylene-blue** (FRANÇOIS and SEGUIN). 1093, **Combination between basic dyes and proteins** (RAWLINS and SCHMIDT). 1113, **Grape pigments** (ANDERSON and NABENHAUER).

PATENTS.

Manufacture of vat dyes [of the perylenetetra-carboxylic di-imide series]. M. P. SCHMIDT and W. NEUGEBAUER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,715,430, 4.6.29. Appl., 17.12.23. Ger., 27.12.22).—See G.P. 412,122; B., 1925, 625. The diphenoxyperylenetetra-carboxylic di-imide (violet-blue vat dye) is specially claimed.

C. HOLLINS.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,506, 2.5.28. Addn. to B.P. 291,546; B., 1928, 598).—As initial materials in the process of the prior patent sulphonated or nitrated 1:13-dihydroxy- $\alpha\alpha'\beta\beta'$ -dinaphthazines are used. The dinitro-compound yields a pure red vat dye.

C. HOLLINS.

Manufacture of derivatives of [indigoid] vat dyes and printing therewith. SOC. CHEM. IND. IN BASLE (B.P. 291,768, 8.6.28. Switz., 8.6.27).—Leuco-compounds of indigoid vat dyes are condensed with halo-genated aliphatic acids (chloroacetic acid, β -chloro-propionic acid) to give soluble derivatives (carboxylated ethers), which regenerate the vat dye on treatment with acid oxidants (nitrous acid) and are suitable for printing.

C. HOLLINS.

Manufacture of [thioindigoid] vat dyes. I. G. FARBENIND. A.-G. (B.P. 288,554, 11.4.28. Ger., 11.4.27).—5-Carboxy-1:2:4-trimethylphenylthioglycollic acid, or the corresponding 5-nitrile or amide, is converted by the usual methods into 4:4':6:6':7:7'-hexamethyl-thioindigo, which has good light-fastness.

C. HOLLINS.

Dyes of the anthraquinone series and their application. IMPERIAL CHEM. INDUSTRIES, LTD., A. SHEPHERDSON, and W. W. TATUM (B.P. 315,905, 19.4.28).—A 4-, 5-, or 8-halogeno-1-amino(or alkylamino)-anthraquinone, or a 2-chloro- or -bromo-derivative thereof, is condensed with 1 mol. of *p*-phenylenediamine (or its monoacetyl or oxalyl derivative, subsequently hydrolysed) in an aqueous medium in presence of potassium carbonate and copper acetate. The 2-halogen atom may subsequently be replaced by a sulphonic group. 1-Methylamino-4-*p*-aminoanilinoanthraquinone gives a bluish-green shade on acetate silk and

may be sulphonated with oleum for an acid dye; the 2-sulphonic acid prepared from 2:4-dibromo-1-aminoanthraquinone dyes wool in level bluish-green shades. 1-Amino-5-*p*-aminoanilinoanthraquinone and the 1:8-isomeride give bluish-red shades on acetate silk.

C. HOLLINS.

Production of [vat and acid] dyes [anthraquinone-acridones]. R. S. BARNES, J. E. G. HARRIS, B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 315,328, 9. and 16.1.28).—Acridones of the anthraquinone series are obtained by heating at 90–100° *o*-carboxylated α -arylaminanthraquinones or their "leuco"-compounds with pyridine-sulphuric anhydride in pyridine or other diluent. 1- β -Naphthylaminanthraquinone-2-carboxylic acid or its leuco-compound gives a red vat dye; 1-anilinoanthraquinone-2-carboxylic acid gives a red acid dye, whilst its leuco-compound yields a reddish-violet vat dye.

C. HOLLINS.

Manufacture of sulphur dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 315,910, 20.4.28).—Dinaphthylene dioxide is heated with sulphur at 240–280° to give a reddish-brown sulphide dye. Sulphur chloride with or without sulphur at 80–260° gives a violet-brown.

C. HOLLINS.

Manufacture of azo dyes capable of after-treatment with metallic salts. I. G. FARBENIND. A.-G. (B.P. 288,983, 16.4.28. Ger., 16.4.27).—An *o*-aminophenol is coupled through a middle component with a 2:3-hydroxynaphthoic arylamide in substance or on the fibre, sulphonic and carboxylic groups being excluded. *E.g.*, *o*-aminophenol \rightarrow 2:5-dimethoxyaniline \rightarrow 2:3-hydroxynaphthoic β -naphthylamide gives a deep black which is improved by coppering.

C. HOLLINS.

Manufacture of dis- and poly-azo dyes [for cotton]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 315,451, 11.4.28).—A heterocyclic compound, having two successively replaceable halogen atoms and carrying no groups attached to the nuclear carbon atoms through nitrogen, sulphur, or oxygen, is condensed (i) with an aminoazo compound (or with a diamine etc., followed by diazotisation and coupling) and (ii) with a coupling component which has been or is subsequently coupled with a diazo compound, the two condensations being in either order. The following heterocyclic compounds are suitable: 2-chloropyridine-5-carboxylic acid, 2:4-dichloroquinoline, 2-chloro-3-ketoindolenine-5-carboxylic chloride, *ms*-chloroacridinesulphonyl chloride, 2:4:8-trichloropurine, the 2:4:6-trichloro-, 2:6-dichloro-4-methyl-, and 2:6-dichloro-4-phenyl-pyrimidines, 2-chloro-*peri*-naphthiminazole-6(7)-sulphonyl chloride, 1:4-dichlorophthalazine, 2:4-dichloroquinazoline, 2-chloro-4-phenylquinazolinesulphonyl chloride, 4-chloro-2-(*m*-chlorocarbonylphenyl)quinazoline, 2:4:7-trichloro- and 2:4-dichloro-5:7-dibromo-quinazolines, 4:4'-dichloro-6:6'-diquinazoline, 4-chloro-1-(*p*-chlorocarbonylphenyl)phthalazine, 2-chloroquinoxaline-3-carboxylic chloride, 2:3-dichloroquinoxaline-6-carboxylic chloride, 2:4-dichlorocopazoline, 1:3-dichloro-2:4:5-anthratiazine. The product from 2:4-dichloroquinazoline, H-acid, and 4-amino-4'-hydroxy-5'-methoxyazobenzene-3'-carboxylic acid is coupled with diazotised [H-acid \rightarrow cresidine] to give a direct yellowish-green. Other

examples include a direct green obtained by condensing 2:5-dichloroquinazoline with [5-sulpho-*m*-aminobenzoic acid \rightarrow *o*-anisidine] and [aniline-2:5-disulphonic acid \rightarrow Cleve acid \rightarrow α -naphthylamine \rightarrow J-acid], and a direct blue (developed to green with phenylmethylpyrazolone) obtained by condensing 2-chloropyridine-5-carboxylic chloride first with J-acid then with *m*-nitromethyl-aniline, reducing, and coupling with diazotised [aniline-*o*-sulphonic acid \rightarrow Cleve acid \rightarrow 2-ethoxy-Cleve acid].

C. HOLLINS.

Preparation of [direct] trisazo dyes. H. JORDAN, ASSR. to E. I. DU PONT DE NEMOURS & CO. (U.S. 1,716,063, 4.6.29. Appl., 22.10.25).—Direct green to olive dyes of the type *o*-toluidine \rightarrow Cleve acid \rightarrow H-acid \rightarrow acetoacetanilide are described. Other first components are sulphanilic acid, naphthionic acid, *o*-chloroaniline, α -naphthylamine; second components are Cleve acid or α -naphthylamine; S-acid may be used for H-acid, and as end-components phenylmethylpyrazolone, 2-methylindole, or acetoacet- α -naphthylamide may replace acetoacetanilide.

C. HOLLINS.

Trisazo [developing] dye and its production. H. E. WOODWARD, ASSR. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,716,098, 4.6.29. Appl., 2.3.28).—A sulphonated aminoazo compound is diazotised and coupled with *p*-aminobenzoyl-J-acid, rediazotised, and coupled with J-acid. Examples of aminoazo compounds are metanilic acid \rightarrow α -naphthylamine and *m*-4-xylylidine-5-sulphonic acid \rightarrow *p*-xylylidine; these give red-violet direct shades on cotton, and may be developed with β -naphthol.

C. HOLLINS.

Diazo salt preparations for dyeing and printing. F. KELLER and K. SCHNITZPAHN, ASSRS. to GRASELLI DYESTUFF CORP. (U.S.P. 1,717,453, 18.6.29. Appl., 24.3.27. Ger., 19.1.25).—See B.P. 246,181; B., 1927, 646.

Production of azo dyes on weighted silk. R. KONRAD, ASSR. to GRASELLI DYESTUFF CORP. (U.S.P. 1,723,183, 6.8.29. Appl., 30.4.27. Ger., 11.5.26).—See B.P. 271,089; B., 1928, 708.

[Manufacture of] nitrosoamine-alkali salts of halogenated and methyl-substituted amines. A. ZITSCHER and W. SEIDENFADEN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,724,062, 13.8.29. Appl., 16.12.27. Ger., 31.12.26).—See B.P. 307,965; B., 1929, 550.

Bisaminoarylanthrone and anthrone derivatives. K. SCHIRMACHER, B. STEIN, and K. STENGER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,724,045, 13.8.29. Appl., 21.7.26. Ger., 30.7.25).—See B.P. 270,840; B., 1927, 518.

Benzanthrone vat dyes (B.P. 316,172).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemistry of viscose. G. KITA and R. TOMIHISA (Cellulosechem., 1929, 10, 134–140. and Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1929, 11, 171–192).—In the formation and ripening of viscose the formation of xanthate by the action of carbon disulphide on the alkali-cellulose, and of sodium carbonate and thiocarbonate by its action on free sodium hydroxide, proceed simultaneously. The first reaction product is probably more unstable than the

second, so that the once-formed xanthate decomposes again. The reaction proceeds towards an equilibrium condition between the xanthate, sodium carbonate, and thiocarbonate until finally all the sodium hydroxide is used up and the viscose is coagulated through the accumulation of the salts formed. Increase of temperature assists the consumption of sodium hydroxide and carbon disulphide, but at higher temperatures the xanthate should be decomposed more quickly. The opinion of Cross and Bevan, that coagulation only occurs if the content of carbon disulphide and sodium hydroxide amounts to $\frac{1}{2}$ mol. per mol. of $C_6H_{10}O_5$, is considered unsound. Coagulation does not depend on the composition of the xanthate, but is caused by the disappearance of free sodium hydroxide. The viscose is already coagulated even if the xanthate contains 0.5 mol. of combined sodium hydroxide. According to Cross and Bevan, Ost, and others the xanthate first formed gradually eliminates carbon disulphide and sodium hydroxide which give sodium carbonate and thiocarbonate according to the second reaction, but experiments by the present authors show that the formation of the latter compounds does not always proceed as a result of the decomposition of xanthate. At lower temperatures xanthate formation occurs during ripening, although at room temperature such is not the case and apparently only decomposition of xanthate takes place. It is not rational to suppose, as do the authors mentioned, that a completely combined xanthate is produced corresponding with 1 mol. of sodium hydroxide and carbon disulphide to each mol. of $C_6H_{10}O_5$. Heuser states that the combined alkali in the xanthate cannot exceed 0.5 mol., and considers the xanthate to be derived from $2C_6H_{10}O_5 \cdot NaOH$. This is so if the viscose is prepared by the Cross and Bevan method, but if other methods are used as much as 0.8 mol. of alkali may be combined.

B. P. RIDGE.

Structure of soft woods as revealed by dynamic physical methods. A. J. STAMM (Coll. Symp. Mon., 1928, 6, 83—108).—The total effective capillary cross-section of various soft woods was determined by an electro-endosmotic flow method, and data obtained by combining the results with those of experiments on hydrostatic flow are recorded. CHEMICAL ABSTRACTS.

***Pinus Hamiltoni* [for paper-making].** J. ECHEVERRIA and S. DE PEDRO (Bull. Inst. Pin., 1929, No. 57, 55—57).—The wood of this variety of *P. maritimus* gave: water 59.60, alcohol-ether extract 0.688, cellulose 21.55%. The pulping qualities are recorded.

CHEMICAL ABSTRACTS.

Chemical determination of mechanical wood pulp in paper. E. H. RIESENFELD and T. HAMBURGER (Cellulosechem., 1929, 10, 125—126).—The microscopical method gives values depending on the skill of the worker to within $\pm 5\%$, but the chemical method of Halse (B., 1926, 8) gives values which are accurate to a fraction of a per cent. provided that the lignin contents of the individual constituents of the paper are known. If, however, it is necessary to assume values between 1.6% and 8% for the lignin content of sulphite-cellulose, and 26.6% for that of mechanical wood pulp, as is suggested, errors of the order of 20% may be obtained

in the values for the amount of mechanical wood pulp present, and entirely wrong conclusions may be drawn.

B. P. RIDGE.

Natural system for the analysis and classification of paper. A. S. HAMMOND (Paper Trade J., 1929, 88, 55—58).—The system is based on determinations of the "bulk index," the porosity, and the apparent density of the fibre substance. CHEMICAL ABSTRACTS.

See also A., Sept., 1901, **Adsorption of acids by wool** (PADDON). 1046, **Lignin** (FREUDENBERG and others; FUCHS and HORN).

Roofing felts. SHAW and others.—See IX.

PATENTS.

Production of artificial silk [from bagasse of sugar cane]. E. C. H. VALET and O. FUNK (B.P. 287,461, 19.3.28. Ger., 19.3.27).—Cellulose is obtained by the processes described in B.P. 277,163 and 287,516 (B., 1927, 839; 1929, 676), and converted into artificial silk by the usual methods.

B. P. RIDGE.

Manufacture of cellulose from vegetable fibres. A. G. POLLARD and J. R. WHINCOP (B.P. 315,902, 16.4.28).—Vegetable fibres are heated in a dilute alkaline solution, washed, drained, and, while still moist and slightly alkaline, treated with chlorine until the mass becomes acid. It is then washed with a dilute solution of a hydroxide or carbonate of an alkali metal or of ammonia.

A. G. POLLARD.

Spinning of artificial silk. I. G. FARBERIND. A.-G. (B.P. 286,292, 17.2.28. Ger., 4.3.27).—Threads of desired strength and extensibility, and of uniform dyeing capacity, are produced by the introduction of a tension device between the spinning nozzle and the receiving box, whereby the incompletely coagulated thread is submitted to a constant tension.

B. P. RIDGE.

Manufacture of artificial filaments or threads by the dry or evaporative method. BRIT. CELANESE, LTD., and H. DREYFUS (B.P. 315,729, 10.4.28. Addn. to B.P. 165,519; B., 1921, 577 A).—Solutions of cellulose derivatives (acetates etc.) are spun into an evaporating chamber in which they traverse a path of 3—6 yds. The threads so obtained are very regular in their physical characteristics, and loss of solvent by evaporation outside the chamber is prevented. Suitable apparatus is described.

B. P. RIDGE.

Treatment of cellulose and cellulose ester filaments. H. P. BASSETT (U.S.P. 1,722,171, 23.7.29. Appl., 1.9.26).—The cellulosic materials are treated with a 1% aqueous solution of an alkali salt of a weak acid.

F. G. CLARKE.

Preparing acetone-soluble, high-viscosity cellulose acetate. W. GRUBER, Assr. to DR. A. WACKER GES. F. ELECTROCHEM. IND. (U.S.P. 1,723,614, 6.8.29. Appl., 1.11.28. Ger., 9.1.26).—A solution of acetone-insoluble cellulose acetate is treated with an acid chloride of a metal of the group consisting of zinc, mercury, and copper.

H. ROYAL-DAWSON.

Manufacture of wood sugar. HOLZHYDROLYSE A.-G., Assees. of E. HÄGGLUND (B.P. 302,313, 13.12.28. Ger., 14.12.27).—Sugar obtained by the hydrolysis of

cellulose with hydrochloric acid, after the removal of the majority of the acid by evaporation, is freed from residual acid by wet-grinding with the oxide or hydroxide of an alkaline-earth metal, preferably lime. The saccharate is separated in a centrifuge wherein it may be washed, and is subsequently suspended in water, treated with carbon dioxide in the usual manner, and finally inverted. A. G. POLLARD.

Manufacture of paper from peat. C. C. ROTH (W. GRAHN, adtor.), Assr. to PEAT PRODUCTS CO. (U.S.P. 1,721,974, 23.7.29. Appl., 23.4.26).—Half-stuff (2.5 pts.), prepared by introducing rag clippings into a combined beater, grinder, and washing machine, is mixed with peat (7.5 pts.) which has been reduced to the fibrous condition. After draining off the surplus water and adding resin and alum the mixture is formed into sheets. F. G. CLARKE.

Thickening of [paper] pulp. A. J. and R. J. HAUG (U.S.P. 1,717,604, 18.6.29. Appl., 4.10.27).—The pulp is supplied to the interior of a horizontal, cylindrical straining surface which is rotated at centrifugal speed by a shaft attached to one closed end, and the accumulated material is removed therefrom by a minor quantity of fluid under pressure propelling it in an axial direction to the open end of the drum. B. M. VENABLES.

Paper-coating composition. A. RUDERMAN (U.S.P. 1,723,581, 6.8.29. Appl., 6.11.24).—A composition containing rubber latex, starch, sodium carbonate, a soluble metallic polysulphide, and formaldehyde is used to render paper water- and grease-proof. H. ROYAL-DAWSON.

Manufacture of textile materials. BRIT. CELANESE, LTD. From CELANESE CORP. OF AMERICA (B.P. 317,026, 9.5.28).

Spinning of artificial threads. I. G. FARBERIND. A.-G. (B.P. 287,553, 3.3.28. Ger., 24.3.27).

Spinning of artificial silk or the like [to obtain cross-winding]. C. J. HEALEY (B.P. 317,125, 11.5.28).

Acetone from acetylation liquor (B.P. 300,548).—See III.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Wetting-out agent for use in textile industries. HARDING CHEM. CO., LTD., W. H. CLUTTERBUCK, and A. WOOLLER (B.P. 316,356, 2.5.28).—Trichloroethylene (15–25%) is mixed with a well-sulphonated castor oil, together with cyclohexanol if desired, and the mixture is neutralised with sodium hydroxide. The product is applied particularly in cold wetting-out and bleaching baths containing hypochlorite. C. HOLLINS.

Dyeing animal fibres [with ice colours]. E. SCHWENK, K. REICHNER, and M. KNOB, Assrs. to VER. F. CHEM. U. MET. PROD. (U.S.P. 1,718,882, 25.6.29. Appl., 1.8.27. Ger., 18.1.26).—Wool, silk, feathers, etc. are padded with a coupling component containing a sulphonamide or sulphonylamide grouping, and treated with a diazo compound. Examples are: 4-nitro-*o*-toluidine \rightarrow 2 : 3-hydroxynaphthoic 3-*p*-

toluenesulphonamidoanilide (red); *o*-chloroaniline \rightarrow *p*-cresotic 3-*p*-toluenesulphonamidoanilide (yellow); *p*-toluidine \rightarrow *p*-toluidine \rightarrow 1 : 4-hydroxynaphthoic 3-*p*-toluenesulphonamidoanilide (red); *m*-nitroaniline \rightarrow 2-hydroxy-5 : 6 : 7 : 8-tetrahydro-3-naphthoic 3-*p*-toluenesulphonamidoanilide (yellow-brown); α -naphthylamine \rightarrow salicylic 4-*p*-toluenesulphonamidoanilide (brown); dianisidine \rightarrow 2 mols. of 2 : 3-hydroxynaphthoic 4-anilidosulphonyl-*o*-toluidide (coppered, blue); *m*-4-xylydine \rightarrow 2 : 3-hydroxynaphthoic 4-amidosulphonyl-*o*-toluidide (bluish-red); *o*-anisidine \rightarrow 5 : 6 : 7 : 8-tetrahydro-2-naphthol-3-sulphonanilide (yellow); *m*-chloroaniline \rightarrow dianilide of R- or G-acid (orange); 2 : 5-dichloroaniline \rightarrow β -naphthol-6-sulphonanilide (brown on chrome leather); 5-nitro-*o*-toluidine \rightarrow *p*-toluenesulphon- α -naphthylamide (yellow-brown); *p*-nitroaniline \rightarrow *p*-toluenesulphon-*m*-aminoanilide (red-brown); *o*-chloroaniline \rightarrow 1-(4-amidosulphonyl-*o*-tolyl)-3-methyl-5-pyrazolone (yellow); *p*-anisidine \rightarrow 1 : 5-dihydroxynaphthalene \leftarrow 2-amino-*p*-toluenesulphonamide (brown on hides). C. HOLLINS.

Dyeing of fabric. G. F. LLOYD, Assr. to SPECTRUM DYES PROPRIETARY, LTD. (U.S.P. 1,717,792, 18.6.29. Appl., 6.4.28. Austral., 6.2.28).—Dyed fabric is stripped to the desired degree by means of sodium hydrogen sulphite solution previously reduced with zinc. The stripping is easily controlled if the hyposulphite contains a little acetic acid. C. HOLLINS.

Colouring agent for fabrics. A. PICAREFF, Assr. to A. PICAREFF ART STUDIOS, INC. (U.S.P. 1,722,392, 30.7.29. Appl., 14.5.27).—A viscous substance is added to a solution of an acid dye in dilute acetic acid. C. HOLLINS.

Coloration of [cellulose ester or ether] fabrics. BRIT. CELANESE, LTD. (B.P. 293,022, 1.6.28. U.S., 30.6.27).—Fabrics of cellulose esters or ethers are dyed by means of a transfer, the material being preferably moistened with a softener (e.g., 25% aqueous acetone) which is a solvent for the dye. C. HOLLINS.

Cloth printing. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 314,761, 28.12.27).—Soaps are added to the thickening agents used to improve the evenness of the prints. Gums, resins, saponins, sulphonic acids, etc. may also be added. L. G. LAWRIE.

Preparation of effect threads. CHEM. FABR. VORM. SANDOZ, and H. LEEMANN. From A. RHEINER (B.P. 314,913, 2.3.28. Addn. to B.P. 280,493; B., 1928, 812).—In the process of the prior patent mercerised cotton is employed, and the temperature is kept above 40°. Alternatively, mercerised fibres are introduced into one or two ingredients of an acetylating mixture (acetic acid, acetic anhydride, and catalyst) and the remaining ingredients are added later, the conditions being so chosen that the activity of the acetylating mixture is considerably modified. L. G. LAWRIE.

Loading silk fabrics. R. CLAVEL (B.P. 300,894, 6.3.28. Ger., 19.11.27).—Natural or artificial silks are padded or printed with a metallic salt such as stannic chloride admixed with an acid or acid salt and thickening agent, the metallic salt being subsequently converted

into an insoluble form, *e.g.*, by treatment with an alkali phosphate. L. G. LAWRIE.

Dyeing of vegetable fibres. J. RATH, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,724,108—9, 13.8.29. Appl., [A] 17.2.27, [P] 2.3.28. Ger., 22.2.26).—See B.P. 266,387; B., 1928, 156.

Providing an embossed design upon cloth or like material. G. R. HUDSON (B.P. 317,578, 19.6., 21.9., and 1.11.28).

[Scorching] process for producing colours and patterns on textile fabrics [without using dyes]. M. GORALSKI and L. VEL L. MITLIN (B.P. 317,685, 13.11.28).

Printing with vat dyes (B.P. 291,768).—See IV. **Colouring horn** (B.P. 298,946).—See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Manufacture of nitric acid by the ammonia oxidation process. I. M. LEBINSON (J. Chem. Ind. Moscow, 1929, 6, 8—12).—To improve the yield and reduce the reaction space, operation under pressure is recommended. Malyrevski and Papkov's contention that pressure adversely affects the oxidation of ammonia is unfounded; the objection is that the reaction $3\text{HNO}_2 = \text{HNO}_3 + 2\text{NO} + \text{H}_2\text{O}$ is affected, affording nitric acid with a high content of nitrous acid, and that the formation of nitric oxide from nitrous acid is retarded. If the yield of nitric acid from nitric oxide is 93—94%, only 35—40% of the reaction space is required to obtain the first 70—75% of the nitric acid. Hence this proportion is absorbed as usual in nitric acid-sprayed towers, whilst the remainder is absorbed and converted into nitrogen trioxide in sulphuric acid-sprayed towers. The nitrogen trioxide is then decomposed with a current of hot nitric oxide, the gases (13.5% NO) then being sprayed with nitric acid and absorbed. The total yield of nitric acid should reach 98%, and the reaction space required is reduced to 25—30 m.² per ton of ammonia oxidised.

CHEMICAL ABSTRACTS.

Conversion of sodium chromate into dichromate. F. F. WOLF and L. J. POPOV (J. Chem. Ind. Moscow, 1929, 6, 12—17).—A cycle of industrial operations for the treatment of sodium chromate solution with ammonia and carbon dioxide at the ordinary pressure is described.

CHEMICAL ABSTRACTS.

Removal of ferrous chloride from solutions of commercial zinc chloride. N. N. EFREMOV and A. M. ROZENBERG (J. Chem. Ind. Moscow, 1928, 5, 1280—1282, 1431—1434).—The methods of oxidation examined were: treatment with a slow current of chlorine, with stirring, the chlorine then being removed by blowing in air or heating; treatment with bleaching powder, followed by zinc sulphate; heating with manganese dioxide in acid solution; boiling with potassium chlorate or nitric acid; treatment with hydrogen peroxide; electrolytic oxidation. The last-named method is preferred. The liquid must be well stirred, particularly in the anode zone, and must not contain an excess of acid.

CHEMICAL ABSTRACTS.

Preparation of crystalline lead iodide. E. TIGNOLA (Boll. Chim. farm., 1929, 68, 639—640).—Economy in this preparation is effected by the use of zinc iodide and lead acetate and crystallisation from a boiling solution of sodium acetate.

F. G. TRYHORN.

Manufacture of aluminium chloride from clays. I. E. ADADUROV (J. Chem. Ind. Moscow, 1928, 5, 1288—1292).—Since clays (45.6—52.3% SiO_2 , 33.07—39.5% Al_2O_3) which are most readily soluble in hydrochloric or sulphuric acid are those which have been heated at 600—800°, and since at 500—800° kaolinite does not decompose into free silica and alumina, but forms a new silicate which is soluble in acids, a knowledge of the kaolinite content of the clay is important. A method for its determination is described. The superiority of carbonyl chloride over chlorine for the production of aluminium chloride is ascribed to the activity of the chlorine released by dissociation at 800°. The reaction is catalysed by potassium chloride; carbon takes part in the reaction by reducing carbon dioxide to monoxide.

CHEMICAL ABSTRACTS.

Purification of gaseous chlorine. H. ECKSTEIN (Chem. Fabr., 1929, 335; cf. B., 1929, 392).—The author does not agree with Wasmuth (*loc. cit.*) that it is difficult to prepare pure chlorine by the manganese dioxide method provided the latter is free from nitrates. He uses an aluminium cooling vessel maintained at —50° with liquid air. Any traces of oxygen the chlorine may contain will not remain in solution at this temperature.

C. IRWIN.

Purification of gaseous chlorine. R. WASMUHT (Chem. Fabr., 1929, 335—336).—In a reply to Eckstein (preceding abstract), the author states that his method (the repeated liquefaction of cylinder chlorine) is more convenient, more easily controlled, and cheaper.

C. IRWIN.

Extraction of bromine from natural bromine waters. V. P. ILINSKI and V. M. FILIPEO (J. Chem. Ind. Moscow, 1928, 5, 833—838).—Alkaline water (I. Saki, Crimea) requires treatment with steam and twice the theoretical amount of chlorine; previous neutralisation is recommended.

CHEMICAL ABSTRACTS.

Extraction of bromine and iodine by solvents. III. **Iodine.** B. G. PANTELEIMONOV (J. Chem. Ind. Moscow, 1928, 5, 1220—1227).—The solvent power of kerosene for iodine decreases as iodine compounds accumulate in solution. Iodine forms with kerosene a compound insoluble in kerosene or water. With rise of temperature the quantity of hydrogen iodide formed decreases as the surface tension diminishes. The solvent power of kerosene for bromine becomes great only when bromine or chlorine compounds accumulate in the solution.

CHEMICAL ABSTRACTS.

Purity of sulphur monochloride. A. E. KRETОВ (J. Chem. Ind. Moscow, 1928, 5, 1268—1273).—Sulphur monochloride has d_{20}^{20} 1.6824; sulphur dichloride has d_{20}^{20} 1.6222. Density determinations serve for factory control. The purity of sulphur monochloride is ascertained by determination of chlorine and sulphur. A sealed tube containing the sample (0.2 g.) is broken in 2N-sodium hydroxide (50 c.c.) in a long-necked flask

and the mixture is heated on a water-bath for 4—4.5 hrs. (or 1.5 hrs. for chlorine only). After cooling, 30% hydrogen peroxide (3—4 c.c.) is gradually added, the mixture heated for 0.5 hr. (max.), slightly acidified with nitric acid, and diluted to 250 c.c. The chlorine is determined volumetrically and the sulphur gravimetrically in aliquot portions. **CHEMICAL ABSTRACTS.**

Manufacture of selenium. A. GARNAK (J. Chem. Ind. Moscow, 1928, 5, 1424—1426).—The sulphite, hydrogen sulphite, and oxidation (nitric acid, potassium chlorate) methods of obtaining selenium from the slimy deposits in sulphuric acid chambers are criticised; the potassium chlorate method has been modified. New methods employing consecutive treatment with sulphuric acid and nitric acid (giving the highest yields, 92—93%) and oxidation with sodium dichromate (yield 80%) are described. The most economical method employs manganese dioxide. Concentrated sulphuric acid (600 g.) is added gradually with energetic stirring to 1 kg. of the deposit (3.5% Se), and 140 g. of manganese dioxide are gradually introduced into the mixture at 90—95°. After 2.5—3 hrs. the selenious acid is extracted with water (3 litres). Hydrochloric acid (10 g.) and thio-sulphate (120 g.) are added to the united solutions, the selenium being coagulated by boiling, washed with hot water (200 c.c.), and dried at 50°. The yield is 90% of the theoretical. **CHEMICAL ABSTRACTS.**

See also A., Sept., 1900, **Adsorption of iodine, bromine, and some halides by carbon in different organic liquids** (TRIVIDIC). **Adsorption of sulphur dioxide by platinised asbestos and platinum-black** (SHEELS). 1003, **Molecularly dispersed silicic acid** (BRINTZINGER and TROEMER). 1010, **Hydrolysis of beryllium salts** (CUPR). 1024, **Beryllium chloride** (SCHMIDT). 1025, **Action of magnesium amalgam on nitrates etc** (NEOGI). **Preparation of fluorescent sulphides** (SHIROV). 1026, **Barium perchlorate** (SMITH). **Extraction of rare-earth residues and concentration of illinium** (BALL and HARRIS). 1027, **Reduction of dichromates to basic salts** (GRATHER and NAGAHAMA). 1029, **Determination of chloride ion in reagents** (FROST). **Potentiometric determination of bromine, octovalent and quadrivalent osmium in hydrobromic acid solutions** (CROWELL and KIRSCHMAN). **Determination of sulphuric acid in presence of chromic acid** (TARASENKOV). 1030, **Iodometric determination of a mixture of sulphide, sulphite, and thiosulphate** (WOLLAK). **Volumetric determination of polysulphides** (SZEBERÉNYI). **Determination of selenium dioxide in sulphuric acid** (RAIKHINSTEIN). **Electrometric titration of boric acid** (GREBENSCHTSCHIKOV and FAVORSKAIA). 1031, **Qualitative reagent for sodium** (CALEY). **Reagent for polysulphides** (FOLCINI).

Blanc fixe. ADADUROV.—See XIII. **Mercury salts as insecticides.** GLASGOW.—See XVI.

PATENTS.

Absorption of dilute nitrous gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 316,735, 13.6.28).—The dilute gases, prior to alkaline absorption, are treated with nitric acid under such conditions that

the nitric oxide is oxidised to nitrogen trioxide or peroxide. L. A. COLES.

Production of sodium hydrogen sulphide. H. P. BASSETT (U.S.P. 1,722,170, 23.7.29. Appl., 9.10.25).—A mixture of calcium sulphate, coal, and coke is heated, and the resulting reduced mass, comprising mainly calcium sulphide, is heated under pressure with less than the equivalent quantity of aqueous sodium sulphate. The sodium sulphide produced is leached from the mixture of coke and calcium sulphate, which is then used in the next reduction process. A part of the sodium sulphide solution is treated with sulphuric acid, and the resulting hydrogen sulphide passed into the remainder to give sodium hydrosulphide, the sodium sulphate being used for treatment of the reduced mass produced in the first step. F. G. CLARKE.

Manufacture of alkali cyanides. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 316,647, 2.5.28. Addn. to B.P. 301,565; B., 1929, 171).—The carbon monoxide used in the process of the prior patent is wholly or partly replaced by a hydrocarbon derivative containing oxygen which when heated above 400° yields carbon monoxide, e.g., methyl alcohol. L. A. COLES.

Obtaining pure hydroxides of the heavy metals. ACCUMULATOREN-FABR. A.-G. (B.P. 305,092, 6.12.28. Ger., 30.1.28).—A solution of a heavy-metal salt, e.g., nickel sulphate, free from foreign cations, is electrolysed using an anode consisting of the heavy metal or of a combination of it with a non-reacting material, and a smaller cathode working at a high cathodic current velocity so that hydrogen alone is liberated at it. L. A. COLES.

Treatment of metallic oxides or other compounds with hydrocarbons. H. WADE. From SOC. ITAL. PER LE IND. MINERARIE E CHIMICHE (B.P. 315,459, 13.4.28).—Preheated hydrocarbons are passed into a reaction chamber containing the material at 400—700°, the material is discharged and sent, if desired, first to a magnetic separator and then to a furnace, and the resultant hydrocarbon vapours are condensed, the residual gases being purified from hydrogen compounds of the metalloids etc. and returned to the reaction chamber. W. G. CAREY.

Manufacture of adsorbent silica and like gels. SILICA GEL CORP., Assees. of E. B. MILLER and G. C. CONNOLLY (B.P. 314,398, 12.3.28. U.S., 12.3.27).—The washed hydrogels prepared as in B.P. 287,066 (B., 1929, 680) are treated, prior to dehydration, with concentrated sulphuric acid, thereby raising their temperature to 52—135°. After gradual cooling, the hydrogel is washed and dehydrated. W. G. CAREY.

Manufacture of hard, highly porous, adsorbent gels. SILICA GEL CORP., Assees. of E. B. MILLER and G. C. CONNOLLY (B.P. 289,890, 3.5.28. U.S., 6.5.27).—An acid and a silicate, in suitable proportion and concentration to form a hydrosol, are mixed, with vigorous agitation, and silica gel "fines" are added during the formation of the gel or afterwards, allowing the sol to set with the "fines" suspended therein. The production of gels of alumina, oxides of tin, titanium, and tungsten is described. W. G. CAREY.

Preparation of sodium silicate solution. C. D. WOOD, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,719,038, 2.7.29. Appl., 26.5.27).—Successive batches of a mixture of sodium silicate glass and water are heated under pressure in a closed rotary cylinder until the glass is substantially completely dissolved, each batch of sodium silicate solution so produced being then discharged into a storage vessel. A. B. MANNING.

Preparation of base-exchange silicate. R. O. FRIEND and E. M. PARTRIDGE, Assrs. to PERMUTIT Co. (U.S.P. 1,720,074, 9.7.29. Appl., 29.10.25).—A batch of greensand is treated with sodium silicate and washed thoroughly; aluminium sulphate is then added in amount greater than that necessary to neutralise the remaining sodium silicate, so that a solution of acid character is produced. A. B. MANNING.

Manufacture of catalysts for production of methyl and higher alcohols. H. G. SMITH, R. G. FRANKLIN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 316,113, 20.4.28).—Metal oxide catalysts for production of methyl alcohol etc. are made by precipitating the metal or metals as carbonates and heating (zinc carbonate at 300–400°; zinc and chromium carbonates at 250–300°). The granules are then made into pellets. C. HOLLINS.

Mechanical dispersion of alkaline-earth and heavy-metal salts of the phosphoric and arsenic acids. A. CHWALA (B.P. 315,254, 10.4.28).—The dispersion is effected in a colloid mill etc. in presence of peptising agents consisting of alkali salts of the arsenic acids or phosphoric acids formed from the ortho-acids by splitting off water, and, if desired, in presence also of protective colloids such as sulphite-liquor. W. G. CAREY.

Production of [citrate-soluble] calcined phosphates. KALI-CHEMIE A.-G., Assocs. of RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 301,022, 8.11.28. Ger., 23.11.27).—A mixture of phosphorites, alkali carbonates or salts, or magnesium compounds, or mixtures of these with silica, is calcined, with or without steam, in such a manner that the bulk of the fluorine contained in the natural phosphate is driven out at temperatures below the fusion point (1000°) of the mixture, after which the decomposition is completed at higher temperatures. W. G. CAREY.

Manufacture of metal sulphates from sulphides and its application to gas purification. I. G. FARBERIND. A.-G. (B.P. 288,977, 13.4.28. Ger., 16.4.27).—A metal sulphide is precipitated from a metal salt by hydrogen sulphide, and while suspended in water is subjected above 120° to the action of oxygen under pressure. Gases may be purified from hydrogen sulphide and/or ammonia with the production of ammonium sulphate by treating a metal sulphate in aqueous ammoniacal solution with the gas to be purified, converting the precipitated sulphide into sulphate by the above process, and recovering ammonium sulphate. W. G. CAREY.

Manufacture of mercury salts. H. S. REID and A. H. MAUDE, Assrs. to CANADIAN ELECTRO PRODUCTS Co. (U.S.P. 1,721,188, 16.7.29. Appl., 18.4.21).—Mercury and a concentrated non-oxidising mineral acid

are brought together in the presence of nitric acid, the last-named being regenerated by supplying oxygen to the solution maintained at 20–80°. H. ROYAL-DAWSON.

Manufacture of desiccating material. II. WADE. From E. THOMAS (B.P. 315,582, 26.7.28. Addn. to B.P. 303,263; B., 1929, 207).—Magnesium perchlorate for use as described in the prior patent is produced by grinding together light magnesium oxide (or carbonate) and anhydrous ammonium perchlorate, for which purpose a ball mill may be used, and the product is afterwards heated. Claim is made for the presence of either reagent in excess. B. M. VENABLES.

Production of hydrogen. OXHYDRIQUE FRANÇ. (B.P. 294,150, 17.7.28. Fr., 18.7.27).—Silicon or ferro-silicon is introduced progressively and in adjustable amount into caustic alkali solution in a closed vessel under pressure by means of an adjustable distributor, e.g., a freely suspended tube having discharge ports at its lower end which fits into a standard gas cylinder pivotally supported to facilitate oscillation and mixing. W. G. CAREY.

Heating hydrogen or carbon monoxide or gases containing these substances under high pressure. K. GORDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 317,131, 12.5.28).—The gases under high pressure are brought into contact with a molten metal or alloy (e.g., lead) inert to the gases, baffle plates being arranged in the bath of metal to prevent the gases from coming into contact with the walls. W. G. CAREY.

[Electrolytic] manufacture of [per-]compounds containing active oxygen. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 316,648, 2.5.28).—Corrosion of the amalgamated cathodes is diminished by allowing mercury to flow over them, or by adding small quantities of a mercury salt to the cathodic electrolyte so that mercury is constantly deposited on the cathodes. (Cf. B.P. 290,750; B., 1928, 523.) L. A. COLES.

Treating [improving the colour of] sulphur. J. W. SCHWAB, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,723,815, 6.8.29. Appl., 10.12.26).—Treatment of the molten sulphur with finely-divided zeolites followed by their removal is claimed. H. ROYAL-DAWSON.

Burning of sulphur. E. J. MULLEN, Assr. to GEN. CHEM. Co. (U.S.P. 1,720,742, 16.7.29. Appl., 11.11.25).—Sulphur is vaporised and the vapour produced, freed from sulphur dioxide, is burned in heat-transfer relation to the sulphur being distilled, so that the heat of combustion of the vapour is used in distilling further quantities of sulphur, and pure sulphur dioxide is produced. H. ROYAL-DAWSON.

Separation of phosphorus from phosphorus [containing] vapours. M. MAYER (U.S.P. 1,721,868, 23.7.29. Appl., 20.10.25. Ger., 28.10.24).—The vapours are passed first through a tower against a counter-current of liquid, and then through an electrostatic separator. H. ROYAL-DAWSON.

Manufacture of calcium hypochlorite. L. MEL-LESH-JACKSON. From MATHIESON ALKALI WORKS (B.P. 317,233, 18.8.28).—See U.S.P. 1,713,668; B., 1929, 643.

Production of green hydrated chromium oxide. K. H. MEYER and H. KRZIKALLA, ASSTS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,723,556, 6.8.29. Appl., 8.3.28. Ger., 15.3.27).—See B.P. 304,809; B., 1929, 283.

Manufacture of gaseous mixtures of nitrogen and hydrogen [for ammonia synthesis]. E. EDWIN (U.S.P. 1,723,772, 6.8.29. Appl., 13.3.25. Norw., 28.9.22).—See F.P. 596, 714; B., 1926, 321.

Production of phosphorus and fusion cement from natural phosphates. W. KYBER (U.S.P. 1,723,791, 6.8.29. Appl., 16.7.26. Ger., 4.8.25).—See B.P. 256,622; B., 1927, 909.

Fireproof composition (U.S.P. 1,720,926). **Apparatus for transforming gases** (U.S.P. 1,719,610).—See I. Hydrogen chloride (B.P. 317,165).—See II. Catalytic hydrogenation (B.P. 306,803). [Catalysts for] catalytic oxidation (B.P. 295,270).—See III. Electrolytic cells (B.P. 316,694). Electrolysis of fused chlorides (B.P. 302,881).—See XI. Titanium pigments (B.P. 315,904).—See XIII. Fertilisers (B.P. 298,196, 302,148, 315,485, and 316,428). Soil fertilisation (B.P. 316,122).—See XVI. Baking powders (B.P. 304,230).—See XIX.

VIII.—GLASS; CERAMICS.

Corrosion of crucible in glass manufacture. M. WATATANI (Rep. Imp. Ind. Res. Inst. Osaka, 1928, 9, No. 6, 1—31).—Corrosion was greater with soda-lead than with potash-lead glass, and when the crucible was more strongly heated before pouring in the molten glass. Crucibles prepared with addition of feldspar show an increased resistance. Crucibles from German clay were more resistant than those from Japanese clay.

CHEMICAL ABSTRACTS.

Distribution of temperatures and block corrosion in glass tank furnaces. F. F. S. BRYSON (J. Soc. Glass Tech., 1929, 13, 140—161).—A series of thermocouples were inserted to definite depths into holes drilled in the blocks along the side wall of the furnace, and temperatures read over a period of time. The temperature distribution in the glass in contact with the wall, and the rate of block erosion along the wall, were deduced from the results.

A. COUSEN.

Durability of [glass] tank blocks. E. J. C. BOWMAKER (J. Soc. Glass Tech., 1929, 13, 130—140).—The relative resistance to corrosion and erosion of the blocks by molten glass was indicated by treatment with boiling hydrofluoric-sulphuric acid mixtures.

A. COUSEN.

Natural rocks in the manufacture of bottle glass. I. I. KITAIGORODSKI and S. V. RODIN (Trans. State Exp. Inst. Silicates, Moscow, 1928, 25, 62 pp.).—A batch containing trachyte 85, sand 5, and chalk 10% gave a satisfactory green bottle glass. The melting time is longer and the temperature higher than usual; the working range is short.

CHEMICAL ABSTRACTS.

Application of nepheline syenites of the Murman coast in the glass industry. V. V. VARGIN (Trans. Ceram. Res. Inst. Moscow, 1929, 15, 61 pp.).—Nepheline (with sand and lime) may be used in glass batches up to an amount giving glass containing less than 15%

Al_2O_3 . The amount of silica should be at least 58%, and of silica + alumina at least 70%. The addition of 0.3—0.5% of sulphate is advised.

CHEMICAL ABSTRACTS.

Relation of the composition of glass to its optical constants. III. T. TAKAMATSU (Rep. Imp. Ind. Res. Inst. Osaka, 1929, 9, No. 12).—In glasses having the composition $0.5x\text{Na}_2\text{O}, 0.5x\text{K}_2\text{O}, y\text{CaO}, 6\text{SiO}_2$, when the silica and lime are kept constant, n_D increases regularly with increase in the sum of the molecular concentrations of sodium and potassium oxides (0.0010 per 0.1 mol.); if this sum is less than 2.4 for each 6SiO_2 , the change of n_D is rectilinear. The increase in total dispersion is proportional to the same sum (average, 0.000059 per 0.1 mol.). The change of γ [$(n_D - 1)/(n_F - n_C)$] is inversely proportional to that sum (average 0.30 per 0.1 mol. increase). When the silica, soda, and potash are kept constant the change of n_D is regular; if the sum is less than 2.4 per 6SiO_2 the change of n_D is rectilinear (average increase 0.0027 per 0.1 mol. CaO). The average increase of total dispersion is 0.000066 per 0.1 mol. increase in lime. The change in γ is inversely proportional to the increase in lime (average, 0.16 per 0.1 mol.).

CHEMICAL ABSTRACTS.

Kilns and kiln firing. VI. **Fuel consumptions up to various times during the firing of a down-draught kiln used for large fireclay lumps.** S. R. HIND (Trans. Ceram. Soc., 1929, 28, 352—362; cf. B., 1929, 682).—A test was carried out on a down-draught kiln firing fireclay lumps to cone 13 to study the progressive relation between time, temperature, and fuel consumption. Full details are given of the construction of the kiln, temperatures, progress of firing, goods set, and fuel consumed. The formula $W = a\theta t / (1 - b\theta)$ is developed, in which W , θ , and t are fuel consumed, kiln temperature, and time from commencement of firing, respectively; a and b are constants. This expression is shown to agree closely with the facts. The fuel consumed to a given temperature is practically proportional to the time of firing, and it increases at a greater rate than the temperature when the time of firing is maintained constant and the temperature varied. The cooling curve is closely accounted for on accepted theoretical grounds, allowance being made for the variation with temperature of the physical constants of firebrick.

F. SALT.

Electrical conductivity of magnesite and some other refractory materials in relation to the temperature and their other properties. E. DIERSCHLAG and F. WULFESTIEG (Iron and Steel Inst., Sept., 1929. Advance copy. 24 pp.).—The electrical resistance of calcined magnesite falls rapidly with rising temperature, the fall becoming slower at high temperatures, though a constant curve does not exist due to the internal variability of the material, previously burned and unburned materials showing great differences in resistance. To obtain stable experimental conditions a minimum burning time of 26 hrs. at 1550° was required. Increase in pressure when manufacturing the specimens reduces resistance, due probably to reduction in the size of pores. Light oxides, e.g.,

magnesia, silica, alumina, and lime, have nearly straight-line curves above 1500°, whereas the curves for dark oxides such as those of iron (Fe_2O_3) and manganese (Mn_2O_4) drop suddenly at lower temperatures; the resistance curves of the light oxides are in the order given above. The addition of white oxides tends to lower the resistance of common magnesite.

C. A. KING.

Refractory formers for electric heating elements; their manufacture and use. P. COOPER (Trans. Ceram. Soc., 1929, 28, 333—337).—The service conditions under which electric heating elements have to function, certain problems and difficulties of manufacture, and progress made in recent years are discussed.

F. SALT.

Aluminium chloride from clays. ADADUROV.—See VII. **Heat-resisting steels.** SARJANT.—See X.

PATENTS.

[Sheet] glass. CHANCE BROS. & Co., LTD., and C. E. GOULD (B.P. 316,464, 8.9.28).—Glass transmitting at least 80% of the incident light, with little selective absorption in the visible region, but cutting off all waves of less than 3800 Å., contains, in addition to the usual ingredients, a small proportion of the oxides of chromium and vanadium.

A. COUSEN.

Non-splinterable glass. A. M. SARGINT, and METAL-ION DEVELOPMENT Co., LTD. (B.P. 316,797, 5.9.28).—The edges of the sheets are sealed by a mixture of a water-soluble silicate, together with a metallic oxide or salt capable of reacting to give an insoluble compound. Powdered metal is normally added as the inert filling material, and, in addition, a small amount of a basic lead compound may be incorporated. When set, treatment with calcium chloride serves to harden the seal.

A. COUSEN.

Means for joining together parts of fused quartz, silica, or the like. L. SAMPLE (B.P. 315,250, 10.4.28).—Parts to be united are provided with bevelled edges at the point of contact, and a groove following the line of fusion is cut in one or both of the parts. The bevelled edges are placed together, and the portion between the grooves is fused.

F. SALT.

Brick, pottery, and like tunnel ovens or kilns. P. J. and J. P. SPENGLER (B.P. 315,591, 4.8.28).—Parallel drying and firing tunnels are arranged side by side and, by means of flues, furnace gases from the firing tunnel are led in part back to this tunnel at a point near to the entrance of the goods, and in part under the drying tunnel. Means are provided for removing ashes from the grates without admitting air. A chamber with openings in roof and floor is arranged over the cooling zone of the firing tunnel. The furnace chambers in the firing tunnel are in two series, one generating reducing gas, the other oxidising gas.

F. SALT.

Refractory arch for furnaces and its production. E. G. BAILEY, Assr. to FULLER LEHIGH Co. (U.S.P. 1,719,416, 2.7.29. Appl., 17.1.22).—A series of water-circulating tubes is installed in the position in which the arch is required, and on them is deposited a layer of

refractory material derived from the solid residue carried by the gaseous products of combustion from the fire.

A. B. MANNING.

Use of dumortierite. J. A. JEFFREY and F. H. RIDDLE, Assrs. to CHAMPION PORCELAIN Co. (U.S.P. 1,723,174, 6.8.29. Appl., 26.3.27).—A batch for a ceramic material contains this mineral.

H. ROYAL-DAWSON.

Manufacture of silica glass articles. P. H. C. LA BURTHE, Assr. to QUARTZ & SILICE (U.S.P. 1,724,844, 13.8.29. Appl., 19.2.27. Fr., 24.2.26).—See B.P. 266,711; B., 1927, 557.

Preparation of vitrified material ["glass-granite"]. P. M. COCHUIS, Assr. to N. V. GLASFABR. "LEERDAM" VOORHEEN JEEKEL, MIJNSSEN, & Co. (U.S.P. 1,724,747, 13.8.29. Appl., 3.3.23. Holl., 4.8.22).—See B.P. 201,880; B., 1924, 598.

Manufacture of non-splintering glass sheets [compounded with cellulose derivatives]. G. E. HEYL (B.P. 317,240, 29.8.28. Addn. to B.P. 310,995).

Tunnel kilns (B.P. 315,540). Repair of furnace linings (B.P. 303,417). Heat exchanger (U.S.P. 1,721,442).—See I. Partitions for electrolytic products (B.P. 302,881).—See XI.

IX.—BUILDING MATERIALS.

Modern facing bricks. A. B. SEARLE (Trans. Ceram. Soc., 1929, 28, 339—349).—Methods of making the different kinds of facing bricks are critically reviewed.

F. SALT.

Experimental production of roofing-felts. M. B. SHAW, G. W. BICKING, and O. G. STREITER (Bur. Stand. J. Res., 1929, 2, 1001—1016).—The use of cheap substitutes for rags in the manufacture of asphalt-saturated and -coated roofing felts is investigated. The felts were made in a semi-commercial paper mill and converted into roofing in a commercial roofing mill. Relatively large amounts of substitute materials, such as old jute and manilla bags, old newspapers, and wood sawdust, can be used in admixture with rag stock without causing great difficulty in manufacturing processes. One satisfactory felt contained 60% of wood-fibre papers and 30% of sawdust. The substitute materials are cheaper and require less time and power in the paper-making process. Durability tests on the experimental roofing felts are being conducted. C. J. SMITHELLS.

Iron slag cement. MABEE.—See X.

PATENTS.

Slaking lime and producing hydrate of lime. W. H. BARTON, Assr. to BLUE DIAMOND Co. (U.S.P. 1,720,026, 9.7.29. Appl., 18.10.26).—Quicklime is admixed with water at or near its b.p., in proportions sufficient to produce a lime putty. A. B. MANNING.

[Quick-setting] hydraulic cement. C. PONTOPPIDAN, Assr. to F. L. SMIDT & Co. (U.S.P. 1,722,480, 30.7.29. Appl., 30.6.27).—Raw materials mixed in such proportions as to yield a product having a hydraulic modulus of not less than 2.20 and a silica modulus of less than 3.00, and ground so that at least 95% passes

200-mesh, are heated to sintering, and the clinker is ground so that at least 95% passes 200-mesh.

L. A. COLES.

Binding and hardening moulded articles. OESTERR.-AMERIKAN. MAGNESIT A.-G., and K. ERDMANN (B.P. 315,620, 8.9.28. Austr., 16.8.28).—Articles are moulded from a composition of fibrous material and Sorel cement and are dried by passing through them under pressure, first in one direction, then in the other, a hot gaseous medium, preferably containing carbon dioxide.

F. SALT.

Manufacture of moulded articles from fibrous materials. K. ERDMANN, Assee. of OESTERR.-AMERIKAN. MAGNESIT A.-G. (B.P. 315,628, 26.9.28. Austr., 16.8.28. Addn. to B.P. 258,731; B., 1927, 13).—The process of the prior patent is modified by the use of a gaseous drying medium heated at 220–450°, and preferably containing carbon dioxide, to which the articles are exposed on perforated moulds or open conveyors.

F. SALT.

Production of light [insulating] bricks. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,205, 5.4.28).—A clay slip is mixed with an organic foaming agent, such as soap solution, saponins, organic sulphonic acids, and the mixture is stirred into foam. Before, during, or after the foam has been produced, substances such as cement and water-insoluble salts of the alkaline-earth metals, which impart rigidity, and weakly acid or alkaline agents such as sodium carbonate, bisulphates, etc., which increase the fired strength of the product, are added. The foamy mass is moulded and fired in the usual way. Bricks produced by this process have a low apparent sp. gr. and high crushing strength.

F. SALT.

Manufacture of bituminous emulsions. G. C. HURRELL (B.P. 315,495, 25.4.28).—Bitumen emulsions stabilised with colloidal clay, e.g., bentonite, are produced by first preparing an unstable but highly dispersed emulsion of the bitumen in water by means of small proportions of protective colloids (sodium oleate, or casein and borax) in a high-speed mill (cf. B.P. 214,308; B., 1924, 542), and then mixing the emulsion with a fine suspension of the clay by mild agitation.

J. H. LANE.

Wood preservative. L. P. CURTIN, Assr. to WESTERN UNION TELEGRAPH CO. (U.S.P. 1,722,323, 30.7.29. Appl., 24.6.27).—Chlorine derivatives of coal-tar acids of higher mol. wt. than the cresols are employed; the toxicity and permanence of these are greater than those of creosote oil.

F. G. CLARKE.

Wood-preserving composition and fungicide. A. L. KLEES and R. P. SOULE, Assrs. to COMBUSTION UTILITIES CORP. (U.S.P. 1,720,905, 16.7.29. Appl., 23.1.24).—Use is made of a refined, dehydrated product comprising primary tar acids, of which at least 50% distils above 220°.

H. ROYAL-DAWSON.

Improvement of porous materials by impregnation. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 316,649, 2.5.28).—Wood, millboard, brick, stone, etc. is impregnated with bleached montan wax or with the products obtained by saponifying or esterifying the

wax, or with mixtures of these with bactericides or fungicides and/or paraffin wax, fats, oils, resins, etc.

L. A. COLES.

Manufacture and ornamentation of building bricks and clay or like products. C. S. JILLINGS and W. E. BERG (B.P. 300,582, 12.11.28. Can., 15.11.27).

[Cement gun for] repair of concrete or like structures, furnace linings, etc. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 317,595, 5.7.28).

Construction of roads or pavements. P. ASCROFT and H. E. G. RICHARDS (B.P. 316,108, 20.1.28).

Preparation and application of road-surfacing compositions. H. E. POTTS. From H. PLAUSON (B.P. 317,100, 10.2.28).

Production of facings, such as road surfaces and floorings, or other structures. M. DENIAU (B.P. 316,906, 4.4.28. Addn. to B.P. 282,039).

Treatment of oils, tars, pitches (B.P. 316,897).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Temperature measurements in Siemens-Martin furnaces. A. SCHACK (Arch. Eisenhüttenw., 1929—30, 3, 7—12; Stahl u. Eisen, 1929, 49, 1196—1197).—The temperature of various parts of the Siemens-Martin open-hearth furnace has been measured by means of an ordinary thermocouple, a glowing-wire pyrometer, and a tube pyrometer. The last-named comprises a silite tube containing a cylindrical magnesia mass in which a bare thermocouple is embedded. The flow of hot gases through the tube heats the magnesia and a correct reading is obtained which is usually 70–80° above that recorded by the ordinary protected thermocouple. Measurements on five furnaces gave temperatures of 1610° or less for the gases entering the flues, whilst the flame temperatures measured by an optical pyrometer varied from 1750° to 1850°. The temperature of the metal just above the hearth was about 1500°, and the surface temperature about 1600°.

A. R. POWELL.

Damping-down and restarting of blast furnaces. C. S. GILL (Iron and Steel Inst., Sept., 1929. Advance copy. 7 pp.).—The general principle in damping a blast furnace is to have the coke of such a size as to prevent spongy iron from reaching the melting zone, to drain the hearth thoroughly, and to prevent infiltration of air. When restarting, room should be made below the tuyères for molten material and the maximum heat to the hearth and tap hole obtained by first blowing through the tuyères nearest to the tap hole.

C. A. KING.

Repair of blast furnaces without drawing the fire. A. COUSIN (Rev. Mét., 26, 395—400).—The charge in the furnace is lowered to a point somewhat below the part to be repaired, the blast is stopped, a breach made about 2 m. above the level of the charge through which cold air can enter and cool the shaft, and the worn bricks are at once renewed. Means to prevent collapse of the shaft when the internal pressure due to charge is

removed are described. Such repairs can be effected in 10–12 days. M. E. NOTTAGE.

Gaseous reduction of iron ores. H. FLEISSNER and F. DÜRSCHMID (Berg- u. Hüttenm. Jahrb., 1926, 74, 42–57; Chem. Zentr., 1929, i, 1500).—The direct reduction of ferrous carbonate by hydrogen is not marked until the whole of the carbon dioxide has been removed, the favourable effect of hydrogen being due to its removal of carbon dioxide. With hydrogen, the reducibility of ferric oxide is the same as that of ferrosferric oxide, reaching 96.6% in favourable cases. With producer gas the degree of reduction is low, but reaches 96% when the carbon dioxide has been removed. With a gas containing CO_2 5–6, CO 35–37, H_2 44–44.7, CH_4 4.9–5.6%, the reduction is better than with pure hydrogen. The moisture content of the gas is not important. The effect of the presence of carbon dioxide in the reducing gas is considered. A. A. ELDRIDGE.

Hardness of vacuum-annealed crystals of iron. H. O'NEILL (Iron and Steel Inst., Sept., 1929. Advance copy. 27 pp.).—Iron prepared by decarburising mild steel and annealing it *in vacuo* has indentation hardness values independent of the grain size or the orientation in the case of single crystals. The Brinell hardness of normalised Armco iron was lowered considerably by vacuum annealing, and finer-grained recrystallised material was softer than the coarser-grained metal. Different faces of single crystals gave the same values as aggregates, though different scratch-hardness values were obtained in different directions on the crystal faces. The strain-hardening capacity of vacuum-annealed or cold-rolled iron increased slightly from -185° to $+100^\circ$, and a small increase in scratch hardness was indicated at about 120° . C. A. KING.

Crystal decomposition and α -veins [in mild steel]. E. AMMERMAN and H. KORNFELD (Stahl u. Eisen, 1929, 49, 1192–1195).—Decomposition of the crystal grains which is observed in cold-worked and annealed mild steel is shown to be the same phenomenon as the α -veining noted by Rawdon and Berghind (A., 1928, 939), and is no indication that the material has been worked in the region of blue-brittleness and subsequently annealed. The phenomenon is essentially one of internal slip and may be rendered visible by etching with a mixture of 40 c.c. of concentrated hydrochloric acid, 30 c.c. of water, 25 c.c. of alcohol, and 5 g. of copper chloride after a preliminary etch with 4% alcoholic picric acid. A. R. POWELL.

Pickling of mild steel sheets. P. BARDENHEUER and G. THANHEISER (Stahl u. Eisen, 1929, 49, 1185–1192).—The formation of blowholes in steel after pickling is shown to be due to absorption of hydrogen from the action of the acid on the metal. This absorption is the greater the thinner the sheet, the higher the temperature of the pickling bath, and the lower the acid concentration; it is less with hydrochloric than with sulphuric acid. The previous treatment of the metal influences to a considerable extent the amount of hydrogen absorbed. A high rolling temperature generally reduces the absorption as small internal cavities are thereby welded up; the lower part of an ingot usually behaves better than the upper part. A. R. POWELL.

Heat-resisting steels with special reference to their application in the glass industry. R. J. SARJANT (J. Soc. Glass Tech., 1929, 13, 167–182).—An account of the structure and properties of a series of steels developed by Hadfield and his colleagues and claimed to be suitable for various uses in the glass industry, such as for recuperator tubes and moulds. The most useful alloys are in two ranges, (1) 10–25% Cr, 7–10% Ni, 65–70% Fe, and (2) 10–15% Cr, 30–40% Ni, 50–60% Fe, with, in all cases, certain additions of other elements such as silicon, tungsten, or molybdenum. A. COUSEN.

Case-hardening of special steels with ammonia. J. P. WALSTED (Abst. Theses Mass. Inst. Tech., 1929, No. 4, 70–71).—The case-hardening of "nitralloy" by treatment with ammonia at 900–1000° F. has been investigated. Nitriding decreases the yield point and increases the maximum strength, whilst the elongation, reduction of area, and impact values are much reduced. The steel also becomes remarkably resistant to corrosion by salt water. The depth of the case increases as the nitriding temperature is raised to 1300° F., but at higher temperatures it becomes less again, the increase being, however, accompanied by a decrease in hardness. Little or no case is produced by ammonia treatment below 800° F. In the nitriding of special steels the iron is changed from the α to the γ state and dissolves the carbide, so that the surface layers do not lose carbon. If nitralloy which has previously been decarburised is nitrided, the case peels off. Nitriding results in the formation of iron nitride; the other metals present merely determine the quality of the case. Nitralloy completely nitrided has a critical point on heating at 1320° F., which is not depressed on cooling. Reheating the nitrided case at above the critical point causes it to become deeper and finer in the grain. Nitriding of steels containing large amounts of aluminium gives unsatisfactory cases. For protecting parts not to be nitrided, aluminium powder mixed with sodium silicate solution, or stannous oxide mixed with water, oil, or sodium silicate solution is recommended. R. CUTHILL.

Hardening of superhardened steel by magnetism. Lattice resonance hypothesis. E. G. HERBERT (Iron and Steel Inst., Sept., 1929. Advance copy. 17 pp.).—Steel which has been superhardened by the impact of hardened steel balls is hardened still further by annealing at 260°. The same degree of hardness has been attained by placing a specimen of superhardened steel between the poles of a powerful electromagnet, and turning it repeatedly through 90°, while applying light taps to the metal. It is considered that the space lattices disturbed by the "cloud-burst" treatment are so unstable that they tend, under the influence of a particular temperature or degree of magnetisation, to be reconstructed with the effect of increased hardness. C. A. KING.

Correlation of fatigue and overstress [in steel]. J. H. SMITH, C. A. CONNOR, and F. H. ARMSTRONG (Iron and Steel Inst., Sept., 1929. Advance copy. 29 pp.).—When a material is overstressed by a symmetrical range of periodic stress its stress-strain

loops below the yield point are closed and symmetrical, though if the overstress is not symmetrical the loops are also unsymmetrical. Overstressing increases the yield range and closed loops are possible with fatigued material, fatigue having similar action to overstressing on the yield ranges of a normal steel. C. A. KING.

High-elastic limit structural steels. J. A. JONES (Iron and Steel Inst., Sept., 1929. Advance copy. 14 pp.).—From the effect of carbon, manganese, silicon, and nickel on the properties of structural steels in the form of plate or flat bar it is concluded that the type containing 0.3% C, 1.3% Mn, and 0.9% Si gives the best tensile properties. Next in order is a steel with a higher manganese and lower silicon content, tending towards a limit of 1.6% Mn when silicon is less than 0.3%. At least 3% Ni is required to produce high-elastic limit steel, and the substitution of a smaller amount of chromium is suggested.

C. A. KING.

Reduction of shrinkage cavities [in metals] and vacuum melting. W. J. P. ROHN (Inst. Metals, Sept., 1929. Advance copy. 12 pp.).—Shrinkage cavities may be diminished if solidification of an ingot proceeds from the bottom upwards, and this control can best be effected with water-cooled copper moulds. Cavities are prevented entirely if the metal is melted, refined, and cooled in an electrically heated melting furnace in a crucible which also acts as an ingot mould.

C. A. KING.

Properties of locomotive firebox stays and plates. O. F. HUDSON, I. M. HERBERT, F. E. BALL, and E. H. BUCKNALL (Inst. Metals, Sept., 1929. Advance copy. 80 pp.).—Severe wastage of stayheads, whilst primarily due to oxidation of copper, is brought about by leakage of water between stays and plates. Leakage results from the fact that arsenical copper possesses poor elastic properties and a very small limit of proportionality which allows deformation and loosening of the stays. It has been shown that the presence of 0.47% of arsenic exerts little or no effect on the rate of oxidation (at 300–600°), this being influenced to a greater extent by the presence of small quantities of hydrochloric acid or sulphur dioxide in the furnace gases. Many foreign elements, *e.g.*, silver, iron, tin, manganese, and silicon, raise the softening temperature of copper to a greater extent than does arsenic, and mechanical tests indicate that alloys which are not greatly softened by annealing at 300° retain in a large measure their elasticity at this temperature.

C. A. KING.

Concentration of ores of Western Quebec. J. S. GODARD (Canada Dept. Mines, Ore Invest., 1927, 164–172).—A brief summary of recent work on the concentration of some copper and copper-zinc ores containing gold and silver by gravity, magnetic, and flotation methods.

A. R. POWELL.

Reports of investigations: [Canadian] ore dressing and metallurgical laboratories. C. S. PARSONS, A. K. ANDERSON, and J. S. GODARD (Canada Dept. Mines, Ore Invest., 1927, 4–122).—The results of cyanidation, amalgamation, gravity concentration, and flotation tests on 9 Canadian gold ores and 1 silver ore,

and of differential flotation tests on 8 complex ores containing lead, zinc, and copper in association with gold and silver, are recorded, and recommendations are made as to the best procedure for treating the ores.

A. R. POWELL.

Reports of investigations: [Canadian] non-metallic laboratory. R. K. CARNOCHAN and R. A. ROGERS (Canada Dept. Mines, Ore Invest., 1927, 123–137).—Accounts are given of work carried out on the grinding of calcite tailings from a lead mine, on the wet milling of asbestos rock, on the elimination of iron from sand and sandstone, and on the concentration of a garnet rock by means of jigs, tables, and Ullrich magnetic separators.

A. R. POWELL.

Reports of investigations: [Canadian] hydro-metallurgical laboratory. [Hydrometallurgical treatment of high-grade iron-copper sulphide concentrates.] R. J. TRAILL, W. R. MCCLELLAND, and J. D. JOHNSTON (Canada Dept. Mines, Ore Invest., 1927, 138–153).—The ferric chloride leaching process on pyritic ores which have been reduced to monosulphides by heating in a neutral atmosphere at 600–700° (*cf.* B., 1929, 132) has been applied to two high-grade copper-iron sulphide ores and the results are recorded in a series of tables. Precipitation of copper from the leach liquors is best effected by means of electrolytic iron produced in the later stages of the process; the cement copper is roasted, the oxide dissolved in sulphuric acid, and pure copper obtained by electrolysis. The ferrous chloride liquor freed from copper is purified by treatment with further quantities of iron sulphide to remove lead and zinc, and electrolysed at 70° with 1.5 volts, using a graphite anode and a monel metal cathode with a current density of 20 amp./ft.² The ferric chloride solution formed during electrolysis is returned to the leaching tanks.

A. R. POWELL.

Report of the [Canadian] chemical laboratory division. [Hydrometallurgical process for treatment of stibnite ore and recovery of metallic antimony; Eckel process for production of iron slag cement]. H. C. MABEE (Canada Dept. Mines, Ore Invest., 1927, 158–163).—A flotation concentrate containing 57.7% Sb and 0.89% As was leached with hot 8% sodium hydroxide to give a solution containing 51 g./litre of antimony. The filtered liquor was electrolysed at 2 volts at 23°, whereby antimony of 99.2% purity was obtained with a current efficiency of 99%. The Eckel process (B.P. 227,837; B., 1925, 762) for obtaining iron and a titaniferous cement from ilmenite gave no satisfactory products with a Canadian ilmenite concentrate containing 35–40% TiO₂, 50–55% Fe₂O₃, and 5% SiO₂.

A. R. POWELL.

Treatment of mixed [copper-zinc-lead] concentrates from [Canadian] base-metal sulphide ores. W. E. HARRIS (Canada Dept. Mines, Ore Invest., 1927, 154–157).—The concentrates are roasted in steam to reduce the formation of ferrites to a minimum and the product is leached with hot sulphuric acid to give a solution of zinc and copper sulphates, which is freed from iron and silica in the leaching tank and electrolysed to recover 60–70% of its copper content. The spent

electrolyte is neutralised with zinc oxide and the remaining copper precipitated with zinc dust. The filtrate is boiled with amalgamated zinc dust to remove nickel, cobalt, cadmium, and the last traces of copper, leaving a pure solution for zinc electrolysis. The results of some tests of this process on Canadian ores are tabulated.

A. R. POWELL.

Bearing bronzes with and without zinc. H. J. FRENCH and E. M. STAPLES (Bur. Stand. J. Res., 1929, 2, 1017—1038).—Copper-rich copper-tin-lead bearing bronzes were tested for wearing properties, hardness, notch-toughness, and deformation at 20—315°. Bronzes with less than 4% Sn are unsuitable for general bearing service, show low resistance to deformation, and wear rapidly in the absence of lubrication. Bronzes with less than 5% Pb are suitable only where lubrication is maintained. Bronzes with 5—15% Pb are best able to operate for short periods in the absence of lubrication. The effect of adding 4% Zn to these alloys is small, and is insignificant in comparison with the change in properties resulting from variation in foundry practice.

C. J. SMITHIELLS.

Bimetals. W. ROHN (Z. Metallk., 1929, 21, 259—264).

—The theory of the construction and use of bimetal strips for temperature regulation is discussed, examples are given of suitable alloys for use within various temperature ranges, and a laboratory testing apparatus is described and illustrated. Up to 150° combinations of nickel-iron alloys (*e.g.*, 27% Ni, 5% Mo, and 68% Fe) with invar give large bending effects which are linearly proportional to the temperature, and between 150° and 400° linear proportionality is shown by the combination of the alloy with 42% Ni, 5% Mo, and 53% Fe with an alloy of 42% Ni and 58% Fe. The amount of bending at any given temperature decreases with increasing thickness of the strip along a hyperbolic curve.

A. R. POWELL.

Pinholes in cast aluminium alloys. N. F. BUDGEN (Inst. Metals, Sept., 1929. Advance copy. 14 pp.).

—The various types of pinholes in the commonly used alloys of aluminium are due to gas evolution on solidification; this is particularly evident in any casting except when made from virgin metal. Gas firing of melting furnaces appears more likely to lead to pinholing than oil firing. Conditions of casting, *e.g.*, temperature, rate of solidification, influence the extent of pinholing, though turbulent pouring has little effect on pinholes. Slow cooling or chlorine treatment of the molten alloy reduces the tendency to form pinholes. Aluminium-silicon alloys behave somewhat differently and, providing the temperature of pouring is not above 700°, pinholes are usually absent in such alloys containing 10—15% Si.

C. A. KING.

Solubility of gases in pure aluminium and in an aluminium alloy. W. CLAUS [with S. BRIESEMEISTER and E. KALAEHNE] (Z. Metallk., 1929, 21, 268—270).—The porosity of aluminium and No. 12 aluminium alloy (8% Cu) after melting at 700° and 800° in various gases has been examined. Nitrogen, oxygen, carbon monoxide, carbon dioxide, and sulphur dioxide are without effect on either aluminium or the copper alloy. Hydrogen and

steam render both metals porous especially at 800°. Coal gas, methane, and ethylene are more readily adsorbed by the alloy than by pure aluminium.

A. R. POWELL.

Production of platinum concentrates from Transvaal ores. T. K. PRENTICE (J. Chem. Met. Min. Soc. S. Afr., 1929, 29, 269—281).—A record is given of numerous gravity and flotation concentration tests on the oxidised and sulphide platinum ores of the Transvaal. The platinum recovery by gravity methods from the oxidised ores was generally less than about 50%, but recoveries of over 80% have been effected by flotation of the Lydenburg sulphide ores in the form of concentrates assaying 4—5 oz./ton from ore containing 4—5 dwt./ton of platinum metals. The flotation agent used is a mixture of eucalyptus and kerosene oils in varying proportions according to the nature of the ore, and small quantities of potassium xanthate are added both to the rougher and cleaning cells. For clayey ores addition of sodium silicate effects flocculation of the colloidal particles and produces a cleaner concentrate. A flow sheet of a test on sulphide norite ore from a Lydenburg mine is given, together with tabulated results of several other tests.

A. R. POWELL.

Recrystallisation of electrolytic copper after hot rolling. W. TAFEL (Z. Metallk., 1929, 21, 265—267).

—The grain size of electrolytic copper after hot rolling to reductions in height of 2—70% at 300°, 500°, 700°, and 900° has been determined and the results have been plotted. The curves for temperatures of 500° and above are all characteristic hyperbolæ, the minimum deformation required to produce recrystallisation falling from 6% at 500° to 2.7% at 900°, and the maximum grain size increasing rapidly with the rolling temperature. After reductions of about 20% the grain size is practically unaffected by more severe work, but increases slightly with the temperature. At 700° and 900° under conditions inducing rapid grain growth a large amount of twinning takes place.

A. R. POWELL.

Cadmium plating. O. H. LOVEN (Metal Ind., 1929, 27, 224—227).—Conditions leading to the production of a good cadmium plate, and the acetic acid-peroxide test of the resistance of the plate, are described.

CHEMICAL ABSTRACTS.

Spotting of plated or finished metals. W. P. BARROWS (Bur. Stand. J. Res., 1929, 2, 1085—1116).—The causes of "spotting-out" on plated metals are investigated. Two kinds are identified. "Crystal spots" appear only on metals which have been given an oxidised finish by treatment with sulphides. They consist of a dendritic growth of cuprous sulphide formed between the sulphide layer on the metal and the lacquer film. Their formation is induced by minute traces of sulphur or hydrogen sulphide which may arise from wrapping paper, cartons, or rubber bands. The application of a thin film of grease to the lacquered surface and the use of waxed wrapping paper are recommended. "Stain spots" appear on plain or lacquered cast metals, and are caused by the absorption in the pores of the metal of acid or alkaline compounds. These cannot be removed by washing, and articles should be exposed to

a humid atmosphere before final finishing, or covered with a lacquer of the phenol-condensation type, which retards spotting more than nitrocellulose lacquers.

C. J. SMITHELLS.

See also A., Sept., 995, Copper-aluminium alloys (MORLET). Copper-beryllium alloys (MASING and DAHL). 996, Iron-beryllium alloys (VON AUWERS). Beryllium-copper alloys (MASING and DAHL; DAHL and others). Aluminium-silicon system (KÖSTER). Silver-germanium (BRIGGS and others). 1003, Preparation etc. of colloidal lead (KEELAN and others). Colloidal gold (DOMANICKI). 1012, System gold-mercury (PLAKSIN). Iron-vanadium system (OYA). 1015, Deposition of cadmium (PINES). 1021, Formation of very thin electrolytic nickel films (OESTERLE). 1024, Preparation of beryllium (ILLIG and HOSENFELD; SCHMIDT). 1031, Analytical chemistry of beryllium (FISCHER). Spectroscopic detection of lead in gold-copper-silver alloys (GERLACH and SCHWEITZER). Sensitive reagent for copper (FOLCINI). 1031—2, Volumetric determination of mercury (DUNNICLIFF; COLOMBIER). 1032, Volumetric determination of manganese (LANG and KURTZ). Determination of manganese and arsenic (REINITZER and HOFFMANN). Determination of cobalt (CUVELIER). Detection of molybdenum (TANANAEV and PANTSCHENKO). 1033, Separation of bismuth from lead (FRICK and ENGEMANN). Separation of bismuth by electrolysis (JILEK and LUKAS). Micro-determination of bismuth (DUMONT and BOUILLENNE). Spectro-determination of iridium, rhodium, and palladium in platinum (GERLACH and SCHWEITZER).

Electric furnaces. TAMA.—See XI.

PATENTS.

Mechanical [multi-hearth ore-roasting] kilns. BALZ-ERZRÜSTUNG GES.M.B.H., and G. BALZ (B.P. 315,477, 14.4.28. Addn. to B.P. 288,823; B., 1929, 451).—In a roaster of the type described in the prior patent the roasting gases are deflected downwards on to the material by rings or segments of heat-, gas-, and acid-proof material (e.g., firebrick) depending from the roofs. A method of construction of a combustion chamber for the fire beneath the kiln is described involving bricks laid without mortar, the binder being loose sand or clay.

B. M. VENABLES.

Ore-roasting retort furnace. R. A. BENNETT (U.S.P. 1,723,006, 6.8.29. Appl., 5.3.28).—A bank of horizontal retorts is provided with a hood at one end having a hollow partition below the floor of the uppermost retort. A V-shaped hopper, capable of being moved on a pivot, normally closes an outlet in the partition.

C. A. KING.

Treatment of oxide iron ores. W. S. MILLAR (B.P. 316,404, 3.7.28).—The ore mixed with pyrites or other sulphide material is heated in chlorine to obtain ferrous chloride and sulphur dioxide. The product is leached with the minimum of hot water, and cooled to obtain ferrous chloride. This is redissolved in water and electrolysed for iron and chlorine for further use. Alternatively, the chlorinated product may be further

heated with excess of chlorine to volatilise ferric chloride, which is roasted in air to obtain pure ferric oxide and chlorine.

A. R. POWELL.

Decarburising ferro-alloys. H. E. POTTS. From ELECTRO METALLURGICAL CO. (B.P. 316,329, 27.3.28). Ferrochromium, ferromanganese, or ferrotungsten having a ratio of alloying element to carbon of less than 30 is blown in a converter with side-blast using air enriched to 48% O until the ratio exceeds 30. To obtain an alloy with a much lower carbon content without serious loss of alloying element the blast may contain 60–90% O, and the blowing may be finished with hydrogen, which is introduced below the surface of the molten metal. In the case of chromium alloys a final short blow with oxygen must be given to remove adsorbed hydrogen.

A. R. POWELL.

Acid-resisting iron alloys. L. KLÜGER, and OESTERR. SCHMIDTSTAHLWERKE A.-G. (B.P. 316,164, 20.4.28).—Alloys containing 28–40% Fe, 1–1.5% C, and molybdenum, nickel, and chromium substantially in the ratio 7.9:53.5:38.6 are claimed. Part or all of the nickel may be replaced by copper, and small quantities of vanadium, titanium, or aluminium may be added as deoxidisers.

A. R. POWELL.

Metal articles for use in chemical and like processes and alloys therefor. W. H. HATFIELD and H. GREEN (B.P. 316,394, 27.6.28).—A malleable steel of the non-rusting type comprises 10–20% Cr, 6–14% Ni, 0.3–1.5% W, and 0–0.25% C. The preferred composition is 18% Cr, 8% Ni, 0.6% W, and 0.12% C.

A. R. POWELL.

Alloy steel. F. C. LANGENBERG (U.S.P. 1,723,868, 6.8.29. Appl., 24.1.27).—A steel contains about 3% Mn, up to 1.25% Mo, and 0.1–0.5% Zr.

C. A. KING.

Steel [for car wheels]. W. C. HAMILTON and C. E. SIMS, ASSTS. to AMER. STEEL FOUNDRIES (U.S.P. 1,721,555, 23.7.29. Appl., 3.12.27).—A high-manganese pearlitic steel containing 0.5–2% Cr is used.

H. ROYAL-DAWSON.

Molybdenum nitriding steels. H. A. DE FRIES, ASSR. to LUDLUM STEEL CO. (U.S.P. 1,732,015, 6.8.29. Appl., 2.3.28).—A steel, free from aluminium and having a nitride-hardened surface, contains 1–9% Cr, 0.5–3.5% Si, 0.15–2% Mo, and 0.1–1% C.

H. ROYAL-DAWSON.

Manufacture of finely-divided metals. I. G. FARBENIND. A.-G. (B.P. 293,749, 28.6.28. Ger., 11.7.27).—Finely-divided copper, nickel, or cobalt on kieselguhr is obtained by reducing the corresponding carbonate or hydroxide precipitated on kieselguhr in the presence of a small quantity of finely-divided nickel with hydrogen under 30–40 atm. pressure at 60–100°.

A. R. POWELL.

[Lead] alloys and their manufacture. K. S. SELJESAETER, ASSR. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,722,358, 30.7.29. Appl., 6.7.27).—A ternary alloy is claimed containing up to 4% Sb, up to 1% As, the remainder being lead.

H. ROYAL-DAWSON.

Lead bearing metals containing nickel and copper. E. ABEL (B.P. 299,773, 18.10.28. Austr.,

31.10.27).—Lead bearing metals containing tin and antimony are treated with 1.5% of copper and 1% of nickel so that the latter is retained in the β -form.

A. R. POWELL.

Manufacture of bearing metal. ELECTROLYTIC ZINC CO. OF AUSTRALASIA, LTD. (B.P. 295,991, 8.8.28. Austral., 22.8.27).—Bearing metal containing 95–97% Cd, 5–3% Cu, with the addition of up to 0.5% Mg is claimed.

A. R. POWELL.

Manufacture of magnesium alloy. E. C. BURDICK, Assr. to DOW CHEM. CO. (U.S.P. 1,721,768, 23.7.29. Appl., 9.12.24).—Magnesium is melted with a relatively small amount of cupromanganese having a Cu : Mn ratio of 7 : 3 (approx.).

H. ROYAL-DAWSON.

Manufacture of tungsten alloys. O. L. MILLS (U.S.P. 1,721,966, 23.7.29. Appl., 11.1.29).—A mixture of a compound containing 3–20% of tungsten oxide, together with metallic tungsten and tungsten carbide, is fused by means of a carbon electric arc, and the waste tungsten carbide formed is oxidised to form part of a succeeding mixture.

C. A. KING.

[Hard] alloys [containing carbonitrides]. R. WALTER (B.P. 316,702, 11.5.28).—Alloys for cutting tools contain a proportion of tungsten or chromium carbonitride; e.g., 10% of tungsten carbonitride is added to an alloy containing 50% Co, 20% W, 28% Cr, and 2% C. An extremely hard alloy comprises 2% CrC₂, CrN, 5% of tungsten carbonitride, 8% Co, 5% W, and 80% WC.

A. R. POWELL.

Recutting or sharpening tools having numerous fine cutting edges. W. R. ROBE, Assr. to REKEEN TOOL CO., INC. (U.S.P. 1,721,414, 16.7.29. Appl., 2.8.27).—Such tools are sharpened by passing an electric current from an electrode through an electrolyte containing copper sulphate, sulphuric acid, borax, and acetic acid, to the tools.

C. A. KING.

Production of tarnish-resisting silver and silver plate. D. GRAY, R. O. BAILEY, and W. S. MURRAY, Assrs. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,720,215–6, 9.7.29. Appl., 5.11.23. Renewed [A] 27.9.28, [B] 26.7.27).—(A) The silver or silver plate is exposed to mercury vapour in an atmosphere free from oxygen or other substances which would interfere with the absorption of the mercury by the silver. (B) Articles are plated with an intimate mixture of silver and mercury, containing over 90% Ag.

A. B. MANNING.

Production of tarnish-resisting silver alloy etc. D. GRAY, R. O. BAILEY, and W. S. MURRAY, Assrs. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,720,894, 16.7.29. Appl., 3.4.24).—A silver alloy or intimate mixture containing at least 90% Ag and sufficient silicon substantially to prevent tarnishing is claimed.

S. S. WOOLF.

Treatment of ores, metallurgical products, residues, etc. for the recovery of precious metals. A. R. POWELL, E. C. DEERING, and JOHNSON, MATTHEY, & CO., LTD. (B.P. 316,063, 1.5.28).—Ores containing the platinum metals in association with sulphide minerals of copper, nickel, and iron are smelted in a blast furnace to obtain the values in the form of a matte containing a deficiency of sulphur over that required to form the

sulphides Cu₂S, FeS, and Ni₂S₃. The resulting matte is melted in a reverberatory with 5–15% of its weight of an alkali hydroxide or carbonate, whereby a disintegrating matte containing coarse crystals of an alloy of nickel, iron, and the platinum metals is obtained. The matte is roughly broken and allowed to disintegrate under water; the material is then ground wet in a ball-mill and passed over shaking tables and/or magnetic concentrators to remove the nickel-iron alloy, which is smelted with sodium sulphate and silicious material to obtain a high-grade nickel matte containing the platinum metals and a soda-ferrous oxide silicate slag. The nickel matte is ground and dissolved by boiling with hydrochloric or sulphuric acid, whereby the precious metals are recovered in the form of a sludge which can be refined by the ordinary methods. The method is also applicable to ores free from sulphides if these are smelted with matte-forming materials containing iron and/or nickel.

A. R. POWELL.

Chromium plating. H. E. POTTS. From TERNSTEDT MANUF. CO. (B.P. 292,094, 11.5.28).—An aqueous solution of chromic chloride and a number of alkali or alkaline-earth chlorides in greater concentration than that obtained by saturation with one such chloride is employed as electrolyte so that a relatively high current density exceeding 150 amp. per sq. ft. of cathode surface, e.g., 250–1000 amp., may be used. Thus the electrolyte may contain, per litre, 300 g. of hydrated chromic chloride (in the violet modification), 250 g. of potassium chloride, 250 g. of sodium chloride, and 150 g. of ammonium chloride; the amounts may be increased to the (cold) saturation values. If desired, about 1% of nitric acid, 75–100 g. per litre of oxalic acid as a reducing agent, and 3–5 g. of sodium fluoride per litre may be added.

J. S. G. THOMAS.

Cadmium plating. E. C. R. MARKS. From GRASSELLI CHEM. CO. (B.P. 315,943, 12.5.28).—A cyanide plating bath with the ingredients claimed in U.S.P. 1,681,509 (B., 1928, 759) may also contain a sulphonated vegetable oil; the nickel may be replaced by cobalt.

Alloy for electrical conductors. M. G. CORSON, formerly KORSUNSKY, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,723,867, 6.8.29. Appl., 9.12.24).—An alloy of copper containing 0.5–3% Cr is annealed at 400–700° for such a time as to increase the conductivity of the cold alloy at least 75% without reducing the tensile strength below 70,000 lb./in.²

C. A. KING.

Manufacture of wires and tapes for loading telephone conductors. W. S. SMITH and H. J. GARNETT (U.S.P. 1,723,215, 6.8.29. Appl., 17.2.26. U.K., 17.2.25).—The carbon-free wires and tapes contain 30–33% Ni, 58–65% Fe, and 2–6% Cu.

H. ROYAL-DAWSON.

Improving the character of grey iron castings by graphitisation. MEEHANITE METAL CORP., Asses. of A. F. MEEHAN (B.P. 292,164, 7.6.28. U.S., 16.7.27).—See U.S.P. 1,683,086; B., 1928, 820.

Treatment of [tinned] scrap iron. A. DOSSMANN (U.S.P. 1,723,411, 6.8.29. Appl., 4.8.27. Italy, 10.6.27).—See B.P. 298,103; B., 1929, 59.

Manufacture of zinc. H. E. COLEY (U.S.P. 1,721,373,

16.7.29. Appl., 8.8.27. U.K., 2.2.27).—See B.P. 281,129; B., 1928, 96.

Production of aluminium castings and moulds for use therein. E. STRASSER, Assr. to M. DIETIKER (U.S.P. 1,724,624, 13.8.29. Appl., 7.12.25. Ger., 13.12.24).—See B.P. 244,441; B., 1926, 547.

Manufacture of drawn tungsten wires. R. JACOBY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,723,862, 6.8.29. Appl., 16.6.21. Ger., 7.5.20).—See B.P. 163,014; B., 1922, 764 A.

Coating and colouring metal articles. A. PACZ (U.S.P. 1,723,067, 6.8.29. Appl., 16.4.26).—See B.P. 302,943; B., 1929, 215.

Method and apparatus for welding. D'A. M. CLARK. From BENDIX BRAKE Co. (B.P. 317,235, 21.8.28).

Tunnel kilns (B.P. 315,540). Repair of furnace refractories (B.P. 303,417).—See I. Flotation oil (U.S.P. 1,722,528).—See II. Treatment of oxides (B.P. 315,459).—See VII. Accumulator plates (B.P. 316,433).—See XI. White lead (B.P. 291,079).—See XIII.

XI.—ELECTROTECHNICS.

Progress in electric furnaces for non-ferrous metals. M. TAMA (Inst. Metals, Sept., 1929. Advance copy. 8 pp.).—Economy in induction furnace working lies in the suitable choice of size of unit, large furnaces having a relatively smaller current consumption. A greater latitude of melting different alloys, *e.g.*, phosphor-bronze, or copper-nickel alloys, is obtained by fritting the granular refractory material in a metallic mould within the furnace to form the lining. Examples of annealing furnaces for wide brass strip etc. are given.

C. A. KING.

New type of [voltaic] pile. O. SCARPA (Giorn. Chim. Ind. Appl., 1929, 11, 307—309, and Atti R. Accad. Lincei, 1929, [vi], 9, 1007—1014).—Wholly metallic piles have been constructed of the types copper|mercury|zinc|copper, copper|mercury|cadmium|copper, copper|zinc amalgam c_1 |zinc amalgam c_2 |copper, platinum|zinc amalgam c_1 |zinc amalgam c_2 |platinum; in the two last-named types of concentration pile cadmium may be substituted for zinc. The *E.M.F.* developed at 15° by the former piles is of the order 0.7×10^{-6} to 1.0×10^{-6} volts, and that of the concentration piles is roughly proportional to the difference in concentration of the amalgams. The energy giving rise to the *E.M.F.* arises in the case of the metallic piles from intermetallic dissolution or the formation of intermetallic compounds.

F. G. TRYHORN.

See also A., Sept., 996, **Magnetic iron-beryllium alloys** (VON AUWERS). 1004, **Electrification of smoke particles** (PATTERSON and others). 1015, **Lead dioxide-lead sulphate electrode** (VOSBURGH and CRAIG). **Deposition of cadmium** (PINES). 1021, **Formation of very thin electrolytic nickel films** (OESTERLE). **Preparation of iodoform** (VYSKOČIL). 1033, **Separation of bismuth by electrolysis** (JILEK and LUKAS).

Conductivity of refractories. DIEPSCHLAG and WULFESTIEG. **Refractory formers for heating**

elements. COOPER.—See VIII. **Superhardened steel.** HERBERT. **Treatment of sulphide concentrates.** TRAILL and others. **Treatment of stibnite ore.** MABEE. **Cadmium plating.** LOVEN.—See X.

PATENTS.

[Electric] furnace. I. B. SMITH, Assr. to LEEDS & NORTHRUP Co. (U.S.P. 1,721,840, 23.7.29. Appl., 2.3.28).—A heating chamber is provided with an inner container, with heating means, *e.g.*, electric resistances, between the two, and air is circulated through the container and the heated space around its exterior.

F. G. CLARKE.

Magnetic-core induction furnaces. P. E. BUNET, and ACIÉRIES DE GENNEVILLIERS (B.P. 291,455—6, 1.6.28. Fr., 2.6.27).—(A) A secondary circuit formed by the whole or part of the charge to be heated is followed as closely as possible by the primary circuit composed of a very small number of turns, *e.g.*, two, and fed at low voltage. The two circuits are arranged around one of the branches of a closed magnetic circuit, the other branch being used for a first transformation of the supply voltage down to the low tension of the primary circuit. (B) Magnetic iron cores, designed to guide the magnetic flux and reduce the reluctance of the magnetic circuit substantially to that of the air in the part to be heated, are arranged outside the primary circuit surrounding the furnace crucible.

J. S. G. THOMAS.

[Self-baking] electrode for electric furnaces. M. O. SEM, Assr. to NORSKE A./S. F. ELEKTROKEM. IND. OF NORWAY (U.S.P. 1,723,582, 6.8.29. Appl., 7.4.27. Nor., 7.4.26).—A raw electrode mass has a casing formed of pre-baked carbon inserts arranged in abutting relation at the periphery of the electrode.

J. S. G. THOMAS.

Primary cell. D. O. WALDEN, Assr. to NAT. CARBON Co., Inc. (U.S.P. 1,720,808, 16.7.29. Appl., 16.6.25).—A mixture of dry caustic alkali and oil is used with a zinc electrode coated with a material which prevents the oily particles adhering to it during the preparation of the cell.

II. ROYAL-DAWSON.

[Bipolar] depolarising plate for galvanic cells. SIEMENS & HALSKE A.-G., Assees. of K. SCHENKEL (B.P. 300,640, 3.11.28. Ger., 17.11.27).—A graphite mass is used for effecting electrical connexion, and a filling material for obtaining mechanical strength of the pressed body and for electrolytic separation of individual cells.

J. S. G. THOMAS.

Manufacture of accumulator plates. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 316,433, 24.7.28).—An alloy of lead with an alkali metal or alkaline-earth metal is further alloyed with one or more other metals so that the m.p. of the final alloy is only slightly above or below that of the lead. Thus, the alloy may contain 80.5% Pb, 17.5% Na, and 2% K, or alternatively, 19% Na and 2% Zn.

J. S. G. THOMAS.

Manufacture of storage battery plates. FURUKAWA DENKI KOGYO KABUSHIKI KAISHA (B.P. 304,231, 3.1.29. Jap., 17.1.28).—Plate grids are filled with a mixture composed of lead ash, produced by adding

lead oxides to molten lead or by stirring molten lead exposed to air, and a dilute solution of sulphuric acid.

J. S. G. THOMAS.

[Plastic mass for] electric accumulator [plates]. H. LEITNER (B.P. 316,328, 27.3.28).—Powdered lead monoxide is worked into a stiff paste with an aqueous solution of glycerin together with the sulphate of magnesium, sodium, or potassium.

J. S. G. THOMAS.

Secondary [alkaline] electric batteries. V. HEROLD (B.P. 317,130, 12.5.28).—A positive electrode consisting of alternate layers of powdered nickel oxide and flakes of nickel, and a negative electrode of powdered cadmium or cadmium hydroxide, are arranged in an electrolyte composed of potassium hydroxide solution (*d* 1.2).

J. S. G. THOMAS.

Liquid for accumulators. G. SCHMAUS (U.S.P. 1,722,343, 30.7.29. Appl., 20.8.28. Czechoslov., 29.9.27).—A mixture of sulphuric acid with ammonium and aluminium salts and a basic acetate is used as the filler prior to charging.

H. ROYAL-DAWSON.

Electrolytic cell. I. A. SHULIMSON, Assr. to AMER. BOSCH MAGNETO CORP. (U.S.P. 1,721,886, 23.7.29. Appl., 20.9.26).—In the cell 10–20% of a neutralised solution containing monobasic ammonium phosphate and 0.1–5% of potassium dichromate is used as electrolyte, and the cathode is of aluminium.

F. G. CLARKE.

Electrolytic cell and solutions therefor. W. H. GRIMDITCH, Assr. to PHILADELPHIA STORAGE BATTERY Co. (U.S.P. 1,723,154–5, 6.8.29. Appl., [A] 18.3.25, [B] 14.11.25).—(A) An asymmetric cell comprises an aluminium rectifying electrode and an electrode of an iron alloy containing at least 10% Si immersed in an electrolyte containing monobasic potassium phosphate, dibasic ammonium phosphate, and malic acid. (B) An electrolyte for rectifiers, condensers, etc. is composed of a phosphate of an alkali metal or radical, an organic acid, and a substance having a benzoate radical.

J. S. G. THOMAS.

Amalgam cells for electrolysis [of alkali or alkaline-earth chlorides]. A CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 316,694, 8.5.28).—An anode forming the top and side walls of the cell and extending to within 15 mm. of the surface of the mercury has open borings or channels for collecting and withdrawing evolved gas.

J. S. G. THOMAS.

Partitions for separating electrolytic products in the fusion electrolysis of chlorides, particularly of magnesium. I. G. FARBENIND. A.-G. (B.P. 302,881, 27.4.28. Ger., 23.12.27).—A plate of ordinary porous refractory material faced with dense ceramic material, *e.g.*, porcelain, is employed.

J. S. G. THOMAS.

Manufacture of photo-electric cells. GEN. ELECTRIC Co., LTD., and N. R. CAMPBELL (B.P. 317,209, 25.7.28).—The surface of the auxiliary electrode(s) is mainly or entirely composed of material, *e.g.*, carbon, that does not readily acquire photo-electric sensitivity from the metal of the cathode.

J. S. G. THOMAS.

Manufacture of oxide-coated cathodes for use in vacuum electric devices. OCTRON, LTD., W. HOLT, and G. H. STEDMAN (B.P. 316,015, 28.7.28).—A wire or

filament is coated with a thick suspension of barium or strontium carbonate (or oxalate), dried, dipped in a soluble salt, *e.g.*, nitrate, of the same metal, and heated in an atmosphere of carbon dioxide to convert the soluble salt into an insoluble compound.

C. A. KING.

Thermionic cathodes of high emissivity. SIEMENS & HALSKE A.-G. (B.P. 291,785, 25.4.28. Ger., 10.6.27. Addn. to B.P. 289,381).—A flux, *e.g.*, phosphorus or boron compounds (especially borax), is added to the thorium nitrate employed in accordance with the prior patent.

J. S. G. THOMAS.

Production of [silent] electric discharges at elevated temperatures. C. EPNER (B.P. 296,019, 8.6.28. Ger., 23.8.27).—The conducting portions of the electrodes are made of a liquid which boils at a temperature above that at which the silent discharge operates. Thus a concentrated solution of sodium chloride, concentrated sulphuric acid, mercury, or Wood's metal may be used.

J. S. G. THOMAS.

[Arc] electrode. W. IRBY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,722,146, 23.7.29. Appl., 7.1.27).—The electrode contains ilmenite (69.0%), chromite (3.0%), magnetite (14.92%), sodium fluoride (0.28%), and ferrotitanium (12.8%).

F. G. CLARKE.

Apparatus [travelling hammer device] for cleaning the electrodes in electrical gas-purifying plants. H. ZSCHOCKE (B.P. 316,071, 19.11.28).

Electric accumulators. E. PARVILLE ET CIE. (B.P. 292,122, 1.6.28. Fr., 14.6.27).

Electric cells. W. R. EDWARDS (B.P. 317,647, 25.8.28).

Construction of selenium bridge or cell. J. NEALE (B.P. 317,158, 31.5.28).

Treatment of hydrocarbons (B.P. 316,336 and 316,352). **Decomposition of hydrocarbons** (B.P. 316,905). **Unsaturated hydrocarbons** (B.P. 315,249).—See II. **Pure hydroxides** (B.P. 305,092). **Per-compounds** (B.P. 316,648).—See VII. **Sharpening tools** (U.S.P. 1,721,414). **Cadmium plating** (B.P. 315,943). **Alloy for conductors** (U.S.P. 1,723,215 and 1,723,867).—See X. **Electrodeposition of rubber** (U.S.P. 1,723,083). **Utilisation of rubber latex** (B.P. 316,924).—See XIV.

XII.—FATS; OILS; WAXES.

Drying of boiled tung oil. F. WILBORN (Farben-Ztg., 1929, 34, 2775–2776).—The increase in weight on drying of boiled tung oil (containing cobalt resinate as drier) was studied under conditions giving clear and frosted films, respectively, *i.e.*, (a) in the absence of all air currents, and (b) in a stream of warm, burnt coal-gas fumes. In both cases initial drying occurs in "islands" which spread until the whole surface is dry. In the production of clear films, drying begins at an increase in weight of 5% and is complete at 8% increase, the maximum increase in weight of the film being 11–15%. Frosted films begin to dry when the film is still losing weight, and over 90% of the surface is dry before the weight again reaches its original value. Drying is

complete at an increase in weight of 2%, a maximum increase of 12–15% being subsequently reached.

S. S. WOOLF.

Nature, manufacture, and use of stearic acid. D. F. CRANOR (Ind. Eng. Chem., 1929, 21, 719–721).—A description of the raw material, production and grading of stearic acid, together with the reasons for its use in rubber, is given.

D. F. TWISS.

Oil of pæony. E. BUREŠ and B. ŠUSTEROVÁ (Časopis Českoslov. Lék., 1928, 8, 185–186).—The oil extracted with carbon tetrachloride from the seeds of *Pæonia peregrina* had d 0.9600, acid value 14.01, saponif. value 211.26, iodine value 187.10, Reichert-Meissl value 1.54, Polenske value 0.56, Hehner value 91.87, acetyl value 12.96. CHEMICAL ABSTRACTS.

Oil of *Cydonia vulgaris*. E. BUREŠ and J. ŠÁTEK (Časopis Českoslov. Lék., 1928, 8, 187–188).—The oil, extracted with ether from the seeds, had d 0.919, acid value 7.82, saponif. value 186.0, iodine value 107.60, Reichert-Meissl value 2.046, Polenske value 0.467, acetyl value 23.58. CHEMICAL ABSTRACTS.

Oil of *Hyoscyamus niger*. E. BUREŠ and A. KRACÍK (Časopis Českoslov. Lék., 1928, 8, 183–185).—The oil, extracted with light petroleum from the seeds, had d 0.921, acid value 23.60, saponif. value 187.70, iodine value 135.70, Reichert-Meissl value 1.06, Polenske value 0.45, Hehner value 93.34, acetyl value (Normann) 21–21.6, (Lewkowitsch) 20.54.

CHEMICAL ABSTRACTS.

Oil of *Nigella sativa*. E. BUREŠ and H. MLÁDKOVA (Časopis Českoslov. Lék., 1928, 8, 186–187).—The oil, extracted with ether from the seeds, had d 0.8930, acid value 14.68, saponif. value 210.60, iodine value 110.9, Reichert-Meissl value 3.38, Polenske value 0.53, Hehner value 89.22, acetyl value 23.92.

CHEMICAL ABSTRACTS.

See also A., Sept., 1001, **Surface properties of soap solutions** (PRESTON and RICHARDSON). **Surface varnishes on water** (MARCELIN). 1002, **Surface solutions and molecular varnishes** (ÉMIR). **Surface solutions of oleic acid** (GUASTALLA). 1024, **Solar oxidation of zymosterol in oil** (ROUSSEAU). 1040, **Oxidation of unsaturated fatty acids** (BAUER and BÄHR). **Fatty acids of rice starch** (LEHRMANN). 1111, **Influence of ultra-violet rays on antirachitic value of soya-bean oil** (IZUME and others). 1114, **Detection of fats and their constituents** (NIETHAMMER).

Stearic and oleic acids in rubber. DINSMORE and others. **Adsorption of stearic acid by carbon.** BLAKE.—See XIV.

PATENTS.

Manufacture of soap. HENKEL & CIE., G.M.B.H. (B.P. 301,020, 30.10.28. Ger., 23.11.27).—Ammonium thiosulphate (e.g., 44% on weight of stock) is added to a potash soap to produce a liquid, non-gelatinising product.

E. LEWKOWITSCH.

Montan wax compositions (B.P. 315,283).—See II. **Treatment of unsaturated carbon compounds** (B.P. 289,414–5).—See III. **Wetting-out agent** (B.P. 316,356).—See VI. **Vitamin-D** (B.P. 316,803).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oil absorption and viscosity of paints. H. WOLFF (Farben-Ztg., 1929, 34, 2667–2668).—A preliminary discussion of the possibility of correlating oil content at “brushing consistency” with viscosity of oil paints. The existence of a critical point in the progressive decrease of viscosity on adding oil to a stiff oil paste is suggested, and is supported by experiment and mathematical reasoning.

S. S. WOOLF.

Setting processes [of paints]. P. NETTMANN (Farben-Ztg., 1929, 34, 2720–2721).—Blom's views on the importance of the initial stages of gel formation in determining the life of paint films are supported, and it is adduced that the settling of pigments is a factor meriting extended consideration in this connexion. Reconstruction of Stokes' formula (from which, at present, many faulty deductions are made) is advocated. The significance of the chemical nature of the falling body is discussed. Thun's apparatus, by means of which the progress of a falling body and the indications of a recording chronometer are simultaneously photographed on a cinematograph film, is described.

S. S. WOOLF.

Abrasion resistance of films. G. G. SWARD (Amer. Paint and Varnish Manufs.' Assoc., July, 1929, Circ. No. 353, 625–630).—Different types of abrasion tests due to various investigators are described, and preference is expressed for the falling-sand method. Abrasion values of films aged for 2 and 7 days under standard conditions are compared with hardness values, as determined by the swinging-beam method. It is pointed out that no proportionality exists, and suggested that abrasion measures actual wear whilst swinging-beam values indicate “imprintability” of the film.

S. S. WOOLF.

Determination of scratch hardness [of paint and varnish films] by means of lead pencils. H. WOLFF and F. WILBORN (Farben-Ztg., 1929, 34, 2721–2722).—Whilst the method of determining the hardness of films from their resistance to scratching with lead pencils of various grades of hardness is considered to be of very limited scope (owing to influences such as temperature, humidity, film thickness, etc.), it is capable of yielding useful information. To overcome the error due to varying pressure on the pencil (especially significant in the case of the softer grades) it is suggested that the panel to be tested be placed on one pan of a balance, the other pan of which is loaded with a definite weight (e.g., 300 g.). The scratch test is now carried out, sufficient pressure being brought to bear on the pencil to cause the pans approximately to balance.

S. S. WOOLF.

Colorimetric determination of turpentine vapours in air. P. N. ANDREEV and A. A. GAVRILOV (J. Chem. Ind. Moscow, 1928, 5, 1282–1287).—Determinations based on colorimetric comparison, after 1–3 hrs., of solutions in concentrated sulphuric acid give results from 99.9 to 101.6% of the quantity present. The results are unaffected by the presence of benzene or petroleum.

CHEMICAL ABSTRACTS.

Manufacture of blanc fixe. J. E. ADADUROV (J. Chem. Ind. Moscow, 1928, 5, 1420–1424).—Precipitation is

preferably effected at 60—70° from a solution of 20 g. of barium chloride dihydrate in 100 g. of water with 20% sulphuric acid in equivalent quantities. The yield is decreased by 2—2.5% by the presence of 5% or more of hydrochloric acid. Unless the precipitate is collected immediately, or kept under water, the crystal size is increased by mother-liquors containing sulphuric acid. The precipitate is dried in a vacuum at 75—80°. **CHEMICAL ABSTRACTS.**

Determination of cobalt in driers, japans, alloys, etc. O. HEIM (Analyst, 1929, 54, 464—465).—See B., 1929, 690.

See also A., Sept., 1911, Soluble lakes of aurintricarboxylic acid (THRUN).

PATENTS.

Coating compositions [containing rubber]. E. I. DU PONT DE NEMOURS & Co., Assees. of [A] C. M. STINE and C. COOLIDGE, [B] C. M. STINE (U.S.P. 1,721,930—1, 23.7.29. Appl., [A] 3.7.25, [B] 25.7.25).—(A) A solution of rubber in a volatile solvent, having viscosity up to 2700 sec. (Scott), and containing a drier and at least 10% of rubber, is agitated in the presence of oxygen until the viscosity has dropped to as low as 80 sec. With the introduction of a metal drier the solution is used as a protective coating. (B) A solution as above, containing a drying oil, a drier, and an organic nitro-compound is claimed. **S. S. WOOLF.**

Coating composition. C. M. STINE and C. COOLIDGE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,723,632, 6.8.29. Appl., 16.7.25).—A solution of rubber, a drying oil, a cellulose derivative, and a substance which acts as a mutual catalyst for the curing of the rubber and the oxidation of the oil is claimed. **S. S. WOOLF.**

Production of sublimed white lead [basic sulphate]. J. D. McLACHLAN, Assee. of S. J. WARREN (B.P. 291,079, 28.3.28. Austral., 28.5.27).—Flue dust and fume, obtained from roasting plant or cold-top smelters operating on sulphur-bearing leady ores, are dispersed at low red heat under conditions which ensure access of oxygen and, if desired, gases containing sulphur to the individual particles, and the sublimate is separated. **S. S. WOOLF.**

Lithopone composition. J. E. BOOGE and M. L. HANAHAN, Assrs. to GRASSELLI CHEM. CO. (U.S.P. 1,722,174, 23.7.29. Appl., 11.2.28).—The particles of light-resisting lithopone are coated with soap to act as a dispersing agent. **F. G. CLARKE.**

Manufacture of titanium pigments. B. LAPORTE, LTD., I. E. WEBER, and A. N. C. BENNETT (B.P. 315,904, 17.4.28).—Barium sulphate is added to or formed in a fine dispersion of titanium oxide, which is then coagulated, preferably by the addition of barium carbonate, and the mixture is calcined. The peptisation of the oxide may be effected by means of hydrochloric or nitric acid, or its barium salt, or strontium chloride. **S. S. WOOLF.**

Production of blue pigments. C. F. E. BUSCH and V. SORESEN (B.P. 317,274, 23.11.28).—Ash of pit coal or of coke is heated with hydrochloric acid and the

resultant ferric chloride solution, after filtration, is treated with potassium ferrocyanide solution.

S. S. WOOLF.

Preparation of luminous surfaces. W. F. BLEECKER (U.S.P. 1,718,626, 25.6.29. Appl., 9.10.22).—After applying an adhesive, the surface is coated first with a radio-responsive substance, e.g., crystalline zinc sulphide, and then with a solution of a radioactive material, e.g., radium chloride. The surface may then be rendered insoluble by treating with a sulphate and applying a protective layer. **F. G. CLARKE.**

Decolorisation of shellac or other lac resins. E. DUTT (B.P. 316,423, 13.7.28).—A methyl (or ethyl) alcoholic solution of the shellac is neutralised with ammonia, ethylamine, lime, etc., agitated with a calcined aluminous earth (e.g., Indian bauxite) rich in titania but with a low content of iron oxide and silica, and filtered. **E. LEWKOWITSCH.**

Bleaching and hardening of resins. C. CORDES A.-G., and P. STUHLMANN (B.P. 289,859, 4.5.28. Ger., 4.5.27).—Dark resins are dissolved in dilute alkaline lye with continuous agitation and in the same operation are treated with an aldehyde (e.g., formaldehyde) and a bleaching agent such as sodium bicarbonate, hydrogen peroxide, etc. Casein, glues, etc. may be added, and the bleached resin is precipitated in a very finely-divided condition by aluminium sulphate etc. **E. LEWKOWITSCH.**

Composite resin ester. C. ELLIS (U.S.P. 1,722,566, 30.7.29. Appl., 26.12.22).—A resinous product of low acid value, which blends readily with nitrocellulose, is prepared from glycerol, phthalic anhydride, and excess rosin. **E. LEWKOWITSCH.**

Manufacture of plastic substances. J. C. PATRICK and N. M. MNOKIN (B.P. 302,270, 12.6.28. U.S., 13.12.27).—Halogen additive (especially chlorinated) products of ethylene and propylene (e.g., from cracked petroleum) are agitated with moderate heating with aqueous solutions of soluble polysulphides of the alkalis or alkaline-earths. The hardness and elasticity of the product varies according to the sulphur content of the polysulphide employed. **E. LEWKOWITSCH.**

Phenolic resin and its manufacture. F. GROFF and G. W. MILLER, Assrs. to BAKELITE CORP. (U.S.P. 1,720,895, 16.7.29. Appl., 25.6.27).—A fusible, non-reactive, resinous product is obtained by condensing a fusible phenol-methylene resin with furfuraldehyde in the presence of a basic condensing agent. **S. S. WOOLF.**

Resinous condensation product of phenol and cellulose. E. E. NOVOTNY and C. J. ROMIEUX (U.S.P. 1,721,315, 16.7.29. Appl., 6.9.24).—A soluble, fusible resinous mass is obtained by heating a mixture of a phenolic substance and cellulose to above 121°. **S. S. WOOLF.**

Treatment of synthetic resins and particularly of coatings thereof. J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 317,225, 8.8.28).—Unhardened (soluble) synthetic resins prepared from polybasic acids and alcohols etc., especially in the form of coatings, are hardened by exposure, at temperatures from 100° to 200°,

to the vapours of ammonia or of acids stronger than the acids forming the components of the original resinous material, *e.g.*, sulphur dioxide, formic acid, hydrochloric acid, which may be diluted with inert gases.

E. LEWKOWITSCH.

Manufacture of new resin-like products. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 316,914, 3.5.28).—A multivalent alcohol is partially esterified with a monobasic aliphatic or aromatic acid, and the reaction product is then condensed with a polybasic acid or anhydride. The reactions may be conducted in an inert gas or by the aid of a catalyst (*e.g.*, an alkaline-earth ethoxide). The products are suitable for incorporation with "glyptal" resins, and in varnishes and lacquers.

E. LEWKOWITSCH.

Manufacture of condensation products from urea, thiourea, or their derivatives, and an alcohol or a ketone. I. G. FARBENIND. A.-G. (B.P. 292,595, 11.6.28. Addn. to B.P. 278,390; B., 1928, 648. Cf. also B.P. 280,238, 287,095, and 290,192; B., 1929, 28, 691, 709).—The oily or resinous products obtained as in the processes described in the patents mentioned above are treated with formaldehyde or with substances producing it.

E. LEWKOWITSCH.

Coating materials. [Water japons.] W. P. DAVEY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,723,844, 6.8.29. Appl., 16.8.26).—See B.P. 275,955; B., 1928, 614.

Production of white lead. F. T. BAILEY and W. AUSTIN (U.S.P. 1,723,001, 6.8.29. Appl., 22.6.26).—See B.P. 273,287; B., 1928, 866.

Treatment of unsaturated carbon compounds (B.P. 289,414–5).—See III.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Time- and temperature-plasticity relations for crude rubber. E. O. DIETERICH (Ind. Eng. Chem., 1929, 21, 768–770).—Measurements with the Goodrich plastometer show that over relatively short intervals the rate of mastication of any one lot of rubber is directly proportional to the time of milling or to the size of the batch. No discontinuity is observable near 70° in the rate of change of plasticity of rubber by heat, but the influence of each degree rise is the greater the higher is the temperature.

D. F. TWISS.

Optical anisotropy of stretched rubber. H. ZOCHER [with H. J. VON FISCHER] (Kautschuk, 1929, 5, 173–175).—The double refraction of various samples of stretched or frozen rubber was examined by means of an Iceland spar compensator. This provides a much more convenient and sensitive index of crystallisation in rubber than do X-ray tests. It is suggested that crystallisation on stretching is due to the complex mixture yielding, on pressure, a pure modification of rubber which immediately crystallises.

D. F. TWISS.

Reduction of rubber stress-strain determinations. W. B. WIEGAND and H. A. BRAENDLE (Ind. Eng. Chem. [Anal.], 1929, 1, 113–117).—In order to find the true value for the tensile strength and elongation, 95 dumb-bell pieces were cut from an inner tube and tested. Mathematical analysis of the results indicated that the true value is not the arithmetical mean. Dividing

the 95 sets of results into groups of five, and examining these by various methods, it was found that, working with five test pieces, values approaching most nearly to the correct were given by the average of the three highest elongations and the corresponding tensile strengths.

D. F. TWISS.

Influence of temperature and humidity control in rubber testing. II. Resistance to abrasion. Rep. Phys. Testing Committee of Division of Rubber Chem. of Amer. Chem. Soc. (Ind. Eng. Chem. [Anal.], 1929, 1, 174–180; cf. B., 1929, 104).—Investigation of the behaviour of four grades of vulcanised rubber, including "pure rubber," two motor tread qualities, and one heel quality, on two types of abrasion-testing machine shows that differences in relative humidity of the atmosphere before or after vulcanisation are unlikely to have any serious influence on abrasion-resistance. Variation of temperature between 15° and 35° has a distinct effect, the degree and direction of which, however, depends on the actual grade under test: the alteration between 15° and 35° amounts in some cases to 25%.

D. F. TWISS.

Early experiments with stearic acid in rubber compounding. W. F. RUSSELL (Ind. Eng. Chem., 1929, 21, 727–729).—A review of the development of knowledge with respect to the function of the rubber resins, stearic acid, and zinc oxide in the vulcanisation process.

D. F. TWISS.

Effect of increased quantities of stearic acid on [rubber] tread abrasions. C. O. NORTH (Ind. Eng. Chem., 1929, 21, 725–726).—The laboratory abrasion-testing machine of Williams (B., 1927, 635) gives erroneously good results with tread rubber samples containing proportions of stearic acid or paraffin wax up to approximately 30%. This is due to lubricating effect of "bloom" of acid or wax. The only final indication of the degree of abrasion-resistance is by means of road tests.

D. F. TWISS.

Effect of stearic acid on reclaimed rubber. H. A. WINKELMANN and E. B. BUSENBURG (Ind. Eng. Chem., 1929, 21, 730–732).—Stearic acid is not as effective as are other plasticising agents in the reclaiming process. Its subsequent presence, however, increases the plasticity and improves the behaviour in extruding and calendaring; it also aids dispersion of powders in the mixing operation and improves the results obtained on vulcanisation. The last-named effect is also produced on rubber mixtures containing reclaimed rubber.

D. F. TWISS.

Stearic and oleic acids as rubber-compounding ingredients. R. P. DINSMORE (Ind. Eng. Chem., 1929, 21, 722–723).—In a mixing containing mercaptobenzthiazole and rubber (100 pts.), carbon black (23), and zinc oxide (1), stearic acid (4 pts.) gives a higher modulus than oleic acid (4 pts.) in the vulcanised product. In the absence of zinc oxide stearic acid retards chemical and physical "cure," but with zinc oxide present it accelerates chemical cure, whilst with zinc oxide and mercaptobenzthiazole together it expedites physical cure. The primary function of stearic acid in rubber is to furnish zinc in a soluble form for the use of organic accelerators.

D. F. TWISS.

Effect of stearic acid on various crude rubbers.

E. W. FULLER (Ind. Eng. Chem., 1929, 21, 723—725).—Comparing natural rubbers with 1.36, 0.79, 0.20, and 0.12% of resin acids, respectively, in a mixing with 10% of sulphur and 1% of stearic acid, the presence of the last-named had very little effect on the strength or extensibility of the vulcanised products. When these rubbers were tested with 3½% of sulphur, 6% of zinc oxide, and 1% of diphenylguanidine or ½% of an aldehyde-amine accelerator, the additional presence of 1% of stearic acid caused a distinct fall in the physical properties of the rubber of high resin acid content but improved the rubber of low resin acid content. With ½% of the aldehyde-amine accelerator the maximum tensile strength and modulus were obtained with a total acid content of approx. 1.4%, whilst for 1% of the accelerator the corresponding total acid content of the rubber was near 1%. With di-*o*-tolylguanidine the maximum tensile strength was obtained at zero acid content. There is probably a definite optimum total acidity of the rubber mixture. D. F. TWISS.

Stearic acid in litharge-vulcanised rubber compounds.

J. R. SHEPPARD (Ind. Eng. Chem., 1929, 21, 732—738).—Smoked sheet rubber generally contains sufficient natural acid for full activation of litharge, and the addition of acidic softeners such as stearic acid impairs the physical properties of the vulcanised product; a low-grade rubber, *e.g.*, Laporì, however, may be distinctly improved, the beneficial influence on vulcanisation more than compensating for any softening effect on the rubber itself. In a mixture of smoked sheet rubber with gas-black, sulphur, and litharge, the presence of stearic acid leads to improved tensile strength, probably by way of improved dispersion of the black, the rate of vulcanisation being practically unaffected. When litharge is used as activator for mercaptobenzthiazole, additional stearic acid has little effect either on "pure rubber" mixtures or on mixtures containing much gas-black; this is in marked contrast with the considerable influence of stearic acid on corresponding mixings containing zinc oxide instead of litharge. Mixtures of rubber with gas-black, litharge, and mercaptobenzthiazole possess high tensile strength over a wide range of vulcanisation with or without stearic acid. D. F. TWISS.

Adsorption of stearic acid by carbon.

G. T. BLAKE (Ind. Eng. Chem., 1929, 21, 718—719; cf. B., 1928, 867).—On the theory that the natural fatty acids in rubber form a unimolecular film over the particles of a compounding ingredient, *e.g.*, carbon black, in rubber, it would be expected that the thermal change when a powder is mixed into rubber would be the heat of wetting of the particles by fatty acid and not by the rubber hydrocarbon. This expectation is confirmed by two independent methods of computation.

D. F. TWISS.

Causes of deterioration of ebonite when exposed to light and air.

J. D. FRY and B. D. PORRITT (India-rubber J., 1929, 78, 307—310).—Experiments are described which demonstrate that hydrogen sulphide is evolved from ebonite in air and daylight at ordinary temperature, due to the decomposition of some caout-

chouc-sulphur compound. The rate of evolution is increased by sunlight or by rise in temperature.

D. F. TWISS.

See also A., Sept., 1908, **Swelling of rubber jellies** (BLOW and STAMBERGER).

Nature etc. of stearic acid. CRANOR.—See XII.

PATENTS.

Treatment of oil-bearing nuts [rubber seeds] prior to transportation or storage. OILSEEDS BALING Co., LTD., and S. W. BUNKER (B.P. 316,631, 2.2.28).—Rubber seeds (nuts) are decorticated and dried at about 100°, or submitted to superheated steam to inhibit enzyme action, and the kernels are lightly compressed to form a bale. E. LEWKOWITSCH.

Concentration of [rubber] latex. S. G. S. DICKER. From RUBBER LATEX RES. CORP. (B.P. 316,006, 23.7.28).—Latex to which hæmoglobin has been added is concentrated by evaporation, with or without spraying, at a temperature below the coagulation point of hæmoglobin in an atmosphere of sub-normal humidity. The products, which may be cheesy or powdery in consistency, are re-dispersible. Vulcanisation renders the hæmoglobin insoluble and yields a mass in which the constituents are intimately and stably associated.

D. F. TWISS.

Manufacture of articles from dispersions of organic materials [e.g., rubber latex]. DUNLOP RUBBER Co., LTD., and G. W. TROBRIDGE (B.P. 315,814, 19.4.28).—A porous or non-porous deposition base provided with desired markings, *e.g.*, by engraving or embossing, is brought into contact with a dispersion, *e.g.*, a natural or artificial rubber dispersion, which may be concentrated, and/or compounded, and/or vulcanised, or otherwise. To produce a multicoloured pattern the deposition base may be treated with colouring matters. The deposit may be submitted to drying and/or vulcanisation before or after removal from the base.

D. F. TWISS.

Coating of solid surfaces [with rubber etc.]. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 315,901, 16.4.28).—Surfaces of articles coated with an aqueous dispersion, *e.g.*, of rubber, compounded or otherwise, are subjected to heat under conditions promoting oxidation; the presence of driers, such as manganese oleate, accelerates the process whereby the coatings are rendered hard and tenacious.

D. F. TWISS.

Utilisation of rubber latex. TOTO Co., LTD., J. L. BUCHANAN, and S. P. SCHOTZ (B.P. 316,924, 5.5.28).—The addition of lignone extract, obtained as described in B.P. 229,002 (B., 1925, 312), to latex facilitates the introduction of the customary fillers into latex, causes a greater yield per unit current in electrodeposition, and assists the co-deposition of viscose. Ammonium persulphate or sulphate, a metabisulphite, or a polythionate may also be added to the latex for electrodeposition purposes.

D. F. TWISS.

Manufacture of articles substantially made of organic materials from dispersions, emulsions, or solutions containing the said organic materials. DUNLOP RUBBER Co., LTD., E. A. MURPHY, and D. F.

TWISS (B.P. 317,129, 12.5.28).—In the use of aqueous dispersions, *e.g.*, rubber latex, for manufacturing purposes, the disadvantages arising from the presence of air bubbles are obviated by the application of pressure to the latex before or during the manufacturing operation, *e.g.*, during the dipping or electrodeposition process, or during any subsequent coagulation or drying process.

D. F. TWISS.

Production of homogeneous deposits from aqueous dispersions of rubber and/or similar materials. ANODE RUBBER CO. (ENGLAND), LTD. From P. KLEIN (B.P. 316,594, 29.3.28).—Latex, artificially concentrated, *e.g.*, by centrifuging (cf. B.P. 219,635; B., 1925, 139), can be used for the electrodeposition of rubber with advantage if precautions such as those indicated earlier (cf. B.P. 257,885; B., 1927, 533) are observed.

D. F. TWISS.

Manufacture and electrolytic deposition of an aqueous rubber emulsion containing sulphur. S. E. SHEPPARD and L. W. EBERLIN, ASSRS. to AMER. ANODE, INC. (U.S.P. 1,723,083, 6.8.29. Appl., 25.3.25).—A mixture of a fluid, alkaline, aqueous emulsion of unvulcanised rubber with a colloidal dispersion of sulphur is subjected to electrodeposition with formation of masses containing the rubber and sulphur intimately associated.

D. F. TWISS.

Manufacture of thin rubber sheet material [leather substitute]. P. SCHIDROWITZ and D. J. BURKE (B.P. 314,783, 2.4.28).—A vulcanisable mixture of rubber (1 vol.) with a substantial proportion (0.5–1 vol.) of a reinforcing agent, *e.g.*, gas black, colloidal clay, or magnesium carbonate, together with rapeseed oil factice (0.3–0.7 vol.) and (if desired) mineral rubber and litharge, is calendered into sheet form and vulcanised. By subsequent dry-heat treatment in air for several hours, *e.g.*, at about 70°, the hardness of the material, which is an efficient leather substitute, is considerably enhanced.

D. F. TWISS.

Manufacture of artificial rubber or rubber substitutes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 315,916, 21.4.28).—Artificial rubber produced by polymerisation from diolefines is mixed, before vulcanisation, with finely-divided carbon or other finely-divided substances, with or without other organic and inorganic fillers and vulcanising agents. The finely-divided substances may be added to the rubber-producing hydrocarbons before their polymerisation to rubber.

D. F. TWISS.

Manufacture of artificial rubber or rubber-like masses. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,030, 9.5.28).—In the polymerisation of butadiene hydrocarbons, singly or in admixture with one another or with other unsaturated hydrocarbons, the process is expedited and the yield improved by the presence of a finely-divided or colloidal metallic oxide, *e.g.*, lead dioxide, mercuric oxide, silver oxide, or manganese dioxide, and, if desired, of an aqueous solution of an emulsoid colloid. The colloidal oxide may be produced by the addition of a metallic salt. Complete conversion into rubber by this process occupies only a few days.

D. F. TWISS.

Preparation of synthetic rubber. GOSUDARSTVENNYI TREST REZINOVOI PROMYSHLENNOSTI (RESINOTREST), and B. W. BYSOW (B.P. 314,932–3, 5.7.29).—Polymerisation of diolefines to synthetic rubber is favoured by the presence of (A) the dimeride of the diolefine, which suppresses undesirable further formation of the dimeride, or (B) mixtures of substances inclined to tautomerism, *e.g.*, ethyl benzoylacetate with diazoaminobenzene or phenyl mustard oil (phenylthiocarbimide).

D. F. TWISS.

Manufacture of synthetic rubber. GOODYEAR TIRE & RUBBER CO., ASSEES. of R. P. DINSMORE (B.P. 297,050, 31.5.28. U.S., 13.9.27).—Unsaturated hydrocarbons capable of polymerisation to form rubber are emulsified with water with the aid of a fatty acid soap; a stabilising colloid such as casein or albumin is added. After several months in a sealed container at 50–70° the mixture is viscous and after treatment with alcohol or acetone yields a mass of rubber.

D. F. TWISS.

Rubber compound and its preservation. GOODYEAR TIRE & RUBBER CO., ASSEES. of A. M. CLIFFORD (B.P. 296,398, 8.5.28. U.S., 31.8.27).—The reaction product of a naphthalene compound such as α - or β -naphthol with a diamine, *e.g.*, ethylenediamine or *m*-phenylenediamine, is incorporated in rubber as an anti-oxidant.

D. F. TWISS.

Vulcanisation of rubber. RUBBER SERVICE LABS. CO., ASSEES. of W. SCOTT (B.P. 297,726, 21.3.28. U.S., 26.9.27).—Vulcanisation is accelerated by the condensation product of an aliphatic aldehyde-ammonia, *e.g.*, butaldehyde-ammonia, with a further quantity of the same or other aldehyde; the condensation process is preferably effected in the presence of a small quantity of an organic acid of at least 4 carbon atoms, *e.g.*, 2–4% of butyric acid.

D. F. TWISS.

Manufacture of vulcanised rubber. W. SCOTT, ASSR. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,721,057, 16.7.29. Appl., 20.6.22).—Vulcanisation is effected with the aid of a ditolylguanidine.

D. F. TWISS.

Manufacture of vulcanised rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,554, 22.6.28).—Rubber is vulcanised with the aid of a compound of selenium and sulphur, which may also contain free selenium and/or sulphur, in the form of a paste obtained by trituration with an oily or fatty product, *e.g.*, petrol-eum jelly, stearic or oleic acid, wool fat, etc.

D. F. TWISS.

Manufacture of vulcanised rubber. CLAYTON ANILINE CO., LTD., and R. ROBINSON (B.P. 317,205, 20.7.28).—Deterioration of rubber is retarded by vulcanising in the presence of a reaction product of an aromatic amine, or a mixture of such amines, with a condensation product from an aromatic amine and an aliphatic aldehyde containing 2 or more carbon atoms. In the case of an amine, *e.g.*, dimethylaniline, or a mixture of amines which does not react with the aldehyde, the amine or mixture can be present during the aldehyde-amine condensation. The condensation product of α -naphthylamine (1 mol.) and acetaldehyde (2 mols.), for example, may be made to react with β -naphthylamine or dimethylaniline.

D. F. TWISS.

Vulcanisation of rubber. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 316,692, 8.5.28).—Vulcanisation is effected in water containing a water-soluble dithiocarbamate, *e.g.*, a dibenzylidithiocarbamate, of an inorganic base, particularly of an alkali metal, an alkaline-earth metal, or magnesium. D. F. TWISS.

Adhesive rubber composition. M. C. TEAGUE, Assr. to GEN. RUBBER CO. (U.S.P. 1,719,948, 9.7.29. Appl., 18.9.24).—A mixture of an aqueous dispersion of rubber with an emulsion of a cumarone-containing resin obtained by the polymerisation of a coal-tar distillate. D. F. TWISS.

Production of rubber articles. M. C. TEAGUE, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,719,633, 2.7.29. Appl., 12.4.26).—A former is repeatedly coated with a rubber dispersion, the retained dispersion being coagulated on the former; the coated former is washed after each coagulation, and the coagulated deposit is dried before removal. D. F. TWISS.

Combining waste or scrap rubber with a natural or artificial aqueous dispersion of rubber. C. MACINTOSH & Co., LTD., and F. H. TOOR (B.P. 315,512, 11.5.28).—Rubber crumb obtained by comminuting scrap vulcanised rubber is mixed with a suitable proportion, *e.g.*, 3%, of rubber in the form of an aqueous dispersion which may also contain any desired vulcanising agents and fillers. The mixture is then compressed and vulcanised. D. F. TWISS.

Condensation of α -naphthylamine and acetaldehyde (B.P. 316,761).—See III. **Coating compositions** (U.S.P. 1,721,930—1 and 1,723,632).—See XIII.

XV.—LEATHER; GLUE.

Bating. G. D. McLAUGHLIN, J. H. HIGHBERGER, F. O'FLAHERTY, and E. K. MOORE (J. Amer. Leather Chem. Assoc., 1929, 24, 339—379).—It is shown that enzyme bating materials partially delime skins which have been swollen with lime liquors, and consequently the skins become fallen and flaccid. The various tissues in a limed skin are unequally swollen, hence bating brings the tissues to a more equal state of swelling so that the grain in particular feels smoother. Lime swelling reduces the stretch of the elastin fibres and bating restores the original stretch. The keratose is removed from the grain layer, coagulable protein is removed from the skin. The reticular tissue is removed or broken up. The physical changes involved in bating can be effected by deliming agents, but enzymes are required for the removal of the keratose, coagulable protein, and reticular tissue. The removal of coagulable or coagulated protein from the skin is probably the most important function of bating, and if more of this is removed in processes prior to the bating there is less for the latter process to remove. A large portion of the coagulable protein is removed from wet salted skins which have been soaked in a salt solution, and such skins require a mild bate only, but dried, unsalted skins contain all the original coagulable protein, which requires drastic bating to remove it unless it is soaked in a concentrated salt solution. D. WOODROFFE.

PATENTS.

Treatment of [tanned] leather. W. E. LANE (U.S.P. 1,720,223, 9.7.29. Appl., 11.5.27).—Leather for

insoles etc. is stiffened by being dipped into a weak solution of sodium silicate. A. B. MANNING.

Manufacture of gelatin capsules for bottle caps. E. C. R. MARKS. From PARKE, DAVIS & Co. (B.P. 315,103, 16.7.28).—The external surface of the still plastic gelatin capsules is treated while they are on the forming moulds with a tanning agent, *e.g.*, formaldehyde, benzaldehyde, tannic acid, in solution with glycerin and water. D. WOODROFFE.

Colouring of horn masses. H. OEXMANN (B.P. 298,946, 15.10.28. Ger., 17.10.27).—The artificial colouring of horn masses is rendered more permanent by adding formaldehyde, substances generating formaldehyde, or polymerides of formaldehyde to the colouring agent; alternatively, such substances may be applied to the horn before or after dyeing. D. F. TWISS.

Automatic device for effecting rapid tanning of hides or skins. G. VALLE and A. PODESTA (B.P. 317,691, 24.11.28).

Dyeing animal fibres (U.S.P. 1,718,882).—See VI. **Leather substitute** (B.P. 314,783).—See XIV.

XVI.—AGRICULTURE.

Vertical distribution of soil bases and acidity in some Illinois soils. H. A. LUNT (Soil Sci., 1929, 28, 137—176).—Results of the examination of depth-samples by a number of well-known methods, for acidity and exchangeable base content, are recorded. Comparisons of the various analytical results of the soil types are thus obtained. A. G. POLLARD.

Ranges of p_H values [of soils] in water and in potassium chloride solution. A. GOY and von BUROW (Z. Pflanz. Düng., 1929, 14A, 348—354).—Differences in the reaction of soils are of more practical value when expressed as mg. of hydrogen ion per litre than as p_H values. The soil classification previously described (B., 1929, 787) is discussed on this basis. A. G. POLLARD.

Soil type and crop adaptation. J. H. STALLINGS (Soil Sci., 1929, 28, 101—124).—The results of attempts to find relationships between various types of Florida soils and suitability of crops are described, and the value of soil surveys in this connexion is shown. A. G. POLLARD.

Utilisation of increasing applications of nitrogen by different varieties of summer barley. III. J. WEIGERT and F. FÜRST (Z. Pflanz. Düng., 1929, 8B, 369—412; cf. B., 1929, 447, 654).—The specific effects of increasing applications of nitrogen between 20 and 70 kg. per hectare as ammonium sulphate, following basal dressings of 60 kg. of phosphoric acid and 90 kg. of potash per hectare as basic slag and potassium chloride or 40% potash salts, respectively, on 14 different varieties of summer barley have been examined during the years 1922—1928. Except in 1922 the average yields of straw increased more rapidly than the yields of grain, of all the varieties, with increasing applications of nitrogen. The highest increases in yields recorded were 99.6% of grain, and 157.7% of straw following the use of 70 kg. per hectare of nitrogen in 1928, whilst the highest increases in yield per kg. of nitrogen were 32.8 kg. per hectare of grain in 1927, and 82.2 kg. of straw per hectare in 1926, each with

25 kg. of nitrogen per hectare in all. The highest ratio of grain to straw, 1:1.13, was recorded in 1924 with 25 kg. of nitrogen, and the lowest, 1:2.43, in 1926 with 70 kg. of nitrogen. Variations in nitrogen application had no significant influence on the volume-weight of the grain, but the weight per 1000 grains showed a small increase. In 1926, but not 1927 or 1928, the percentage of medium-sized grains was increased by increasing nitrogen application, and with the exception of 1925 the effect of nitrogen was to lower the crude protein content of the grain. As compared with earlier work, barley appears to show more response to nitrogen than either wheat or rye. E. HOLMES.

Availability of nitrogenous fertilisers to rice. R. P. BARTHOLOMEW (Soil Sci., 1929, 28, 85—100).—The assimilation of nitrogen by rice cultures from various nitrogenous fertilisers was examined under anaerobic conditions. Ammonium sulphate, Leuna saltpetre, cottonseed meal, sodium nitrate, blood meal, and ammonium phosphate proved reasonably efficient in the order named. Cyanamide and calcium and sodium nitrates mixed with cottonseed meal were much less satisfactory. There is a danger of considerable loss by denitrification from organic nitrogenous substances. The lowered efficiency of sodium nitrate is largely attributed to the sensitiveness of rice to alkaline conditions. Nitrogen losses due to denitrification occurred with all materials examined, and appeared to be due to the formation of elementary nitrogen. Nitrite formation was small and irregular. A. G. POLLARD.

Use of dyes in the isolation of a nitrite-oxidising organism. C. C. PROUTY (Soil Sci., 1929, 28, 125—136).—In the preparation of pure cultures of nitrite-oxidising bacteria, the contaminating forms surviving the usual "enrichment" process may be eliminated by exposure to the action of a 1% solution of rosaniline hydrochloride for 5—30 min. Morphological descriptions of the nitrite organism and the contaminating forms are given. A. G. POLLARD.

Effects of manganese sulphate and chloride on nitrification. D. H. NELSON (J. Amer. Soc. Agron., 1929, 21, 547—559).—Small concentrations of manganese salts irregularly stimulate the nitrification of dried blood or ammonium sulphate in the soil. The toxic action of high concentrations is reduced by lime. CHEMICAL ABSTRACTS.

Soil reaction and vines with regard to various lime-sensitive varieties. O. SARTORIUS (Z. Pflanz. Dting., 1929, 14A, 354—370).—The effect of the reaction of the media on the growth of vines in water, sand, and in soil cultures is examined. In all cases growth declined with an alkaline reaction. The effect was most pronounced in water culture and increased with the concentration of the nutrient media. This is probably due to changes in the permeability of the root cell membranes with reaction. In soil and sand cultures the optimum growth range was p_H 6.0—6.5. A. G. POLLARD.

Meadow fertilisation experiments in Ticino. B. SCHMITZ (Landw. Jahrb. Schweiz, 1928, 42, 783—801; Chem. Zentr., 1929, i, 1499).—Fertilisation experiments on hay with potassium and phosphorus are described. A. A. ELDRIDGE.

Substitution of stable manure by fertilisers, green manure, and peat. III. B. L. HARTWELL and F. K. CRANDALL (Rhode I. Agric. Exp. Sta. Bull., 1928, No. 216, 1—20).—A summary of 12-yr. rotation experiments with cabbage, tomatoes, celery, lettuce, beet, spinach, and oats with respect to acidity, phosphate, nitrogen, and manganese. CHEMICAL ABSTRACTS.

Fundamental principles of the preparation of artificial fertilisers. G. RUSCHMANN (Fortschr. Landw., 1929, 4, 81—84; Chem. Zentr., 1929, i, 1390).—The value of stable manure lies chiefly in its content of organic substances. In Krantz's method of fermentation of straw complete humification takes place. Addition of nitrogen in the fermentation of straw favours the activity of the bacteria concerned and improves the C:N quotient. A. A. ELDRIDGE.

Insecticidal tests with oils and alkaloids of larkspur (*Delphinium consolida*) and stavesacre (*Delphinium staphisagria*). W. M. DAVIDSON (J. Econ. Entomol., 1929, 22, 226—234).—The pests killed by soap emulsions of the oils or alkaloids are recorded. CHEMICAL ABSTRACTS.

Correlation between oil sprays and chlorophyll content of foliage. J. M. GINSBURG (J. Econ. Entomol., 1929, 22, 360—366).—Leaves of apple trees sprayed during July and August with a refined lubricating oil contained more chlorophyll than leaves of untreated trees. CHEMICAL ABSTRACTS.

Bentonite as a dust carrier for nicotine [in plant sprays]. L. R. STREETER (J. Econ. Entomol., 1929, 22, 234—235).—Decomposition of nicotine was not observed, but the colloidal clay has a high absorptive power, and should not be used if volatility of nicotine is desired. CHEMICAL ABSTRACTS.

Mercury salts as soil insecticides. H. GLASGOW (J. Econ. Entomol., 1929, 22, 335—340).—Effective insecticidal action by aqueous solutions of mercuric chloride and by suspensions or dusts of mercurous chloride on the cabbage maggot, carrot rust fly, and onion maggot is recorded. CHEMICAL ABSTRACTS.

Storing beets by drying. G. S. BENIN (Nauch. Zapiski, 1928, 6, 68—71).—Dry beet cosettes were unchanged when stored in the laboratory at 10—20°, but gained up to 13% of moisture when stored outside at -9.3° to +20.7°, although protected from rain and snow. Loss of sugar was only 0.2% of the weight of the raw beet; increase of invert sugar was slight. CHEMICAL ABSTRACTS.

Storage of truck crops. Girasole (artichoke). H. P. TRAUB, C. J. THOR, J. J. WILLAMAN, and R. OLIVER (Plant Physiol., 1929, 4, 123—134).—*Helianthus tuberosus* tubers are preferably stored at 0—3° and a relative humidity of 89—92%. From maturity to the end of January the ratios of levulose to dextrose and to water-soluble carbohydrates decrease. At harvesting no appreciable amount of free reducing sugar is present. CHEMICAL ABSTRACTS.

PATENTS.

Soil fertilisation. D. GARDNER (B.P. 316,122, 23.4.28).—To materials containing fixed nitrogen, phosphates, and potassium, singly, or in combination are added small proportions (e.g., 5%) of titanium

compounds (e.g., titania). Still smaller proportions of manganese compounds may be used in addition. Titanium compounds may be used in conjunction with silica and lime and/or magnesia, preferably in molecular proportions. Titanium compounds used must be substantially free from iron and titanium nitrides.

A. G. POLLARD.

Production of fertilisers. K. GORDON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 316,428, 19.7.28).—Crude phosphates are treated in a series of vessels with a countercurrent of sulphuric acid containing ammonium sulphate. The process may be effected above 80°, in which case the mixture before filtration is cooled to convert the precipitate into gypsum, or at about the ordinary temperature, when the precipitated gypsum is converted into calcium sulphate hemihydrate and then into large gypsum crystals by successive heating and cooling. The precipitated calcium sulphate is removed and converted into ammonium sulphate by treatment with ammonium carbonate. L. A. COLES.

Manufacture of mixed fertilisers. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 315,485, 18.4., 27.4., 6.10., and 8.10.28).—Fertilisers in which the ratio N:K₂O is from 1:1 to 1:2.7 are made by treating with potassium nitrate a solution of calcium nitrate and/or magnesium nitrate and converting the mixture into a solid product. The calcium or magnesium nitrate may be partly replaced by ammonium nitrate and/or urea, and phosphates of fertilising value may be added. The potassium nitrate may be added in the solid form or may be formed in solution by adding potassium chloride and treating with nitric acid or nitrous gases.

W. G. CAREY.

Manufacture of a fertilising mixture capable of being strewn. STOCKHOLMS SUPERFOSFAT FABR. AKTIEB. (B.P. 298,196, 1.10.28. Swed., 5.10.27).—Mixtures consisting of ammonium nitrate and ammonium phosphate and, if desired, ammonium sulphate are prepared by passing ammonia through a mixture of the acids at a temperature not much exceeding 100°, and of such concentration that solid salts are obtained, or, alternatively, by passing ammonia into a solution of ammonium phosphate or nitrate in nitric or phosphoric acid, respectively, with or without the addition of sulphuric acid or ammonium sulphate. L. A. COLES.

Production of a complete fertiliser. CHEMIE-VERFAHREN GES.M.B.H. (B.P. 302,148, 16.10.28. Ger., 10.12.27).—Crude phosphate rock is decomposed by a mixture of potassium sulphate and nitric acid in proportions necessary to yield gypsum, potassium nitrate, and free phosphoric acid. The last-named, after the separation of gypsum from the liquor, is neutralised with ammonia, and the whole evaporated to dryness. The proportion of nitrogen, phosphates, and potash may be varied by means of the lime used or the composition of the crude phosphate. A. G. POLLARD.

Preparation of [dry ground] limestone [for fertilisers]. W. E. CARSON (U.S.P. 1,721,803, 23.7.29. Appl., 21.8.26).—Quicklime is added to moist limestone in such proportion that the mixture after being pulverised does not contain sufficient free moisture to form a hard cake when frozen.

H. ROYAL-DAWSON.

Compositions for destroying insect and fungoid

pests in vineyards, hop plantations, orchards, gardens, and fields. E. D. FELDMAN (B.P. 316,991, 5.5.28).—The material consists of a copper sulphate-lime suspension to which is added an emulsified oil containing 15% of a mixture of resin and oleic acid, 2.5% of ammonium chloride solution (*d* 0.910), 7% of methylhexalin, and 75.5% of mineral oil (*d* 0.890). Sulphur and other insecticidal substances may also be incorporated.

A. G. POLLARD.

Producing a liquid insecticide containing effective ingredient of *Derris* species. S. TAKEI, Assr. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,724,626, 13.8.29. Appl., 31.8.25. Jap., 8.9.24).—See B.P. 239,483; B., 1926, 614.

Calcined phosphates (B.P. 301,022).—See VII. **Fungicide** (U.S.P. 1,720,905).—See IX.

XVII.—SUGARS; STARCHES; GUMS.

Chlorine as sugar decolorant. L. R. BLISS (Sugar, 1929, 31, 3—5, 57—59, 99—101).—Chlorine had little effect on the polarisation of sugar, dextrose, invert sugar, and their mixtures. Diluted molasses defecated with Horne's dry lead reagent, followed by treatment with chlorine, gave easily polarised solutions. Cloudy solutions are flocculated before filtration by addition of potash alum or aluminium chloride. Directions for the use of chlorine in the Clerget determination are given, and a tentative method for the approximate determination of purity by destruction of reducing sugars with alkali, followed by chlorine-bleaching and polarisation, is described.

CHEMICAL ABSTRACTS.

Application of the Seliwanoff reaction. R. OFNER (Chem.-Ztg., 1929, 53, 682—683).—Under the usual conditions the Seliwanoff test for ketoses in the presence of aldoses (A., 1887, 459) is not always reliable; thus a 4% solution of dextrose will give a colour equal in intensity to that obtained with a 0.02% solution of levulose. The following modification will detect 1 mg. of levulose or 2 mg. of sucrose in 5 c.c. of a sugar solution having *d* < 1.012. The solution is mixed with 5 c.c. of a 0.05% solution of resorcinol in 24% hydrochloric acid (*d* 1.12), a small piece of pumice is added, and the mixture boiled for exactly 20 sec. and cooled in running water; the resulting rose tint is a measure of the amount of ketose present.

A. R. POWELL.

Reagent of low copper content for determining small quantities of invert sugar. R. OFNER (Z. Zuckerind. Czechoslov., 1929, 53, 733—738).—The proposed reagent contains per litre 5 g. of crystalline copper sulphate, 10 g. of anhydrous sodium carbonate, 300 g. of Rochelle salt, and 50 g. of crystalline disodium phosphate, and is filtered clear, after addition of kieselguhr or charcoal. Of this solution 50 c.c. are mixed with 50 c.c. of a solution of the sugar sample containing not more than 15 mg. of invert sugar, in a 300-c.c. flask, heated to boiling in 4—5 min., maintained in moderate ebullition for exactly 5 min., and then placed in cold water. To determine the unreduced copper, the cold liquid is treated with 5 c.c. of a solution containing 12 g. of potassium iodide and 4 g. of potassium thiocyanate per 100 c.c., and then with 25 c.c. of 1:1 hydrochloric acid, after which a measured excess of 0.02N-thiosulphate solution is run in and the excess

determined by titration with 0.02*N*-iodine solution in presence of starch indicator. A blank titration is made on the original copper solution or better still on an unboiled mixture of 50 c.c. of this with 50 c.c. of the solution of the sample. Only a slight correction is necessary for sucrose, viz., 0.1 c.c. of copper solution per g. of sucrose in the reaction mixture. The copper solution is 0.02*N* and each c.c. corresponds to 0.606 mg. of invert sugar in the reaction mixture, up to 15 mg., but proportionality is not strict for larger amounts. Impure sugar products clarified with basic lead acetate are dealed with disodium phosphate before analysis, and any appreciable deviation from neutrality should be corrected.

J. H. LANE.

See also A., Sept., 1945, **Volumetric determination of invert sugar** (GABREËLS and VAN SCHERPENBERG). **Solidification of sucrose** (TIAN). 1961, **Derivatives of phenylhydrazines with sugars** (VOTOČEK and RYS).

Storage of beets. BENIN.—See XVI.

PATENTS.

Sugar centrifuge. E. SCHULTZ (Russ. P. 3477, 31.8.27).—In a centrifuge on a horizontal shaft, with an outer imperforate drum wall and an inner perforated one, a flange is provided so as to form with the floor of the drum an annular space, from which the separated liquid is withdrawn by a suction device. J. H. LANE.

Effecting a reaction between sucrose and quicklime. R. W. SHAFOR (U.S.P. 1,713,925, 21.5.29. Appl., 5.4.27).—The formation of a precipitate of lime and sucrose is effected in two stages, each of which is carried out continuously in a series of steps. In the first stage the reaction between lime and sucrose solution is carried to the point of concentration of the lime at which the alkalinity approaches a maximum, and in the second stage it is carried further and the precipitate is formed. The material in the second stage is prevented from passing back and mixing with that in the first stage.

J. H. LANE.

Production of a sugar solution [from manioc]. E. J. CROSS. From E. LANGFELDT (B.P. 311,979, 7.7.28).—Comminuted roots of the manioc or cassava plant are freed from part of their juice by washing and/or pressing, and then dried. The product is saccharified by acid for the manufacture of dextrose, alcohol, or yeast, and in the latter cases ammonia may be used for neutralising the acid after saccharification, to provide nitrogenous nutriment for the yeast.

J. H. LANE.

Non-granulating sugar compositions. A. W. FRAME (B.P. 311,916, 24.4.28).—To prevent the development of grittiness, due to slow crystallisation of sugar, in chocolate cream centres, nougat, ice-cream, and other compositions used in confectionery and baking, the compositions are made by mixing the sugar constituents with some water and "maltose," i.e., diastatic conversion products of starch such as malt extract powder or syrup, or malt powder or syrup, heating the mixture, and then cooling rapidly. The products not only retain their smoothness of texture, but can also be kept longer than usual without becoming dry.

J. H. LANE.

Manufacture of dextrose. INTERNAT. PATENTS DEVELOPMENT Co. (B.P. 303,142, 1.10.28. U.S., 30.12.27).

—Mother-liquors from the manufacture of dextrose are further hydrolysed to saccharify polysaccharides present, by diluting them to about *d* 1.1, acidifying to *p_H* 1.8 with mineral acid, and heating under a pressure of about 45 lb./in.² as in the saccharification of starch. To recover granular crystalline dextrose the liquid is then neutralised with sodium carbonate to *p_H* 4.2, filtered over bone char or otherwise decolorised, evaporated to *d* 1.26, again filtered over char, evaporated to *d* 1.37, crystallised by slow cooling in crystallisers, and centrifuged while still fluid.

J. H. LANE.

Manufacture of dextrose. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,722,761, 30.7.29. Appl., 24.10.27).—Water is continuously removed from a supersaturated solution of dextrose produced from starch, while crystallisation is effected, in order to obtain crystals which are readily cleansed.

F. G. CLARKE.

Apparatus [conveyor] for drying bagasse (megass) or the like. H. W. HOLGATE (B.P. 317,172, 14.6.28).

Artificial silk (B.P. 287,461). **Wood sugar** (B.P. 302,313).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Nourishment of press yeast with inorganic ammonium compounds. W. STACH (Z. angew. Chem., 1929, 42, 842–843).—It is claimed that the figures on which Claassen (B., 1928, 908) based his criticism of the work of Wohl and Scherdel (B., 1921, 191 A) are inconsistent.

F. E. DAY.

[Nourishment of press yeast with inorganic ammonium compounds.] H. CLAASSEN (Z. angew. Chem., 1929, 42, 843).—A reply to Stach (cf. preceding abstract).

F. E. DAY.

Influence of turbidity of the pitching-wort on the primary [bottom-]fermentation of beer. F. WINDISCH (Woch. Brau., 1929, 46, 326–331, 337–340).—The addition of deposit from the coolers to the wort at pitching favours the fermentation rate and final attenuation. The yeast crop which results is more vigorous in fermentation and reproduction than that from normal wort. Prolonged boiling of the wort, which removes some haze particles, has slight effects in the reverse direction, whilst centrifuging for 10 min. at 3500 r.p.m. leads to marked slackening of the fermentation and weakening of the yeast.

F. E. DAY.

Turbidity and turbidity flavour [in brewing]. F. WINDISCH (Woch. Brau., 1929, 46, 325).—Suggestions are made for the better definition of the terms "Trub" (turbidity) and "Trübung" (haze). Material removable by filtration is classed under the former term, and may be further differentiated by particle-size and by the stage in the brewing process at which it appears. "Trübung" is not removable by filtration. Though turbidity, being mainly protein, has no flavour of its own, the association of turbidity with undesirable flavours, e.g., by adsorption, is discussed.

F. E. DAY.

Silicic acid in beer. II. H. NETSCHER (Woch. Brau., 1929, 46, 335–337; cf. B., 1929, 145).—Laboratory brewings with distilled water, water containing sodium silicate, and waters with sodium silicate

neutralised by hydrochloric and lactic acid are compared as regards haze and deposit, and the silicic acid contents of beers and deposits before and after pasteurisation. The results generally support the author's previous conclusions that silicic acid aids clarification and filtration, but increases the tendency to haze formation after pasteurisation.

F. E. DAY.

Detection of fruit wine in grape wine. B. BLEYER and W. DIEMAIR (Chem.-Ztg., 1929, 53, 621--622, 641--642).—Methods involving starch determination, spectrographic analysis, or fluorescence phenomena are considered unsatisfactory, and a chemical method is described based on Werder's observation (cf. B., 1929, 619) that the juice of fruit stones contains sorbitol. The material (100 c.c.) is clarified with 7 g. of animal charcoal, shaken mechanically for 20 min., boiled, and filtered. The filtrate is evaporated, under reduced pressure, to about 5 c.c., centrifuged to remove tartar, and further concentrated to a syrup weighing 1.2--1.5 g. To this 0.2 c.c. of benzaldehyde and 1 c.c. of 50% sulphuric acid are then added, and the mixture is agitated mechanically for 1 hr. After remaining overnight, the syrup is diluted with distilled water; the formation of a precipitate of dibenzylidenesorbitol indicates the presence of fruit wine. Confirmation may be secured by treating the syrup with an equal weight of formalin and a similar weight of concentrated hydrochloric acid. After refluxing on the water-bath for 1½ hrs., and then cooling, characteristic crystals of the triformylacetal separate having m.p. 206° on recrystallisation from alcohol. The crystalline forms obtained with fruit wines, grape wines, and mixtures of these are described, and it is shown that the presence of 10% of fruit wine in a grape wine can be readily detected.

H. J. DOWDEN.

See also A., Sept. 1000, **Adsorption of weak electrolytes by pure charcoal** (PHELPS and PETERS). 1106, **Inactivation of enzymes by tannins** (OPARIN and KURSSANOV). **Catalase** (MADINAVEITIA). **Peroxidase** (WILLSTÄTTER and others). **Qualitative test for invertase** (KERTÉSZ). 1107, **Purification of pepsin** (PLÉ). 1108, **Influence of metals on acetic acid fermentation** (ROSENBLATT and MORDKOVITSCH). 1109, **Action of minute doses of sea-water on fermentation** (RICIET and FAGUET).

Artificial fertilisers. RUSCHMANN.—See XVI.

PATENTS.

Carrying out biochemical processes. DEUTS. HYDRIERWERKE A.-G., Assees. of S. BAKONYI (B.P. 293,015, 13.4.28. Ger., 30.6.27).—In carrying out bacterial fermentations, natural mixed cultures are used, which are attached to suitable nutrient substrata ("microplankta") and are subjected to one or more "selection" processes, by being cultivated in the presence of the substance or substances to be produced, so that only those strains survive that are strongly resistant to the substance or substances. Such cultures are highly virulent, and the processes are considerably quickened.

H. J. DOWDEN.

Manufacture of fermented beverages of reduced alcohol content. H. HEUSER, Assr. to UNITED STATES PROCESS CORP. (U.S.P. 1,720,329, 9.7.29. Appl., 25.9.26).—Fermented cereal wort is heated to reduce its

alcohol content, and at the same time supplied with liquorice extract, after which it is cooled and re-fermented.

J. H. LANE.

Simultaneous distillation, purifying, and dehydration of alcohol obtained from fermented mashes. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 289,051, 12.4.28. Fr., 21.4.27).—Distillation, rectification, and dehydration are carried out in a combined plant, in which a system of heat interchange reduces the steam consumption from 700 kg. to 350 kg. per hectolitre of alcohol, starting from a 7% molasses mash. The vapour issuing from the still at 78° is used to heat the dehydrating column, whilst the wash at 105° is used to heat the mash. If higher working temperatures are required, the distillation tower may be operated under an increased pressure, up to 5 m. of water.

H. J. DOWDEN.

Industrial use of inferior vegetables [yeast etc.].

M. KAHN, E. LE BRETON, and G. SCHAEFFER, Assrs. to SOC. FRANÇ. DES PROD. ALIMENTAIRES AZOTÉS (U.S.P. 1,724,027, 13.8.29. Appl., 19.11.24. Fr., 20.11.23).—See B.P. 225,228; B., 1925, 608.

Extracting juice from juicy materials (B.P. 301,322).—See I.

XIX.—FOODS.

See A., Sept., 1099, **Buffer intensities of milk** (WHITTIER). **Aroma of butter** (VAN NIEL). 1113, **Banana** (MAY). **Pineapple** (HENDRICKSEN).

PATENTS.

Production of baking powder mixtures with acid end-reaction. L. WEIL (B.P. 304,230, 31.12.28. Ger., 17.1.28).—An alkali pyrophosphate is mixed with calcium tartrate, citrate, or acetate, with a calcium casein compound, or with a mixture of calcium carbonate and, e.g., tartaric acid, in such proportions that the product gives a slightly acid reaction after use.

L. A. COLES.

Producing pulverised milk serum product. N. M. KRONBERG (U.S.P. 1,721,867, 23.7.29. Appl., 23.11.28. Swed., 16.9.27).—See B.P. 306,374; B., 1929, 338.

Dietary composition. F. GOEDECKE (U.S.P. 1,721,214, 16.7.29. Appl., 14.11.27. Ger., 11.8.25).—See B.P. 308,403; B., 1929, 453.

Treating foods and products thereof [by irradiation]. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of A. KNUDSON (B.P. 292,926, 25.6.28. U.S., 24.6.27).

Process and apparatus for puffing cereals. J. L. FERGUSON (B.P. 291,036, 24.5.28. U.S., 24.5.27).

Mixing machine (B.P. 291,732).—See I. **Non-granulating sugar compositions** (B.P. 311,916).—See XVII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

[Preparation of] urea-stibamine. S. GHOSH, R. N. CHOPRA, and N. R. CHATTERJEE (Indian J. Med. Res., 1929, 16, 461--468).—A solution of antimony trioxide in hydrochloric acid is gradually added, with stirring, to 20% sodium hydroxide solution, and the opalescent liquid is treated at 1--2° with diazotised *p*-aminoacetanilide, stirring being continued (at 1--2°) for 2 hrs. After dilution and neutralisation with sulphuric acid,

carbon dioxide is passed through the solution, which is then filtered from unchanged antimony oxide. The precipitate resulting from saturation with sodium chloride is collected, dried in air, and purified from methyl alcohol. It is dissolved in 5% sodium hydroxide solution at 60°, treated with an equal weight of carbamide, and concentrated on the water-bath to 0.33 vol. After cooling, it is treated with absolute alcohol to precipitate urea-stibamine. CHEMICAL ABSTRACTS.

Keeping properties of digitalis and some of its preparations. H. B. HAAG and R. A. HATCHER (Amer. J. Pharm., 1929, 101, 474—480).—Observations extending over a number of years indicate that dried digitalis leaf is stable. Fluid extracts and tinctures, however, decompose at variable rates when kept, the latter somewhat more rapidly. No foreign toxic substance is developed. The decomposition is attributed to the action of a substance which is itself decomposed. Ampoules of glass containing a minimum of soluble alkali are recommended for its storage. R. K. CALLOW.

Evaluation of belladonna leaves. E. I. VAN ITALLIE (Pharm. Weekblad, 1929, 66, 657—660).—The moisture content is first determined by drying at 103—105°. The dried material (*n* g.) is treated with 100—*n*/3 g. of alcohol and filtered by suction through a 6-cm. Buchner funnel; the filtrate (*b* g.) is evaporated to about 10 g. and 10 drops of dilute sulphuric acid and sufficient water to make the total weight 20 g. are added. The well-stirred mixture is centrifuged, and the clear liquid (*c* g.) is shaken with about 60 c.c. of ether and 4 c.c. of ammonia solution for 1 min., and for a like period after addition of 4 g. of tragacanth powder. The clear solution (*d* g.) is decanted and the ether distilled off, and a further 3 c.c. of ether are added and removed by evaporation; the residue is dissolved in 5 c.c. of absolute alcohol and, after dilution with 5 c.c. of water, is titrated with 0.04*N*-acid; 1 c.c. of acid = 11.56 mg. of alkaloid. The alkaloid percentage = $13,872,000e/(100 - a)bcd$, where *e* is the acid titre in c.c. H. F. GILLBE.

American and Philippine cigarettes. V. G. LAVA and S. B. ETORMA (Philippine Agric., 1929, 17, 565—577).—The benzene, ether, and alcoholic extracts are smaller, the aqueous extract, ash, total nitrogen, and protein larger, in Philippine than in most American cigarettes. The ratio of the sum of the benzene, ether, and alcoholic extracts to the aqueous extract should be not less than 2. CHEMICAL ABSTRACTS.

Radiation in connexion with essential oils and perfumery chemicals. R. A. MORTON (Perf. & Essent. Oil Rec., 1929, 20, 258—267).—A review of the applications of photochemical changes to perfumery chemicals shows that, apart from the use of ultra-violet rays for the production of vanillin from isoeugenol, little work has been published in this field. The possibilities of the quantitative investigation of absorption spectra in the detection and determination of essential oils and oil constituents are described, together with notes on modern technique. Absorption data on the following oils are given, the figures in parentheses denoting absorption maxima in μ : wintergreen (307, 237), almond (281, 243.5), clove (281), thyme (277), cinnamon, cassia (286), sassafras (285, 233), lemon (311), mustard (238), sage (232). Celery, flag, wormseed, and mace oils

show little selective absorption. The available data on pure substances occurring in essential oils are summarised. R. A. MORTON.

See also A., Sept., 1924, Effect of ultra-violet light on toxins and antitoxins (ARTHUR and COLLINS). 1066, Amino-alcohols (HARTUNG and MUNCH). 1073, Synthesis of ephedrine and of $\alpha\beta$ -diketones (COLES and others). 1084, Determination of methylene-blue (FRANÇOIS and SEGUIN). 1085, Alkaloids of Chinese *Corydalis ambigua* (CHOU). 1085—6, Lobelia alkaloids (WIELAND and others). 1086, Synthesis of lobelia alkaloids (SCHEUING and WINTERHALDER). Phenol bases from *Angostura* bark (SPÄTH and PAPAIOANOU). 1087, Quaternary bases from *Berberis vulgaris* (SPÄTH and POLGAR). Synthesis of aporphine alkaloids (CALLOW and others). 1089, Bromination of natural alkaloids (MOREL and others). 1090, Bromination of novocaine (MOREL and others). 10-Chloro-5:10-dihydrophenarsazine and its derivatives (GIBSON and JOHNSON). Substances of phenarsazine type containing the acenaphthene nucleus (GIBSON and JOHNSON). 1104, Detoxication of chloroform (HOLTZ). 1105, Ouabain as physiological standard (SCHWARTZ and others). Pharmacological action of optical isomerides of ephedrine (CHEN and others). 1109, Purification of *Bacillus botulinus* toxin (HOSOYA and others). 1111, Testing of ovarian preparations (KOCHMANN). Preparation of secretin (TAKÁCS).

PATENTS.

Preparation of stable medicinal salts of acetylsalicylic acid. M. COPLANS (B.P. 315,330, 12.1.28).—Calcium or magnesium acetylsalicylate is stabilised by admixture of 5—25% of calcium or magnesium chloride. C. HOLLINS.

Manufacture of basic phenol alkyl ethers. I. G. FARBENIND. A.-G. (B.P. 288,555, 11.4.28. Ger., 11.4.27).—Phenols carrying in *p*-position an unsaturated alkyl group (e.g., allyl) and in the two *o*-positions other substituents are treated in presence of alkali with a dialkylaminoalkyl halide (e.g., β -diethylaminoethyl chloride), or with an alkylene dihalide followed by a secondary alkylamine. 3:5-Dimethoxychavicol β -diethylaminoethyl ether, b.p. 146—151°/5 mm., has a pronounced action on normal muscle. C. HOLLINS.

Preparation of derivatives of quinoline [atophan]. R. WOLFFENSTEIN (B.P. 304,655, 8.1.29. Ger., 23.1.28).—Atophan is converted into its chloride and condensed with urethane to give ethyl 2-phenylquinoline-4-carbamide-*N*-carboxylate, m.p. 167—168°. The methyl ester is similarly prepared. The products have analgesic properties. C. HOLLINS.

Manufacture of betaine thiocyanate. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 316,693, 8.5.28).—The product is prepared by combining betaine with thiocyanic acid or by the double decomposition of a betaine salt and a thiocyanate. L. A. COLES.

Recovery of camphor [from celluloid etc.]. R. VAN REES (B.P. 316,041, 29.8.28).—Caustic alkali is added to celluloid etc. in a vessel through which steam is being passed from below. The camphor distils with the steam. C. HOLLINS.

Manufacture of vitamin-D. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 316,803, 17.9.28).—Solutions of ergosterol in volatile solvents (*e.g.*, alcohol, ether) are irradiated at temperatures above 70°, under pressure if necessary. E. LEWKOWITSCH.

Manufacture of lipoids, particularly phosphatides, having different vitamin actions. "PHARMAGANS" PHARM. INST. L. W. GANS A.-G., W. KOLLATH, and H. MAGISTRIS (B.P. 315,340, 10.1.28. Cf. B.P. 311,436; B., 1929, 576).—The dialysed solution obtained by the process of B.P. 285,417 (B., 1929, 110) is fractionally treated with precipitants or solvents to separate the different vitamins. *E.g.*, if a silver or barium salt is added and the solution is made alkaline with baryta, the precipitate contains the antineuritic vitamin; addition of alcohol to the filtrate gives a precipitate containing vitamin-B, and vitamin-C may then be precipitated with benzene, leaving the antirachitic vitamin in solution. The products may with advantage be irradiated. C. HOLLINS.

Manufacture of growth-promoting substances for animal cells, suitable for use in medicine and surgery and for the cultivation of tissues. I. G. FARBERIND. A.-G. (B.P. 287,465, 19.3.28. Ger., 18.3.27).—Expressed cell juices or extracts, albumins or degradation products thereof are inoculated with *B. coli*, and after incubation for an optimum period (determined by trial) the micro-organisms are separated, and the solution is pasteurised and centrifuged. The clear liquid may, if desired, be evaporated to dryness in a vacuum, and is applied for accelerating the healing of wounds and treatment of fractures. C. HOLLINS.

Manufacture of sexual hormones. A. HOME-MORTON (B.P. 316,359, 7.5.28).—Thelykinins (cf. Loewe and others, A., 1927, 282) of therapeutic value equal to that of the ovary and placenta hormones are obtained by extracting bile with ether, benzene, or chloroform. L. A. COLES.

Manufacture of chloro-iodides of 2-aminopyridine. M. DOHRN and R. DIRKSEN, ASSTS. to CHEM. FABR. AUF ACTIEN (FORM. E. SCHERING) (U.S.P. 1,723,457, 6.8.29. Appl., 5.1.27. Ger., 15.1.26).—See B.P. 264,508; B., 1927, 572.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Binder for priming compounds. L. W. JORDAN, ASST. to WINCHESTER REPEATING ARMS CO. (U.S.P. 1,718,358, 25.6.29. Appl., 7.2.28).—The binder comprises essentially an alkali caseinate or casein and an alkali salt. A mixture of casein, sodium tetraborate, gum tragacanth, thymol, and alcohol may be used. F. G. CLARKE.

Dinitrophenol (U.S.P. 1,723,761).—See III.

XXIII.—SANITATION; WATER PURIFICATION.

Oxidation of sewage when hard or soft waters are the carriers. J. E. PURVIS (J. Roy. San. Inst., 1929, 49, 587).—The rate of oxidation by aeration is smaller the larger is the inorganic salt content of the sewage.

CHEMICAL ABSTRACTS.

Ultramicroscopic studies of colloids in water. C. H. CHRISTMAN (J. Amer. Water Works' Assoc., 1929, 21, 1076—1080).—An ultramicroscopical examination of the behaviour of colloidal particles in a number of samples of water under the influence of a direct electric current showed that the particles were all negatively charged, and that the rate of migration thereby induced varies and is a function of the charge carried, but is independent of the p_H value of the raw water. C. JEPSON.

Methylene-blue and bromocresol-purple in differentiating bacteria of the *colon-aerogenes* group. J. F. DOMINICK and C. J. LAUTER (J. Amer. Water Works' Assoc., 1929, 21, 1067—1075).—The time required to obtain the *B. coli* index number of a sample of water may be reduced from 3 days to 24 hrs. or less by means of a lactose-peptone direct medium containing methylene-blue and bromocresol-purple, the preparation of which is described, thus greatly diminishing the danger of admitting inadequately purified water into the service mains. C. JEPSON.

Preammoniation [of water] at Springfield, Illinois. C. H. SPAULDING (J. Amer. Water Works' Assoc., 1929, 21, 1085—1096).—The efficiency of chlorination of a water having a normal p_H value and a low ammonia content may be increased by preammoniation, which is also an effective means of preventing chloro-tastes and also chlorophenol tastes in a lime-softened water. To obtain maximum sterilisation when the p_H value is high, a contact period of 8 hrs. is desirable. The addition of ammonia permits the presence of sufficient residual chlorine to overcome any subsequent contamination without its presence being revealed by taste. C. JEPSON.

Prevention of boiler corrosion by addition of alkali. A. SPLITTGERBER (Chem. Fabr., 1929, 253—256).—A review of recent literature. A. R. POWELL.

See also A., Sept., 1929, **Determination of bromine in sea-water and estuary waters** (KOGAN).

PATENTS.

Treatment of sewage by the activated sludge process. J. C. DALLAS and M. WILSON (B.P. 316,795, 4.9.28).—The circulation of sludge and sewage is effected by means of air lifts operating from the apex of an inverted pyramid. In order to obtain further aeration the mixed liquor as it emerges from the mouth of the lift may be spread in thin sheets over perforated or riffled plates or broken up into fine drops or spray by special fittings attached to the air lift. When the sides of the tank above the conical bottom are made concave better results are obtained. C. JEPSON.

Purification and softening of water. W. EVANS (B.P. 316,023, 10.8.28).—In the softening of water by such materials as soda ash and lime, barium aluminate is added simultaneously or subsequently, whereby the removal of salts is effected more rapidly and more completely, whilst any suspended matter is carried down by the coagulated salts. H. J. DOWDEN.

Prevention and removal of boiler scale. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 315,287, 10.4.28).—Humic salts, especially those of the alkaline bases, are added to the feed water. B. M. VENABLES.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCT. 25, 1929.

I.—GENERAL; PLANT; MACHINERY.

Evaporation at low temperatures. E. JANTZEN and H. SCHMALFUSS (Chem. Fabr., 1929, 387—391, 399—401).—An apparatus for the rapid evaporation of liquids under low pressure near 0° has been devised. The still is of the type previously described (B., 1929, 77), but the vapours are condensed on ice. The temperature and pressure at which various liquids will boil in the apparatus have been calculated. R. CUTHILL.

All-glass evaporator. D. F. OTHMER (Ind. Eng. Chem., 1929, 21, 876—877).—The evaporator consists of seven lengths of 2.5 cm. pyrex tubing with return bends welded in place enclosed in an 8-in. pipe forming the steam space. The concentrate trap and receiver is formed of two 5-litre flasks welded together and connected to an ejector, and the vapour passes to a coil condenser and vacuum pump. It serves for the concentration of corrosive liquids, and under favourable conditions gives an evaporation of 1 lb. of water per minute. C. IRWIN.

Determining the type of an emulsion. J. F. CARRIÈRE (Chem. Weekblad, 1929, 26, 413—416).—The differences in refractive index of aqueous liquids and oils may be utilised to determine the type by inspection of the emulsion illuminated from one side in the microscopic field. The method may be applied to any emulsion when the relative refractive indices of the components are known. S. I. LEVY.

Meaning and measurement of plasticity. E. KARRER (Ind. Eng. Chem., 1929, 21, 770—773).—The author defines plasticity as the susceptibility to and the retentivity of deformation; hence, a substance has unit plasticity (1 "pla") if it deforms a definite amount under some standard conditions when a force of 1 kg./cm.² acts on it for 1 sec. and the whole of this deformation is permanently retained. This conception is analysed mathematically, plasticity being expressed in terms of "softness" and "retentivity." The range of usefulness of plasticity measurements is indicated and values for the plasticity of some common materials are quoted. S. S. WOOLF.

Measurement of surface tension in the laboratory and works. H. CASSEL (Chem.-Ztg., 1929, 53, 479—480).—A modified apparatus for the determination of the surface tension of a liquid by the bubble method comprises a spherical flask connected to a manometer and provided with a short tube inclined upwards and terminating in a cone-shaped capillary opening instead of the usual capillary tube pointing vertically downwards. The method of standardising and using the instrument is described. A. R. POWELL.

PATENTS.

Furnace. J. W. COX, Assr. to G. R. METCALF and E. E. WALKER (U.S.P. 1,724,455, 13.8.29. Appl., 12.12.24).—A furnace has two combustion and melting chambers, with individual fuel supply and a common stack. Flues and dampers are provided so that the waste gases from either melting chamber can pass to the stack either direct or through the other chamber. B. M. VENABLES.

Heating-furnace apparatus. E. WIRZ, Assr. to A.-G. BROWN, BOVERI & Co. (U.S.P. 1,723,319, 6.8.29. Appl., 18.2.26. Ger., 24.2.25).—A furnace, such as one for the heat treatment of discrete articles, with heating elements (preferably electrical) above and below the space for the articles, is provided with a number of girders or walls protecting the lower heating elements and spaced apart so that a number of elements of a lifting carriage may enter the spaces and deposit the goods on the girders, the carriage being withdrawn during the heating. The floor actually supporting the goods may thus be kept as light as possible or even omitted altogether, so that the heat capacity of the support is reduced to a minimum. B. M. VENABLES.

Gas producer and boiler plant for liquid-heating and/or steam-raising purposes. T. R. WOLLASTON (B.P. 317,189, 30.6.28).—A single gas producer supplies gaseous fuel to a number of boilers and the producer itself is provided with a jacket that serves as a water heater. Valves are provided for the independent regulation of the gas and fluid supplies to each boiler. In an example all the heaters are connected to one main circulation for hot water. B. M. VENABLES.

Heat exchanger. H. HENDERSON and J. G. GLASGOW, Assrs. to GULF REFINING Co. (U.S.P. 1,724,351, 13.8.29. Appl., 4.9.26).—The tubes of a heat exchanger are expanded, at one end, into a tube plate which is provided with flanges and a cover plate, the space thus enclosed forming the header. The tube plate is not rigidly attached to the casing, but a bellows-like device is attached to the casing on the one side and the cover plate on the other. The tube plate at the other end may be rigidly attached to the casing. B. M. VENABLES.

Carrying out of gas reactions. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 317,996, 29.8.28).—Vessels used for effecting reactions at high temperatures between materials in the gaseous phase are protected by adding to the materials small quantities of non-reacting substances non-volatile at the temperature of the reaction and capable of forming a liquid film on the interior of the apparatus; e.g., 5% of paraffin wax is

added to middle oil prior to entry into apparatus for hydrogenation at a high temperature and pressure.

L. A. COLES.

Carrying out endothermic chemical reactions of various kinds. F. KRUPP GRUSONWERK A.-G. (B.P. 309,942, 1.6.28. Ger., 18.4.28).—In a process for the volatilisation of metals, for the reduction of sulphates to sulphides, etc., in a rotary tube furnace, in which solid fuel is added to the charge and by combustion with oxygen taken from the materials produces most of the heat required, the additional air and/or gas necessary, the products of combustion, and the charge of material all travel in the same direction in the furnace, thus preventing re-oxidation of the products.

B. M. VENABLES.

Spray dryers. W. ALEXANDER (B.P. 317,166, 9.6.28).—The air is admitted in a whirling manner to the top of a cylindrical chamber, an inner supply of cold and an outer supply of hot air being usually provided; the centrifugal spraying disc for the liquid is also at the top. The air is exhausted axially at the lower part of the cylindrical chamber but above the conical hopper bottom. Conical baffles surround the air outlet and help to guide the dried dust into the hopper and prevent disturbance of the outgoing air. B. M. VENABLES.

Drying, heating, or cooling machines. W. G. and F. R. SIMON (B.P. 317,581, 25.6.28).—A dryer or similar machine, of the type in which a reel with lifting and agitating bars rotates in a casing and surrounds a bundle of tubes conveying heating or cooling fluid, is provided with hammers pivoted on a fixed portion of the machine and raised either by contact with the reel or by separately driven means, which drop upon the reel and keep it free from accretions. B. M. VENABLES.

Non-conducting covering for pipes. G. T. DE KLERK (B.P. 316,202, 24.10.28. Belg., 25.7.28).—Asbestos with or without air spaces is placed in contact with the pipe, and paper or cardboard, alternately plain and corrugated and glued together, is used for the cooler outer layers. B. M. VENABLES.

Thermal insulation. E. SCHMIDT and E. DYCKERHOFF (B.P. 317,678, 3.11.28).—A form of thin metal sheet casing and supporting structure for use in conjunction with insulating material, which may also be composed of thin metal sheets, is described.

B. M. VENABLES.

Single-roll grinding mills. A. E. G. MACCULLUM (B.P. 316,907, 5.4.28).—A roller mill with single grinding block is provided with a narrow scraper blade within the feed hopper which removes the material from the surface of the roll after it has passed under the block; the partly ground material overflows the blade and falls on the roll to be again ground by the same block.

B. M. VENABLES.

Pulverising apparatus. F. H. DANIELS, Assr. to RILEY STOKER CORP. (U.S.P. 1,724,072, 13.8.29. Appl., 21.8.26).—A roll crusher is situated at the inlet to an impact pulveriser. One roll can yield to permit hard masses to pass; these are caught in a special pocket and not admitted to the pulveriser. B. M. VENABLES.

Pulverisation and separation of cereals, minerals, etc. H. A. LIVINGSTONE (B.P. 316,922, 5.5.28).—Up-

wardly extending from a disintegrator chamber is an exhaust trunk which is divided by a triangular partition into two conduits up one of which the ground material is flung by the action of the beaters and down the other branch oversize returns to the disintegrator, a separating zone being situated at or near the apex of the triangle. A second triangular baffle is situated above the first, forming a second separating device. Adjustable flaps are provided to regulate the action.

B. M. VENABLES.

Apparatus for pulverising material. W. T. DOYLE, Assr. to STURTEVANT MILL CO. (U.S.P. 1,723,726, 6.8.29. Appl., 19.11.28).—In a pulveriser operating with two or more stages (such as a ball mill with more than one compartment) and provided with an air separator, the oversize from the separator normally passes for re-grinding to an intermediate stage, but any excess over a certain definite amount passes through another passage to mix with the original feed; this excess operates a device which reduces the amount of the original feed, preferably by reducing the speed of the motor operating the feeder. B. M. VENABLES.

Means for concentrating or separating solid substances. A. A. LOCKWOOD (B.P. 317,141, 15.5.28).—A sloping impermeate table is reciprocated and is also subject to percussion by means of loosely-mounted devices attached to it. Examples of such devices are balls in boxes, or pendulums operated either solely by the reciprocation of the table or by engagement with fixed tappets to an external rigid support. B. M. VENABLES.

Dust separator and collector. T. S. MONAGHAN, Assr. to G. E. OLMSTED (U.S.P. 1,723,703, 6.8.29. Appl., 24.5.27).—A cyclone separator is provided with cylindrical, inverted frusto-conical, and upright conical baffles, intended to improve the separation of dust and to direct the clean gas into the axial outlet.

B. M. VENABLES.

Mixer. C. W. HOTTMANN (U.S.P. 1,723,620, 6.8.29. Appl., 5.5.26).—The mixer is similar, in principle, to a Root's blower, but with the shafts vertical, and scraping and mixing bars instead of solid impellers.

B. M. VENABLES.

Mixer. A. CROSSMAN (U.S.P. 1,724,740, 13.8.29. Appl., 21.8.24).—A mixer for solid and fluid constituents is constructed so that the former enters through an inclined chute above a beater, which rotates in a direction to drag the solid material below the surface of the fluid which is admitted through a separate pipe. The mixture is exhausted through a separate nozzle.

B. M. VENABLES.

Mixing machine. S. SNYDER, Assr. to SPROUT, WALDRON & CO. (U.S.P. 1,724,808, 13.8.29. Appl., 26.2.29).—Liquid is injected downwardly into the mixing mass by means of a hollow arm rotating with the agitator blades and situated above them.

B. M. VENABLES.

Blending or mixing. H. E. VAN DEN BERGH (B.P. 317,128, 12.5.28).—The material, such as tea, is charged into an upper chamber having a floor composed of a number of louvre-like slats which are kept closed during the charging but afterwards opened to allow the material to fall into the lower chamber. From the lower chamber

the material falls by gravity (under control) to an elevator which returns it to the upper chamber. This process is repeated as desired. B. M. VENABLES.

Centrifugal emulsifier or homogeniser. E. G. WAIT (B.P. 316,959, 7.5.28).—The material is supplied to the centre of a rapidly rotating dish which is so contoured that as the centrifugal force increases with increase of radius so also does the resistance to flow of the film of liquid, i.e., the surface of the dish rises with increasing slope outwardly, finishing in some cases with a slightly in-turned edge. The centre of the bowl may be formed in a curve running to a point at the axis to minimise shock at entry. B. M. VENABLES.

Agitating or mixing liquids, slimes, etc. H. J. COLLINS (B.P. 317,529, 19.5.28).—Liquid flows from an upper tank through a regulating valve to an air-tight tank provided with a non-return air-outlet valve and with a float-controlled siphon which empties the air-tight tank rapidly after the liquid has attained a certain height. The liquid may be caught in a lower storage tank and pumped back to the upper one for re-use. The air-tight tank has one or more pipes connecting with bells or other devices immersed in the liquids to be agitated. While the siphon is operating, a partial vacuum is produced and the liquids will rise in the bells; on completion of the siphon action air enters the air-tight tank and the liquid in the bells falls suddenly and causes agitation. B. M. VENABLES.

Causing solids to be penetrated by or impregnated with liquids or solutions of various substances. H. J. COLLINS (B.P. 317,427, 16.5.28).—The solids (such as hides) are placed in a strong container which is then completely filled with liquid by means of a pump. When a desired mean pressure is reached the reciprocation of the pump is continued, but by opening a by-pass from the outlet back to the pump chamber delivery of liquid ceases, the effect of the pump being to cause pulsations of pressure. The motion of the plunger may be suddenly accelerated towards the end of the delivery stroke. B. M. VENABLES.

Filtering system. R. AUDUBERT (U.S.P. 1,723,997, 13.8.29. Appl., 7.3.28. Fr., 27.10.27).—The liquid is forced through a mass of filtering material having capillary passages, under a head of 5–100 cm. of water and at a rate low enough to allow the electrical surface charges to attract, thus preventing any impurities from passing. B. M. VENABLES.

Assembling of filter stack units. V. R. HEFTLER (B.P. 303,153, 17.12.28. U.S., 30.12.27).—Starting with a ribbon of the filter material, the discs (usually of two alternate shapes) are formed by punching and correctly assembled as a pack by purely mechanical means: B. M. VENABLES.

Thickener. E. J. SWEETLAND, ASSI. to OLIVER UNITED FILTERS, INC. (U.S.P. 1,724,436, 13.8.29. Appl., 24.1.24).—In a vacuum filter, in which the filter elements are submerged in a tank containing the prefilter, the outlet pipe for filtrate and the accompanying vacuum-producing device are below the level of the prefilter in the tank. B. M. VENABLES.

Centrifugal separator. J. C. BUCKBEE (U.S.P.

1,724,254, 13.8.29. Appl., 11.4.28).—An approximately spherical bowl has an upwardly-extending, hollow driving shaft and a comparatively large overflow mouth at the bottom. Within the hollow shaft is a feed tube which carries on its lower end two baffles, the upper one to distribute the feed material; the lower, which has apertures near the axis for emission of lighter liquid, is shaped to the bowl and more or less closes the outlet mouth. The tube and baffles are not prevented from rotating with the bowl. B. M. VENABLES.

Centrifugal separator [for sewage etc.]. T. TOHLIN (U.S.P. 1,723,495, 6.8.29. Appl., 25.1.28).—The material, such as sewage, from which it is desired to separate a proportion of clear water, enters the lower small end of a frusto-conical bowl which is rotated by a shaft extending upwards through the outlet for clear water; the arms connecting the shaft to the bowl serve also to propel the sewage. The thickened sewage flows over the upper large end of the bowl into a surrounding casing and is exhausted downwardly; the clear water leaves axially at the top through a pipe surrounding the driving shaft, the pipe being provided with a bend through which the driving shaft emerges. B. M. VENABLES.

Centrifugal separators. AKTIEBOLAGET SEPARATOR (B.P. 306,948, 14.2.29. Swed., 29.2.28).—A sight glass is described for use on a separator of the type which emits dirty feed fluid when the bowl requires cleaning. B. M. VENABLES.

Apparatus for washing or scrubbing gas. T. A. CLAPHAM (B.P. 317,412, 10.4.28).—A scrubbing tower is divided into a number of stories, each having a concave tray at the bottom to catch the washing liquid. The liquid is lifted from the tray (of every storey) and spread over the top of the filling in the same storey by means of a centrifugal device attached to a vertical shaft serving all the stories. There may be a continuous feed of a minor quantity of liquid to the top of the tower. B. M. VENABLES.

Purification of furnace and like gases. D. RADULESCU (B.P. 289,825, 2.5.28. Roum., 2.5.27).—Supersaturated or condensing water vapour is added to the gas, the mixture is subjected to rapid whirling (by which means the collected particles are flung out), and then passed through a centrifugally produced veil of water; these three steps are repeated as often as may be necessary. The apparatus described comprises an inclined cylindrical conduit through which the gas passes and a rapidly rotating shaft, co-axial with the conduit, provided with a number of radial stirrers. To the odd-numbered compartments thus formed condensing vapour is supplied; in the even-numbered ones water is directed against the rotating blades. B. M. VENABLES.

Analysis of gaseous mixtures. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE & L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 290,259, 11.5.28. Fr., 11.5.27).—The apparatus is operated by means of a Woulff's bottle containing absorption liquid which can be put into communication with a source of inert gas at moderate pressure, say 500 mm. of water. The gas is blown through and collected in a measuring burette at atmospheric pressure; the burette is bounded top and bottom

by capillary tubes, the lower one being always at atmospheric pressure when measurements are being effected. After absorption of a constituent of the gas and return of the remainder to the burette, the latter will be partly full of liquid, and the liquid in the upper capillary (and the gas) will be under pressure less than atmospheric by a depression which is equal to the height of the liquid in the burette. This height is balanced automatically by a depression in a hydrostatic tube which is connected to the upper capillary of the burette by way of the absorption chamber, all connecting passages being full of liquid. This depression is readily indicated by a float in the hydrostatic tube, and the level may be recorded by a stylus operating on a chart which is graduated in accordance with the shape of the burette; that shape is so chosen that the upper level of the liquid (after absorption) stays in a narrow but not capillary part of the burette.

B. M. VENABLES.

Rotary filter or strainer. F. W. BRACKETT (U.S.P. 1,726,608, 3.9.29. Appl., 29.11.26. U.K., 28.10.26).—See B.P. 284,403; B., 1928, 248.

Recovery of reaction products from gases treated with electric arcs. O. EISENHUT, ASST. to I. G. FARBENIND. A.-G. (U.S.P. 1,726,547, 3.9.29. Appl., 29.12.26. Ger., 30.12.25).—See B.P. 263,859; B., 1928, 415.

Rotary refrigerating machine. M. AUDIFFREN and A. SINGRUN (B.P. 299,724, 29.10.28. Fr., 28.10.27).

Absorption refrigerating apparatus. ELECTROLUX, LTD., ASSEES. of A. LENNING (B.P. 297,057, 12.9.28. U.S., 13.9.27).

Absorption refrigerating apparatus. H. D. FITZPATRICK. From C. F. M. VAN BERKEL (B.P. 317,340, 12.12.28).

Mechanical device for regulating the quantity and composition of a gas mixture. I. G. FARBENIND. A.-G. (B.P. 296,063, 23.8.28. Ger., 24.8.27).

Gas washer (U.S.P. 1,718,988).—See X. **Humidity of gases** (B.P. 317,306).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coke-oven heat balances. V. FEODOROV (Chim. et Ind., 1929, 22, 231—248).—In considering the heat balance the net thermal effect of the reactions which occur during carbonisation can be neglected as they are within the limits of experimental error. The loss of heat unaccounted for by other observers (cf. B., 1923, 389 A, 436 A) is considered to be largely due to losses of combustible gas by percolation through the walls of the heating flues and to partial combustion within the oven itself; these, in turn, depend on the temperature and duration of the coking process. Attention to these factors indicates the lines along which improvements in coke-oven efficiency may be achieved.

C. JEPSON.

Correlation of physical and chemical properties of cokes with their value in metallurgical processes. II. W. T. K. BRAUNHOLTZ, G. M. NAVE, and H. V. A. BRISCOE (Fuel, 1929, 8, 411—437; cf. B., 1928, 432).—Investigation of the "Micum" trommel

test for determining the mechanical strength of coke shows that this is essentially a modified shatter test in which the force of impact alone plays a significant part. Determinations of the volatile matter in coke give results which vary with the method of carrying out the test, and particularly with the time of heating; evolution of volatile matter at 950° continues at an appreciable rate for at least 30 min. Marked differences in the weight of coke occupying 1 cub. ft. are observed according to the origin of the coal carbonised and the carbonising conditions. Full-scale experiments have shown that (1) cokes from an uncompressed coal are more resistant to shatter, but have a lower volume weight and a higher porosity than those made from compressed charges; (2) variations in the width of the oven and the carbonising time have little effect on the quality of the coke; (3) although samples of coke from different parts of the oven show some differences, tests on samples from the middle of the charge are in good agreement with those on average samples from the same ovens; (4) "soaking" after completed carbonisation slightly lowers the shatter indices and porosities; (5) blending anthracite with a coking coal may cause an improvement in the shatter test results. A reliable indication of the quality of a coke produced from a given coal under given conditions is obtained by carbonising 1—2 cwt. of the coal in tins embedded in full-scale oven charges of another coal. The behaviour of a series of cokes in a full-size cupola has been compared. The relative melting efficiencies of the cokes correspond with their relative efficiencies as determined by the "combustibility" test. A slightly modified form of the laboratory apparatus has been devised for the latter test, and experiments have been carried out on the influence of mode of production, ash content and composition, and graded size on the combustibility of coke.

A. B. MANNING.

Influence of size of coke on degree of combustion in front of the tuyères. W. MATHESIUS (Stahl u. Eisen, 1929, 49, 1220—1221).—The depth of penetration of the blast into the furnace is chiefly dependent on the number of obstructions which cause deviations in its direction, and hence on the size of the pieces of coke used. The correct size of the coke for any furnace depends on the reactivity of its surface and the rate at which the furnace is run. For the production of white iron, where a rapid rate is required, small coke gives the best results, whereas for the production of grey iron, which is a slower process, larger pieces of coke should be used.

A. R. POWELL.

Action of iron catalysts on mixtures of carbon monoxide and hydrogen. E. AUDIBERT and A. RAINEAU (Ann. Off. nat. Combustibles liquides, 1928, No. 3; Ind. Eng. Chem., 1929, 21, 880—885).—Ferric oxide, unlike reduced iron, yields liquid as well as gaseous products when used as catalyst for the interaction of carbon monoxide and hydrogen at 150 atm. and 250°. The yield of liquid products quickly falls to zero with the reduction of the ferric oxide. The reduction of the ferric oxide is prevented to some extent by the addition of alkali, or by the use of a gas mixture containing excess of hydrogen. The addition of alkali salts, or copper, is of no benefit,

neither is the use of lower pressures and temperatures practical, nor the replacement of the iron by manganese. In all these cases carbon is deposited on the catalyst, and leads to irregular action. Better results were obtained by converting the ferric hydroxide into phosphate or borate by addition of acid, adding a solution of the nitrates of copper and manganese, precipitating as a paste by soda, and then adding potassium carbonate. With this no carbonaceous deposit was formed, and a yield of 15–17% by wt. of organic liquids was obtained. The remainder is gaseous hydrocarbons, carbon dioxide, and water vapour. About two thirds of the organic liquid distil below 180°, this including a large proportion of aliphatic alcohols which must be recovered from the aqueous layer. These results were obtained at 380–450° with a space velocity of about 10,000 and at 150 atm. pressure (cf. B., 1928, 920).

C. IRWIN.

Ignition of firedamp by the heat of impact of metal against rock. M. J. BURGESS and R. V. WHEELER (Safety in Mines Res. Brd., Paper No. 54, 1929, 25 pp.; cf. B., 1929, 41).—Mixtures of methane and air containing 7.5–9.0% of methane could not be ignited by the sparks produced on pressing a steel block against a revolving carborundum wheel. The sparks, however, were able to set fire to thin tissue paper, and thereby indirectly to ignite the gas. Similar tests with the sparks produced from steel blocks pressed against a revolving wheel of hard steel also failed to ignite the gas, either directly or by allowing the sparks to fall on tissue paper, oiled paper, or coal dust. The firedamp was, however, ignited by the heated edge of a steel rod pressed against a rapidly revolving wheel made from a hard quartzitic sandstone. Tests under similar conditions with wheels made from "Derbyshire grit" or carborundum failed to cause ignition. No ignition was obtained in tests made by pressing various hard rocks against a steel disc in the manner of the "steel mill." Tests with a cutting disc produced ignition with certain hard rocks; very little sparking was produced with any of the rocks, ignition being due to a heated area of rock produced by the cutters. Tests with coal-cutter picks set in the circumference of a rapidly revolving wheel caused the ignition of firedamp through the heating of the rock surfaces through which they cut. A chain coal-cutter cutting through hard rock in an experimental chamber filled with an explosive methane-air atmosphere also produced ignitions.

A. B. MANNING.

Extinction of flames. W. P. JORISSEN and B. L. ONGKIEHONG (Chem. Weekblad, 1929, 26, 433–436).—Analyses are given of the gases remaining when various inflammable substances cease to burn in air, and of mixtures of oxygen and nitrogen, and of oxygen, nitrogen, and carbon dioxide adjusted just to extinguish burning materials plunged into them. The literature is reviewed.

S. I. LEVY.

Flash point determination. J. TERPUGOFF (Petroleum, 1929, 25, 1161–1163).—The method and apparatus of Schlüter (B., 1928, 325) have been examined and compared with those of Sommer and Runge and of Marcusson. The Pensky-Martens apparatus (closed flash point) is frequently too sensitive for commercial

heavy oils, since differences of many degrees are often observed in oils which are commercially acceptable as of the same quality.

S. I. LEVY.

Iodine values of mineral oils. B. M. MARGOSCHES, B. KRAKOWETZ, and F. SCHNABEL (Petroleum, 1929, 25, 1179–1186).—After a critical review of the literature, an account is given of comparative determinations, using the Hübl method, and the more recent "rapid" method (cf. Margosches, Hinner, and Friedmann, B., 1924, 640), in which the oil is emulsified with water and an alcoholic solution of iodine. The conditions under which the latter gives the same results as the Hübl method were determined. The heavier oils cannot be made to emulsify satisfactorily with alcohol alone, and it is therefore necessary first to precipitate asphalt by addition of ether or acetone, and to emulsify the asphalt-free oil with a mixture of alcohol with ether or acetone for the determination; concordant results, however, could not be obtained with heavy fractions.

S. I. LEVY.

Emulsions of hydrocarbons. MEUNIER. **Effect of hydrogen ions on emulsions.** KRANTZ and GORDON. **Solvents for waxes.** PICKETT.—See XII.

PATENTS.

Coke oven. M. KELTING (U.S.P. 1,726,494, 27.8.29. Appl., 13.7.25. Ger., 18.7.24).—A heating chamber for a coke oven has alternate uptake and downtake conduits connected together at their top and bottom ends, respectively. A burner with an air nozzle is situated at the lower end of each uptake conduit, and an outlet for discharging part of the products of combustion at the lower end of each downtake conduit.

A. B. MANNING.

Retort ovens for low-temperature carbonisation. COMP. GÉN. DE DISTILLATION ET COKEFACTION À BASSE TEMPERATURE ET MINÈRE "HOLCOBAMI" SOC. ANON., ASSECS. OF INTERNAT. HOLDING DE DISTILLATION ET COKEFACTION À BASSE TEMPERATURE ET MINÈRE "HOLCOBAMI" SOC. ANON. (B.P. 308,760, 28.9.28. Ger., 28.3.28).—The ovens described in B.P. 229,880 (B., 1925, 245) are made more efficient by subjecting the retorts to indirect heating, i.e., the heating gases are ignited in a separate combustion chamber and the hot gaseous products are passed over the retorts, which are constructed in staggered formation.

C. B. MARSON.

Manufacture of fuel. L. KIRSCHBRAUN (U.S.P. 1,725,198, 20.8.29. Appl., 14.3.21. Renewed 17.3.26).—A combustible agglomerate is made by mixing powdered coal with an emulsion containing an argilliferous emulsifying agent and a bituminous base, and then breaking down the emulsion so that the adhesive agent binds the coal into the required agglomerate.

A. B. MANNING.

Complete gasification of fuels. O. GROSS (B.P. 289,080, 15.3.28. Ger., 23.4.27).—Fuel with the usual amount of volatile constituents is gasified, without previous coking, for the production of hydrogen or a gas mixture containing it suitable for the preparation of synthetic ammonia etc. This is effected by gasifying the fuel in a mixture of oxygen (and/or air) and

water vapour so that the nitrogen content of the resulting gas is not greater than that required for ammonia synthesis; the hydrocarbons are converted by oxygen or oxygen and water vapour into carbon monoxide and hydrogen.

C. B. MARSON.

Gasification of bituminous coal. J. M. RUSBY, ASSR. to U.G.I. CONTRACTING Co. (U.S.P. 1,725,739, 20.8.29. Appl., 7.6.23).—In a retort for the complete gasification of coal containing a descending fuel bed comprising coal at the top and coke below, hot zones are established in the bottom layer of coke and in the layer immediately supporting the coal by admitting independent air blasts to each zone; during this stage the blast gases are excluded from the coal, which is subsequently carbonised by a current of blue water-gas formed by the introduction of steam into the lower hot zone and strongly heated during its passage through the upper hot zone.

L. A. COLES.

Destructive hydrogenation of carbonaceous materials. A. J. VAN PESKI (B.P. 315,780, 18.1.28).—The materials are heated with hydrogen in the presence of catalysts such as calcium, iron-tungsten, or nickel carbides in an autoclave at 460° and 45 atm. (cf. B.P. 315,193; B., 1929, 770).

C. B. MARSON.

Gas producer. H. F. SMITH, ASSR. to GAS RES. Co. (U.S.P. 1,724,525, 13.8.29. Appl., 30.1.22).—A gas producer adapted to be assembled in the inverted position comprises a producer shell open at the lower end, a ring supported within the shell, and a lining extending beneath the ring and supported thereby when the shell is in the inverted position. The lining can be inserted into the shell through the lower end, for which a closure plate is provided.

A. B. MANNING.

Gas retorts. H. KOPPERS, ASSR. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,725,148—9, 20.8.29. Appl., 8.7.21. Renewed [B] 10.3.27. Ger., [A] 17.11.19, [B] 2.2.20).—(A) A gas retort setting consists of a number of inclined retort chambers surrounded by heating flues, below which are regenerators. The latter are connected to intermediate chambers which in turn are connected by ducts to the heating flues. Movable nozzles which can be operated from outside the structure and which communicate with the ducts leading to the flues can slide along guides in these chambers to a point adjacent to the ends of the flues. (B) A setting of retorts is arranged in a common flame chamber which is partitioned into two groups of flues, for alternate inflow and outflow. The partition extending into the crown space of the chamber is provided with a port which determines the place of flow from the inflow to the outflow side. Regenerators are provided connected, respectively, to the inflow and outflow flues.

A. B. MANNING.

Desulphurisation of gas. R. M. CRAWFORD (U.S.P. 1,724,909, 20.8.29. Appl., 3.7.26. Renewed 23.1.29).—The gas is washed with a solution containing an alkali carbonate derived from the decomposition of an alkali phenoxide.

A. R. POWELL.

Apparatus for hot purification [desulphurisation] of gases. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 298,236, 5.10.28. Fr., 6.10.27).—

Distillation, producer, and other gases are desulphurised by passing them over the purifying material which is mounted on a carrier of porous material and is situated in chambers of annular cross-section. The heat liberated during regeneration is thereby easily dissipated owing to the large radiating surfaces of the annular compartment, which may be of a width progressively increasing from the zone which, during regeneration, is at the highest temperature.

C. B. MARSON.

Treating coke-oven gases and like gases. UNION CHIM. BELGE SOC. ANON. (B.P. 316,174, 17.9.28. Belg., 24.7.28).—The gases are treated with an ammoniacal solution which not only contains ammonia recovered from the condensate, but is made alkaline by the absorption of anhydrous ammonia. The resulting solution is caused to react with a suspension of calcium sulphate as described in B.P. 262,320 (B., 1927, 107). The anhydrous ammonia used for making the ammoniacal solution is utilised as a cooling agent for condensing benzol from the gas.

A. B. MANNING.

Recovery of gas tars from their emulsions with water. R. R. RIPLEY and S. C. SCHWARZ (U.S.P. 1,724,222, 13.8.29. Appl., 22.4.24).—A small proportion of a demulsifying agent is added to the emulsion and the mixture is rapidly heated to its b.p. under atmospheric pressure. The heating is continued, under correspondingly increased pressure, to a temperature below that at which appreciable cracking occurs, and the mixture is maintained thereat until the water particles coalesce. The heating is then discontinued and the mixture allowed to separate by gravity.

A. B. MANNING.

Obtaining light hydrocarbon oils from tar sands. A. RULE, H. G. WATTS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 317,346, 11.5.28).—Light hydrocarbons are produced by the destructive hydrogenation of carbonaceous material which has been extracted from tar sands. The light oil or the middle oil from a previous hydrogenation forms a suitable solvent for the extraction, which is preferably carried out at temperatures above the normal. The middle oil produced may be separately treated, either by destructive hydrogenation or by cracking, to produce a further quantity of light oil.

A. B. MANNING.

Heat treatment of oil shale or similar materials. R. V. WHEELER, A. P. and A. H. PEHRSON (B.P. 316,652, 2.5.28).—To destroy or minimise the adhesive or sticking properties of the materials when heated, they are subjected to a low-temperature preheating treatment at 100—150°, and through them are passed heated gases (other than air) containing at least 10% O₂ (e.g., flue gases) for $\frac{1}{2}$ —2 hrs. The shale is preferably subjected to a tumbling-over action during the treatment.

W. S. NORRIS.

Hydrogenating and cracking organisation [for mineral oils]. M. J. TRUMBLE (U.S.P. 1,725,320, 20.8.29. Appl., 24.12.24. Renewed 12.11.28).—The apparatus comprises an equilibrium chamber having an outlet for lighter fractions, a superposed hydrogenating chamber, and means, independent of one another, for supplying fresh material and for circulating the heavier fractions through the two chambers.

L. A. COLES.

Decomposition of hydrocarbons into those containing fewer carbon atoms. A. J. VAN PESKI (B.P. 315,890, 18.1.28).—Decomposition of hydrocarbons, without the use of hydrogen, is effected by passing them over catalysts consisting of one or more of the elements sulphur, antimony, arsenic, selenium, or tellurium, and/or of compounds of sulphur, antimony, or arsenic, with halogens or halogen compounds at ordinary, low, or high pressures and at ordinary or increased temperatures. C. B. MARSON.

Converting hydrocarbons of high b.p. into those of low b.p. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 315,991, 7.7.28).—Mineral oils, tars, and their hydrogenation products are converted into hydrocarbons of low b.p. by subjecting them, at 410–600° and in the presence of gases or vapours and of suitable catalysts, to the action of aldehydes or ketones which contain more than 3 atoms of carbon in the molecule. C. B. MARSON.

Conversion of hydrocarbons of high b.p. into others of low b.p. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 316,637, 26.4.28).—Cracking of heavy hydrocarbon oils is carried out in the presence of a catalyst consisting of or containing salts of metaphosphoric acid, particularly those of the metals of group VI; the catalyst may be carried on pumice, silica gel, etc. The process is preferably operated by passing the vaporised hydrocarbon, which may be mixed with nitrogen, carbon dioxide, or other inert gas, over the catalyst heated at 400–800°, under pressures up to 50 atm. In addition to liquid products of low b.p., gaseous unsaturated hydrocarbons are usually obtained. In an example, brown-coal tar, passed in vapour form at 600° over uranyl metaphosphate on pumice, yielded 35% of benzene (b.p. below 200°), 20% of cracked gases containing 35% of olefines, and a coke-free residue (b.p. above 200°). W. S. NORRIS.

Conversion of heavy into light hydrocarbon oils. E. C. R. MARKS. From DEUTS. ERDÖL A.-G. (B.P. 316,600, 30.4.28).—In a plant for cracking heavy mineral oil, the heating of the heating zone is subdivided into sections in such a way that from the point where the oil reaches the lowest cracking temperature (about 390°) the heating per unit length of section is increased by comparison with the preceding section; for this purpose a long and thin tube (e.g., 200 m. long, 60–80 mm. diam.), conveniently serpentine in form, is employed. At least two heating spaces are employed, and these may be associated. W. S. NORRIS.

Recovery of products from [distillation of hydrocarbons in] fractionating condensers. L. W. SNELL (U.S.P. 1,725,219, 20.8.29. Appl., 23.8.26).—Predetermined vapour temperatures are maintained at each end of one of the condensing zones of a fractionating condenser with the production of fractions of predetermined initial- and end-points, by discharging portions of two different fractions, after they have been cooled, into the vapours respectively entering and leaving the zone, the rates of flow of these portions varying in accordance with variations of the temperature of the vapours into which they are discharged. The remaining portions of the fractions are recovered separately. L. A. COLES.

Sweetening of hydrocarbon distillates. T. B. KIMBALL (B.P. 291,379, 25.5.28. U.S., 31.5.27).—Sulphur compounds (mercaptans) are removed from normally liquid hydrocarbons by vaporising the hydrocarbons and bringing them into countercurrent contact with non-oxidising, aqueous, alkaline solutions (e.g., of alkali hydroxides) heated to a temperature above the condensation point of the hydrocarbon vapours but below the b.p. of the alkaline solution at the pressure employed. W. S. NORRIS.

Refining of lubricating oil in hydrocarbon motors. W. B. CLIFFORD, ASSR. to CLIFFORD CORP. (U.S.P. 1,725,392, 20.8.29. Appl., 16.5.24).—An oil distilling chamber situated in the casing of an internal-combustion engine and heated by the exhaust gases is provided with means, controlled by the temperature in the chamber, for withholding the supply of oil until the distilling temperature is reached and with an oil discharge adapted to maintain a constant oil level in the chamber. L. A. COLES.

Apparatus for separating impurities from oils. DEUTS. WERFT A.-G. (B.P. 308,752, 21.6.29. Ger., 29.3.28).—The small water drops in the oil are conglomerated by allowing a current of the oil to impinge on a wall of rolled sheet iron, inclined to the direction of motion of the oil. A. B. MANNING.

Stabilisation of clay-treated [lubricating] oils. G. F. OLSEN, ASSR. to GEN. PETROLEUM CORP. OF CALIFORNIA (U.S.P. 1,724,510, 13.8.29. Appl., 31.8.26).—After separation from the clay, and while still hot, the oil is atomised by a non-oxidising fluid in order to vaporise deleterious substances. F. G. CLARKE.

Revivifying [oil-treated] adsorbent materials. W. M. STRATFORD, ASSR. to TEXAS CO. (U.S.P. 1,724,531, 13.8.29. Appl., 11.2.25).—Decolorising clay which has been used for refining hydrocarbon oils is extracted with a non-aqueous solvent, e.g., gasoline, at a temperature above the normal b.p. of the solvent, and under a pressure sufficiently high to maintain the solvent in the liquid state. A. B. MANNING.

Manufacture of soaps from sulphonated mineral-oil acid sludge. O. E. CUSHMAN and T. W. DOELL, ASSRS. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,718,335, 25.6.29. Appl., 28.7.23).—A demulsifying agent is produced by treating a mineral oil with sulphuric acid, separating the sludge from the oil, which is then treated with sulphuric acid of above 97% concentration, and the sludge again separated. The sulphonated products of this second sludge are separated from the free sulphuric acid therein and neutralised, after which oil-soluble colouring matter is extracted with benzol. S. S. WOOLF.

Cold asphalt and application thereof. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 317,496, 16.3.28).—Bituminous substances are dispersed in water by adding to the mixture finely-ground humic acid, brown coal, or other substances containing humic acid. The dispersions are prepared by simple stirring, and do not require the use of a colloid mill. Materials for road making, or for the construction of ceilings, walls, etc., are made by adding basalt chips, sand, cement, etc. to the dispersion. A. B. MANNING.

Coal distillation apparatus. W. M. CARR and T. J. ASHLEY (A. E. ASHLEY, extrin.) (U.S.P. 1,726,058, 27.8.29. Appl., 25.2.26. U.K., 3.3.25).—See B.P. 248,557; B., 1926, 477.

Distillation and burning of coal. H. S. REED and R. D. LAMIE (B.P. 318,373, 17.8.28).—See U.S.P. 1,696,731; B., 1929, 704.

Manufacture of carbon. A. LEDERER (U.S.P. 1,725,359, 20.8.29. Appl., 7.10.26. Austr., 24.4.25).—See B.P. 251,244; B., 1927, 244.

Removal of carbon deposits from engine cylinders. C. A. HOCHWALT, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,726,437, 27.8.29. Appl., 18.9.23).—See B.P. 222,077; B., 1924, 1005.

Manufacture of porous mass [for storage of acetylene]. E. STEIL, Assr. to AMER. GASACCUMULATOR CO. (U.S.P. 1,726,323, 27.8.29. Appl., 22.10.25. Ger., 1.11.24).—See G.P. 420,150; B., 1926, 230.

Production of gases rich in ethylene. H. SUIDA, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,726,048, 27.8.29. Appl., 24.6.24. Austr., 26.6.23).—See B.P. 237,415; B., 1925, 748.

Containers for dry-cooling of hot coke. SULZER FRÈRES SOC. ANON. (B.P. 306,933, 1.1.29. Switz., 29.2.28).

Safety device for burners. SPENCER THERMOSTAT CO., Assees. of N. T. SELLMAN (B.P. 300,507, 12.11.28. U.S., 12.11.27).

Coal-dust, gas, and oil burner for constant velocities of ejection under variable loads. WITKOWITZER BERGBAU- U. EISENHÜTTEN-GEWERKSCHAFT, and C. SALAT (B.P. 298,080, 1.10.28. Ger., 1.10.27).

Gas reactions (B.P. 317,996).—See I. **Unsaturated hydrocarbons (B.P. 298,090 and 315,895).**—See III. **Protecting cables etc. (B.P. 293,835).**—See X. **Solid grease (B.P. 317,630).**—See XII.

III.—ORGANIC INTERMEDIATES.

Microchemical detection of acetic acid as sodium uranyl acetate. D. KRÜGER and E. TSCHIRCH (Pharm. Ztg., 1929, 74, 1096—1097).—0.5 mg. of free acid may be readily detected by bringing a crystal of sodium formate and a crystal of uranyl formate into contact with opposite edges of the drop on the object glass. Acetate solutions are evaporated to dryness on the object glass, and the residue is treated with a drop of sodium uranyl formate solution. Benzoic acid interferes even in small quantities, and should be removed before the test; other common acids interfere only when present in large quantities. S. I. LEVY.

Use of aldehydes and dihydroxyacetone in the detection and differentiation of phenols. I. **Colour reactions given in sulphuric acid solution.** II. **Precipitation and staining tests involving the use of hydrochloric acid.** III. **Detection of cresol in carbolic acid by means of vanillin.** A. H. WARE (Quart. J. Pharm., 1929, 2, 249—253, 254—264, 265—266).—I. The methods adopted in the use of dihydroxyacetone, tartaric acid (giving glycollaldehyde), and form-

aldehyde are described, and their colour reactions with 22 phenolic substances tabulated.

II. Details of the procedure adopted whereby formaldehyde in particular (tabulated results) and dihydroxyacetone may be employed to effect the separation of phenols in the presence of hydrochloric acid are given. A new staining test for distinguishing phenols in which a filter paper is used as the material to be stained and drops of the reagents are allowed to fall thereon and subsequently dried, is described, and the colour reactions are tabulated together with those of the deal shaving test for seven phenols.

III. By means of a 2% solution of vanillin in alcohol a minimum of 1.5% of *o*- and *m*-cresol can be detected in carbolic acid. C. C. N. VASS.

Tests for phenols involving the use of hydrogen peroxide. A. H. WARE (Quart. J. Pharm., 1929, 2, 267—270).—The colour reactions of certain phenols in sulphuric acid solution when acted on by hydrogen peroxide alone or in conjunction with dihydroxyacetone or formaldehyde are described. Apparently specific, or relatively specific, results are indicated for the catechins, pyrocatechol, phloroglucinol, resorcinol, thymol, and gallic acid. C. C. N. VASS.

Action of catalysts on carbon monoxide-hydrogen. AUDIBERT and RAINEAU.—See II.

PATENTS.

Production of liquid polymerisation products from gases containing hydrocarbons. C. EPNER (B.P. 317,344, 9.5.28).—Methane, or gases containing methane, are subjected to the action of an alternating, high-tension electric field producing a silent electric discharge and having a frequency of $8-12 \times 10^3$ cycles/sec. The reaction is carried out in the presence of catalysts which promote condensation or hydrogenation, and at temperatures, *e.g.*, 150—500°, at which the products are in the form of vapour or a mobile liquid. A. B. MANNING.

Manufacture of unsaturated hydrocarbons. SOC. CHEM. IND. IN BASLE (B.P. 298,090, 1.10.28. Switz., 1.10.27).—Unsaturated aliphatic hydrocarbons are made by reaction between methyl alcohol and an alkaline-earth carbide at 200—300° and under ordinary pressure; the uncondensed gases such as acetylene are either collected, allowed to escape, or returned by a circulating pump to the heated reaction vessel so that they may undergo further reaction to produce hydrocarbons of higher b.p. which are removed by condensation. The carbide is automatically introduced into the reaction vessel, and means are provided for continuously freeing the carbide from the oxide which is formed during the reaction. C. B. MARSON.

Manufacture of unsaturated hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,895, 16.3.28).—Pure, unsaturated, gaseous hydrocarbons such as ethylene, propylene, butylene, and amylene are manufactured by cracking mineral oils and tars at 600—800° in the presence of gases or vapours, such as water vapour, and, if desired, with the aid of catalysts (*e.g.*, silicates with difficultly reducible oxides deposited thereon; also certain metals or alloys). The

condensates obtained by fractional liquefaction are rectified by fractional distillation. C. B. MARSON.

Recovery of methyl alcohol. W. J. V. WARD, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,708, 21.3.28).—Crude methyl alcohol from the hydrogenation of carbon oxides is shaken with brine and a solvent (benzene) which is immiscible with the solution of the alcohol in brine. On removal of the solvent and distillation of the brine, methyl alcohol is obtained free from unsaturated compounds and unpleasant odour. C. HOLLINS.

Manufacture of a stable diazo compound. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 316,691, 8.5.28).—The diazo compound of 5-nitro-*o*-toluidine is precipitated as a fluoborate. C. HOLLINS.

Manufacture of 2-naphthol-3-carboxylic acid. P. DIETERLE, ASSR. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,725,394, 20.8.29. Appl., 31.10.27).—A dry alkali β -naphthoxide is treated with carbon dioxide at 120–285° under 1 atm. pressure. L. A. COLES.

Manufacture of hydroxythionaphthens [thio-indoxyls] and of vat dyes therefrom. I. G. FARBENIND. A.-G. (B.P. 302,175, 10.12.28. Ger., 8.12.27. Addn. to B.P. 284,288; B., 1929, 674).—Phosphorus pentoxide distributed on an inert carrier (silica) is used as condensing agent for the conversion of arylthioglycolic acids in boiling benzene solution into thio-indoxyls. Examples are *m*-chlorophenyl- and β -naphthyl-thioglycolic acids. C. HOLLINS.

Production of concentrated acetate liquors from acetic acid-containing gases [from carbonisation of wood]. E. LÖW, ASSR. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,726,401, 27.8.29. Appl., 26.1.27. Ger., 4.2.26).—See G.P. 448,348; B., 1928, 805.

Manufacture of crotonyl bromide. K. MEISENBURG, ASSR. to WINTHROP CHEM. CO., INC. (U.S.P. 1,725,156, 20.8.29. Appl., 5.4.28. Ger., 30.4.27).—See B.P. 289,777; B., 1929, 806.

Production of halogenated alcohols. J. CALLEN, ASSR. to WINTHROP CHEM. CO., INC. (U.S.P. 1,725,054, 20.8.29. Appl., 17.3.27. Ger., 19.6.26).—See B.P. 286,797; B., 1928, 360.

Production of benzoic acid and like processes. D. A. W. FAIRWEATHER, E. G. BECKETT, and J. THOMAS, ASSRS. to SELDEN CO. (U.S.P. 1,727,102, 3.9.29. Appl., 16.11.28. U.K., 29.9.27).—See B.P. 309,202; B., 1929, 427.

Preparation of complex compounds of aromatic *p*-diamines with sulphur dioxide. R. WOLFFENSTEIN, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,726,252, 27.8.29. Appl., 18.4.27. Ger., 17.4.26).—See B.P. 269,583; B., 1927, 742.

Electrolytic desulphonation of anthraquinone-sulphonic acids. A. J. HAILWOOD, ASSR. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,724,928, 20.8.29. Appl., 8.12.26. U.K., 15.4.26).—See B.P. 273,043; B., 1927, 647.

Preparation of α -anthraquinonyl ketones. G. KRÄNZLEIN, H. VOLLMANN, and K. WILKE, ASSRS. to

GEN. ANILINE WORKS, INC. (U.S.P. 1,725,927, 27.8.29. Appl., 24.5.27. Ger., 25.5.26).—See B.P. 271,884; B., 1928, 665.

Decomposition of hydrocarbons (B.P. 315,890). **Conversion of hydrocarbons** (B.P. 315,991).—See II. **Vat dye intermediate** (B.P. 297,002).—See IV. **Polymerisation products of diolefines** (B.P. 292,103).—See XIV. **Butyl and isopropyl alcohols** (U.S.P. 1,725,083). **Fusel oil** (U.S.P. 1,725,084). **Gluconic acid** (U.S.P. 1,726,067). **Alcohol and glycerol** (B.P. 316,567 and 316,597). **Lactic salts** (U.S.P. 1,726,768).—See XVIII.

IV.—DYESTUFFS.

Determination of the colouring power of sulphide dyes. N. A. TOUSCHKOF-VTOROV (Rev. Gén. Mat. Col., 1929, 33, 313–316).—The sulphide dye (0.1–0.5 g.) is dissolved in aqueous sodium sulphide, filtered if necessary, and precipitated in a pure state by adding an excess of a solution of a basic dye (*e.g.*, safranin or methylene-blue, 0.5 g. per litre). The precipitate is washed on a tared filter, first with water to remove sodium sulphide, then with glacial acetic acid, and finally with glacial acetic acid and alcohol to remove iron sulphide and leuco-compound of the basic dye; the dried precipitate is then weighed. C. HOLLINS.

Fluorescence of dyes in Wood's light and its application to their identification. A. SEYEWETZ and J. BLANC (Bull. Soc. chim., 1929, [iv], 45, 611–616).—See B., 1929, 350.

PATENTS.

Manufacture of dyes of the diaminotriphenylmethane series. I. G. FARBENIND. A.-G. (B.P. 289,092, 23.4.28. Ger., 23.4.27).—A benzaldehydedisulphonic acid (*e.g.*, the 2:4-acid) is condensed with a propyl- or higher alkyl-substituted arylamine to give blue dyes of greater colouring power than the lower alkyl compounds. C. HOLLINS.

Manufacture of dyes of the anthraquinone series [for wool or acetate silk]. I. G. FARBENIND. A.-G. (B.P. 289,807, 27.4.28. Ger., 2.5.27).—Hydroxyalkylated aminoanthraquinones are prepared by condensing a chloro-, nitro-, or methoxy-anthraquinone or a 1:4-dihydroxyanthraquinone (or its leuco-compound) with hydroxylated alkylamines, *e.g.*, β -aminoethyl alcohol. 1- β -Hydroxyethylaminoanthraquinone (sulphonated for yellow-red on wool), 1:4-di-(β -hydroxyethylamino)-anthraquinone and its 5-hydroxy-derivative (blue on acetate silk, blue wool dye by sulphonation), and the product from leuco-1:4:5:8-tetrahydroxyanthraquinone (bluish-green on acetate silk, and on wool after sulphonation) are described. C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series] and an intermediate product. SOC. CHEM. IND. IN BASLE (B.P. 297,002, 10.9.28. Switz., 9.9.27).—Benzanthrone is sulphonated with oleum at 15° and the product is fused with alkali at 255–310° and alkylated with an alkyl toluenesulphonate to give a grey-blue vat dye. C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. J. Y. JOHNSON. From I. G. FARBENIND.

A.-G. (B.P. 316,156, 24.2.28).—The benzanthranyl-aminoanthraquinones of B.P. 307,947 (B., 1929, 551) are converted by alkaline or acid agents into grey to black vat dyes. C. HOLLINS.

Manufacture of azo dyes [ice-colours]. I. G. FARBERIND. A.-G. (B.P. 304,744, 28.3.28. Ger., 26.1.28. Addn. to B.P. 287,908; B., 1929, 711).—2:3-Hydroxynaphthoic arylamides are coupled in substance or on the fibre with *p*-toluidines containing halogen in positions 2:3 or 2:5, one halogen atom at least being bromine. Examples are: 5:2- or 2:5-chlorobromo-*p*-toluidine or 2:5-dibromo-*p*-toluidine with *o*-anisidine or 4-chloro-*o*-anisidine. The scarlet-red dyeings obtained are faster than those of the prior patent. C. HOLLINS.

Manufacture of azo dyes [ice-colours and pigments]. O. Y. IMRAY, Assee. of I. G. FARBERIND. A.-G. (B.P. 298,907, 15.10.28. Ger., 13.10.27. Addn. to B.P. 296,473; B., 1928, 849).—2:3-Hydroxynaphthoic or acetoacetic arylamides are coupled in substance or on the fibre with diazotised 5-halogeno-*p*-2-xylydines. Good fastness to light is claimed. Examples are 2:3-hydroxynaphthoic anilide and *p*-anisidine. C. HOLLINS.

Manufacture of azo dyes and application thereof [as varnish pigments]. SOC. CHEM. IND. IN BASLE (B.P. 294,583, 26.7.28. Switz., 26.7.27).—Pyrazolone-3-carboxylic esters are coupled with non-sulphonated and non-carboxylated homologues or substitution derivatives of aniline (including aminoazo compounds) to give pigments suitable for varnishes; e.g., with diazotised *m*-nitro-*p*-toluidine, chloroanilines, toluidines, xylydines, nitroanilines, *o*-nitroaniline-*p*-sulphonamide, or tetrazotised benzidine, dianisidine, diaminodiphenylcarbamides. C. HOLLINS.

Manufacture of complex metal compounds of azo dyes. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 316,847, 24.7.28. Addn. to B.P. 310,343; B., 1929, 638).—*m*-Phenylenediamine-4-sulphonic acid may replace the 5-sulphonic acid specified in the prior patent. C. HOLLINS.

Manufacture of complex chromium compounds of azo dyes. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 316,198, 19.3.28).—Couplings of *o*-aminophenols with β -diketones (acetylacetone, benzoylacetone) are prechromed. [Stat. ref.] C. HOLLINS.

Manufacture of chromiferous dyes. I. G. FARBERIND. A.-G. (B.P. 316,231, 23.1.28. Addn. to B.P. 269,522; B., 1928, 704).—Chromable *o*-hydroxyazo dyes are (a) treated in substance with larger amounts of chromium salts under milder conditions than in the prior process, or (b) treated in substance or in the dye-bath with a prechromed dye (the same or different) containing more than the minimum amount of chromium, or (c) are dyed on fibre already dyed with such a prechromed dye. Ten examples are given. C. HOLLINS.

Manufacture of substantive trisazo dyes. I. G. FARBERIND. A.-G. (B.P. 286,227, 27.2.28. Ger., 28.2.27).—5-Nitro(or 5-acetamido)-anthranilic acid is diazotised and coupled with a middle component (A), the product is reduced (or hydrolysed), tetrazotised, and coupled with 2 mols. of an end-component (B; especially a

m-diamine or *m*-aminophenol); the dye may be coppered on the fibre. In the examples A = γ -acid, and B = *m*-phenylenediamine (greenish-black), *m*-aminophenol (greenish-black, not staining wool), or *m*-tolylene-diamine (black). C. HOLLINS.

Preparation of [leuco]-ester-like derivatives of vat dyes. M. BADER, C. SUNDER, and T. VOLTZ, Assrs. to DURAND & HUGUENIN SOC. ANON. (U.S.P. 1,727,267—8, 3.9.29. Appl., 27.7.23. Ger., 21.8.22).—See B.P. 202,630 and 202,632; B., 1924, 375.

Manufacture of [perylene] dyes. A. PONGRATZ, Assr. to F. BENSA (U.S.P. 1,725,596, 20.8.29. Appl., 13.12.26. Austr., 2.10.26).—See B.P., 278,325; B., 1928, 399.

Sulphur dyes from dihydroindole-indophenolic bodies. F. BALLAUF, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,725,046, 20.8.29. Appl., 25.10.26. Ger., 2.11.25).—See B.P. 286,005; B., 1928, 740.

[Manufacture of] thiazole derivatives of 1:4-naphthaquinone. W. HERZBERG and G. HOPPE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,726,265, 27.8.29. Appl., 28.12.26. Ger., 28.11.25).—See B.P. 262,141; B., 1928, 8.

[Manufacture of insoluble] azo dye. A. SCHMELZER, F. MUTH, and E. GLEITENBERG, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,726,681, 3.9.29. Appl., 20.12.27. Ger., 22.12.26).—See B.P. 282,682; B., 1929, 552.

Vat dyes from thioindoxyls (B.P. 302,175).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Sulphur content of wool. II. Distribution of sulphur along the fibre, variation with colour, and effect of exposure to ultra-violet light. III. Effects of chemical processing on sulphur content. J. BARRITT and A. T. KING (J. Text. Inst., 1929, 20, T 151—158, 159—161; cf. B., 1926, 870).—II. The distribution of sulphur along the fibre is variable. Dipping causes no change in the sulphur content, whilst weathering diminishes the content at the tips of the hairs; in general, however, the highest percentage is found towards the tips. Marked differences are found in the sulphur content of coloured wools from the same animal; the higher values are found for white wool. On exposure to ultra-violet radiation wool loses part of its sulphur as sulphur dioxide, which is partly evolved as such and partly retained as sorbed sulphur dioxide or is oxidised to sulphuric acid. It is suggested that the epithelial scales have a higher sulphur content than that of the cortex, and that the loss of sulphur due to exposure is due in part to a diminution in the scale portion of the fibre. In an addendum the statement due to Marston (B., 1929, 277) that the sulphur content of wool shows no variation which cannot be attributed to the analytical method used is criticised, and his procedure is regarded as less capable of giving constant results than that used by the present authors. The biological significance of variable sulphur in wool is discussed in its relation to the cystine nucleus.

III. The effects of certain commercial processes on the sulphur content of wool have been determined.

Commercial scouring with 2% sodium carbonate solution, treatment with saturated lime water for times up to 250 hrs., and with chlorine water of various concentrations for 1 hr. have little effect on the sulphur content. It is concluded that changes in the physical properties accompanying processing, especially normal chlorination, are out of all proportion to the actual change in chemical composition so far as total sulphur is concerned.

B. P. RIDGE.

Adsorption of water by wool. J. B. SPEAKMAN (*Nature*, 1929, 124, 411—412).—The amount of water adsorbed by wool which had previously been stretched 30% of its length in water at 25° for 24 hrs. and then allowed to return to its original length was greater than that adsorbed by wool which had been immersed in water at 25° for 24 hrs.; hence plastic flow in the fibrillar structure alone increases the affinity of wool for water (cf. King, B., 1929, 773). A. A. ELDRIDGE.

Rotting of textiles by micro-organisms. I. A. laboratory test. G. O. SEARLE (*J. Text. Inst.*, 1929, 20, 162—174).—Strips of fabric of a size suitable for use in a Goodbrand cloth tester are bound round filter candles which have been covered with a thin layer of a soil in such a way that the layer contains a large proportion of the fungus spores and bacteria which were previously in the sample of soil from which the layer was obtained. The strips are kept in an incubator and some are tested for strength at intervals. No appreciable difference is found between the resistance of a cotton and a linen duck tested by this method over a period of six weeks. It is shown that the viscosity in cuprammonium of cloths tendered by micro-organisms is the same as that of normal cellulose, and this is suggested as a basis for distinguishing between micro-biological decay and tendering by light, since the latter causes a considerable decrease in viscosity.

B. P. RIDGE.

Decomposition of red beech and white birch by the sulphite process. O. WURZ (*Papier-Fabr.*, 1929, 27, 495—498).—If special attention be paid to the composition of the cooking liquor and to the time of saturation of the wood, satisfactory cooking of red beech and of white birch may be carried out by the ordinary Ritter-Kellner process. Cooking acid containing 4—5% SO₂ (total) and 0.9% CaO is suitable, and a sufficiently long time for saturation should be given at 100°. A yield of about 49% of soft, short-fibred cellulose is obtained which can be bleached satisfactorily after thorough washing. For similar conditions of cooking, beech cellulose is bleached somewhat more easily than birch cellulose. Beech cellulose yielded about 0.6% of ash and contained about 86—88% of α -cellulose, but an investigation of the other properties, such as chlorine consumption, viscosity, copper number, etc., is necessary to complete the present work.

B. P. RIDGE.

Causes of variations in the quality of mechanical wood pulp and paper pulp during manufacture. J. ENCKELL (*Papier-Fabr.*, 1929, 27, 498—500).—The influence of grinding pressure, temperature, stone quality, method of sharpening, dry content of the wood, etc. on the quality of the pulp obtained has been investigated by determining the "bursting surface" of sheets

of standard dimensions formed from the pulps. The apparatus used was the Mullen tester, and results obtained by this means are compared with those given by the Schopper-Riegler method. In general, a lower grinding pressure is accompanied by an increase in the bursting-surface values. Temperature of grinding is of little importance so far as bursting-surface values are concerned, but for equal values the higher the temperature the lower is the power consumption per ton. If it is suitably sharpened, natural stone generally gives better pulp than artificial stone, whilst some varieties of the latter give relatively long fibres and others give coarse material. The method of sharpening natural stone has a considerable effect on the properties of the pulp, and with the same sharpening rollers different results are given according to the pressures used. The grinding process is also considerably affected by the moisture content of the material; dry wood is harder than moist, requires a sharper stone surface, and gives a relatively short-fibred and finely ground pulp, whilst moist wood gives a long-fibred and more uniform pulp. The bursting-surface values for pulp prepared from fresh wood and from wood attacked by red-rot are about the same, whilst the Schopper-Riegler values are higher for the former; the influence of red-rot wood on the quality of the pulp is smaller, however, than is generally supposed.

B. P. RIDGE.

Chemical control of cooking in the sulphite-cellulose process. B. RASSOW and H. KRAFT (*Papier-Fabr.*, 1929, 27, 489—495, 508—514, 524—527).—The course of the cooking process under different conditions and for various kinds of wood has been investigated by a study of the α -lignin content of the cooking liquors. Klason's theory, that α -lignin contains a coniferyl aldehyde group, is supported by the fact that vanillin is obtained from lignosulphonic acid- β -naphthylamine and -benzidine compounds isolated from ordinary and from dialysed waste liquor by oxidation with nitrous acid in the cold. As a result of the indirect determination of the α -lignosulphonic acid by precipitation with benzidine dihydrochloride, and of the examination of the precipitated compound, the following observations are made. By the precipitation, at least two substances are formed, one of which is salted out, whilst the other is considered to be a compound of α -lignosulphonic acid with the amine. The α -lignosulphonic acid which is dissolved during the cooking still absorbs sulphurous acid, so that a further absorption of the amine occurs. The analysis values for the benzidine precipitations show a pronounced change in the liquor during the quality-cooking period. In agreement with Hägglund's theory it is shown that the cooking process is divisible into sulphonation and hydrolysis periods. If the beginning of decomposition is denoted by the beginning of the hydrolysis, it occurs at different times for woods of different origin. Completion of the hydrolysis period does not coincide with complete separation of the fibres, but the latter occurs during the quality-cooking period, which is shortened or prolonged according to the quality of cellulose desired. From the sodium hydroxide values obtained by titration of the α -lignosulphonic acid, curves are obtained showing the increase in the concentration of

this acid in the liquor during cooking. These curves show two characteristic breaks, the second of which is important in the control of cooking since it corresponds with the beginning of the quality-cooking period. In general, for bleachable stuff the cooking should be stopped 4 hrs., and for solid stuff 1 hr., after reaching this point.

B. P. RIDGE.

PATENTS.

Manufacture of embossed knitted fabrics. BRIT. CELANESE, LTD. (B.P. 291,445, 22.5.28. U.S., 3.6.27).—Circular knitted fabrics of cellulose derivatives are sprayed with a softening agent, which should be a solvent of the cellulose derivative suitably diluted with a non-solvent, *e.g.*, aqueous acetone, before being subjected while still in double thickness to a permanent embossing operation.

F. R. ENNOS.

Manufacture and treatment of cellulose derivatives. H. DREYFUS (B.P. 316,160, 18.4.28).—Cellulose materials are acylated with anhydrides of hydroxylated aliphatic acids, *e.g.*, glycollic, lactic, β -hydroxypropionic acid, preferably in presence of a catalyst. Mixed derivatives may be produced by using two anhydrides together.

C. HOLLINS.

Manufacture of artificial materials from viscose. L. LILIENFELD (B.P. 317,608, 13.7.28. Addn. to B.P. 274,521; B., 1927, 745).—Viscose made from an alkali-cellulose which has not been matured or has been matured for only a short time (1–36 hrs.) is precipitated in a bath containing at least 48–50% by wt. of sulphuric acid.

F. R. ENNOS.

Manufacture of artificial silk filaments. J. A. SINGMASTER (U.S.P. 1,725,742, 20.8.29. Appl., 28.9.27).—The filaments are spun from solutions containing inorganic pigment-like material present in such quantity and in such a form as not to impair the continuity of the filaments during spinning.

L. A. COLES.

Production of filaments, threads, bands, ribbons, etc. from cellulose derivatives. A. EICHENGRÜN (B.P. 317,408, 8.2.28).—Fine filaments of less than 3–4 denier are produced by the extrusion through normal apertures of cellulose acetate dissolved in a mixture of a volatile solvent, *e.g.*, acetone, or a latent solvent, *e.g.*, methylene chloride, together with a higher-boiling coagulant, *e.g.*, alcohol, benzene, water; the dimensions of the extruded filaments are regulated by adjusting the relative amounts of the constituents, keeping the cellulose derivative in solution, and by suitably varying the winding speeds, which should be above the normal (75 m. per min.).

F. R. ENNOS.

Fibrous material for odourless containers. J. D. RUE, S. D. WELLS, and F. G. RAWLING (U.S.P. 1,724,778, 13.8.29. Appl., 13.9.26).—Straw is gently heated with an aqueous solution containing sodium carbonate and sulphite, so as to soften it without producing a pulp. Containers made therefrom are odourless in cold storage and suitable for foodstuffs.

F. G. CLARKE.

Preservative package for articles, especially fruits and the like. L. MELLERSH-JACKSON. From CELLACOTE Co., INC. (B.P. 317,513, 15.5.28).—The article is coated by spraying, immersion, etc. with a layer of viscose which is coagulated in two stages, first

with a neutral salt (ammonium sulphate) and then with dilute acid, washed, and dried so as to cause it to shrink on the article.

F. R. ENNOS.

Treating [textile] materials to render them undesirable as habitation for insects. H. MAXWELL-LEFROY, ASSR. to GRAESSER MONSANTO CHEM. WORKS, LTD. (U.S.P. 1,725,656, 20.8.29. Appl., 15.6.25. U.K., 24.3.25).—See B.P. 253,993; B., 1926, 738.

Production of wood pulp. W. D. MOUNT (U.S.P. 1,725,818, 27.8.29. Appl., 10.9.25).—See B.P. 269,256; B., 1927, 438.

Extraction of pure cellulose from bagasse of sugar cane. E. C. H. VALET, ASSR. to CELULOSA HEMMER VALET SOC. ANON. MEXICO (Re-issue 17,422, 3.9.29, of U.S.P. 1,630,147, 9.8.26. Mexico, 14.6.26).—See B., 1927, 552.

Production of cellulosic material. C. LEYST, ASSR. to A. H. LYMN (U.S.P. 1,726,078, 27.8.29. Appl., 11.11.26. U.K., 14.11.25).—See B.P. 265,697; B., 1927, 328.

Treatment of vegetable fibres in presence of organic derivatives of cellulose. C. DREYFUS, R. G. DORT, and H. PLATT, ASSRS. to CELANESE CORP. OF AMER. (U.S.P. 1,725,857, 27.8.29. Appl., 8.7.26).—See B.P. 274,074; B., 1928, 811.

[Domestic] washing and centrifuging machines [for fabrics etc.]. SIEMENS-SCHUCKERTWERKE A.-G., and W. WEIMERS (B.P. 317,939, 12.6.28).

[Apparatus for] treating [granulated] cork [for use as insulating material etc.]. N. MASSA (B.P. 318,345, 14.7.28).

Spinning nozzles for manufacture of artificial silk. H. COLOMB (B.P. 301,080—1, 16.11.28. Switz., 25.11.27). C. RUZICKA (B.P. 318,464, 17.12.28).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Treatment [with ozone] of wool and other animal fibres or textiles containing them. S. W. WILKINSON and R. B. BROWN (B.P. 317,133, 8.5.29).—Wool and other protein-containing fibres of animal origin are ozonised as in B.P. 242,027 and 289,578 (B., 1926, 11; 1928, 480) except that the treatment is discontinued before the fibres gain a substantially increased affinity for dyes; the resulting fibres thereby lose their property of felting and shrinking when further subjected to such processes as scouring, milling, and dyeing. The treatment with ozone may be shortened by previously impregnating the fibres with oxidising agents or oxygen carriers such as the peroxides of hydrogen, calcium, barium, sodium, and potassium, or soluble salts of manganese and vanadium.

A. J. HALL.

Treatment of textile fibres of cellulosic or other composition. COMP. NAT. MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES ÉTABL. KUHLMANN (B.P. 304,227, 22.5.28. Fr., 17.1.28).—Apparatus suitable for treating cotton and wool fibres in order to modify their affinity for dyes as described in B.P. 195,619 and 198,166 (B., 1924, 128; 1923, 652 A) comprises one or more fixed containers for the fibrous

materials in communication with an automatic liquor-circulating system, whereby any suitable liquid, particularly acetic acid and acetic anhydride, is pumped through a coiled heater, then through the fibrous material, collected in a heated storage vessel, and returned to the pump.

A. J. HALL.

Production of resist effects in dyeing with vat dyes. I. G. FARBENIND. A.-G. (B.P. 290,177, 7.5.28. Ger., 6.5.27).—Fabric is printed with a resist paste containing an oxidising agent (e.g., sodium *m*-nitrobenzenesulphonate or sodium dichromate) and a water-soluble alkyl ether of cellulose, dried, and dyed in the usual manner in a hot alkaline solution of a vat dye. A suitable paste consists of 250 g. of a 15% aqueous solution of cellulose methyl ether, 500 g. of water, and 250 g. of sodium *m*-nitrobenzenesulphonate.

A. J. HALL.

Producing fast tints on ethers or esters of cellulose. SOC. CHEM. IND. IN BASLE (B.P. 293,006, 29.6.28. Switz., 29.6.27).—Tetra-aminoanthraquinones which contain no sulphonic or carboxylic acid group and in which one or two amino-groups are arylated are suitable for dyeing the cellulose derivatives in fast blue to green shades.

A. J. HALL.

Stripping rags or like coloured materials with sulphur dioxide. VIRGINIA SMELTING Co., Assees. of F. W. BINNS (B.P. 295,007, 10.4.28. U. S., 6.8.27).—Coloured textile materials are stripped, and can then be re-dyed by washing them with a slightly alkaline solution and subjecting them to the action of a reducing liquor prepared by passing about 40 lb. of gaseous sulphur dioxide into 28 lb. of zinc dust suspended in 42 gals. of water and then adding 1.5 lb. of formaldehyde. [Stat. ref.]

A. J. HALL.

Thickening ingredient for liquids for altering the colour of hair. J. F. SCHWARZLOSE SÖHNE G.M.B.H. (B.P. 297,838, 27.9.28. Ger., 30.9.27).—Plant mucus of various algae, particularly agar-agar and caragheen, is used.

A. J. HALL.

Improvement of cellulosic materials. L. DE WOLF (U.S.P. 1,726,142, 27.8.29. Appl., 9.11.25. Belg., 25.5.25).—See B.P. 252,360; B., 1927, 70.

Making textiles having the appearance of leather. G. A. SALLMANN (U.S.P. 1,725,675, 20.8.29. Appl., 7.12.26. Ger., 8.12.25).—See B.P. 262,783; B., 1927, 249.

Jigger machines for dyeing. J. DEAN (B.P. 318,411, 9.10.28).

Apparatus for [multi-colour] dyeing. ECLIPSE TEXTILE DEVICES, INC., Assees. of J. P. GAREY and L. B. HASBROUCK (B.P. 303,892, 7.1.29. U.S., 12.1.28).

Treating with a liquid artificial threads, fibres, or ribbons of cellulose. H. ELLING (B.P. 318,327, 20.6.28).

Manufacture of decorative sheet material [of celluloid or cellulose acetate]. SOC. DES BREVETS J. PAISSEAU (B.P. 293,391, 29.6.28. Fr., 5.7.27).

Sulphonated linseed oil (B.P. 293,806).—See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Evolution of hydrocyanic acid from calcium cyanide. H. D. YOUNG (Ind. Eng. Chem., 1929, 21, 861—863).—Calcium cyanide, when used as an insecticide, is dependent for its efficacy on atmospheric moisture which reacts with it to liberate hydrogen cyanide. The rate of liberation of hydrogen cyanide from various samples of calcium cyanide by known volumes of air of varying humidity was determined. The rate was found to increase with the humidity and with the fineness of division of the calcium cyanide.

C. IRWIN.

Platinised silica gels as catalysts for oxidation of sulphur dioxide. H. N. HOLMES, J. RAMSAY, and A. L. ELDER (Ind. Eng. Chem., 1929, 21, 850—853).—Platinum was deposited on Holmes' chalky gel, Patrick's glassy gel, and asbestos by treatment with platinum chloride and reduction with formaldehyde. The conversion efficiency in the oxidation of sulphur dioxide by catalysts of each type at 395° was determined with varying amounts of platinum. The optimum temperature decreases with increase of platinum content. Either at fixed or optimum temperatures the efficiencies of the gels were about equal to each other, and rather greater than that of platinised asbestos.

C. IRWIN.

Iron oxide reduction equilibria from the point of view of the phase rule and thermodynamics. O. C. RALSTON (U.S. Bur. Mines, Bull. No. 296, 1929, 326 pp.).—A critical discussion is given of the data concerning the physical and chemical properties of ferric oxide, including density, thermal expansion, thermal and electrical conductivity, m.p., b.p., dissociation pressure, polymorphism, sp. heat, entropy, heat of formation, and free energy; also of the properties of the ferromagnetic form. Similar accounts are given for ferrous oxide and for magnetite (including thermal *E.M.F.*, thermal emissivity, and magnetic properties). The methods of preparation and the properties of ferrites, ferrates, and perferrates are summarised. Thermal and magnetic investigations on the system ferric oxide-magnetite are described. Using the data of Sosman and Hostetter (A., 1916, ii, 331) for the oxygen pressures over mixtures of ferric oxide and magnetite between 1100° and 1200°, the heat of the reaction $6\text{Fe}_2\text{O}_3 = 4\text{Fe}_3\text{O}_4 + \text{O}_2$ is calculated to be 88,480 g.-cal. at 1423° Abs., and that of the reaction $2\text{Fe}_3\text{O}_4 = 6\text{FeO} + \text{O}_2$ to be approximately 141,950 g.-cal. The mutual solubilities of ferrous oxide and magnetite are discussed. These compounds are partially miscible below 1175°. The evidence as to the mutual miscibility of iron and ferrous oxide is indefinite, and complicated by the probability that carbides were present in many of the experiments recorded. The known limits of miscibility do not support the reported existence of the oxides Fe_2O , Fe_3O , and Fe_4O . The crystal structures of iron and its transformation temperatures, as determined by different methods, are recorded. The recognised points are: 210°, Curie point of Fe_3C ; 725°, iron-carbon eutectic; 768°, Curie point of iron; 906°, α - γ ; 1400°, γ - δ ; 1537°, m.p. The b.p. and vapour pressure

data are discussed, and also the magnetic, electrical, and thermal properties, and the heats of transition, latent heat of vaporisation, and entropy. The system iron-carbon is discussed in detail, together with other questions involving iron carbide: the region of stability of oxyaustenite, the oxyferrite-oxyaustenite and austenite-carbon oxide equilibria, the carbon-oxygen-iron equilibrium in liquid steel, and carburisation involving methane. Attention is drawn to the necessity of oxygen determination, *e.g.*, by the method of Oberhoffer (*cf.* B., 1928, 409), and to the need of a more rigorous application of the phase rule. Equilibrium diagrams of complete oxidation-reduction systems for the iron oxides cannot be constructed with certainty, on account of the complexity of the systems involved, but an ideal oxidation-reduction isotherm is discussed, from pure triferrous tetroxide to pure iron, the gas consisting entirely of carbon monoxide and dioxide. The apparent failure to prepare pure ferrous oxide is discussed in the light of the work of Schenck (B., 1926, 663) and of Benedicks and Löfquist (B., 1927, 445). The methods of preparation and properties of the iron carbonyls are reviewed.

C. W. GIBBY.

Determination of lead peroxide. A. V. PAMFILOV [with O. K. DUNAIEVA, F. G. FILIPYČEV, (Miss) E. G. IVANČEVA, and A. A. TROIZKAJA] (Z. anal. Chem., 1929, 78, 40–52).—Methods for the determination of lead peroxide have been examined. It is concluded that for fairly pure material the modified Diel-Hopf method of the American Bureau of Standards gives the best results. If the sample contains particles of metallic lead the thio-sulphate methods of Liebig or Figg are preferable. The preparation of pure lead peroxide electrolytically from lead was found to be impossible, probably owing to the presence of occluded lead nitrate.

H. F. HARWOOD.

Determination of nitrates in bismuth carbonate. G. J. W. FERREY (Quart. J. Pharm., 1929, 2, 205–216).—The B.P. phenoldisulphonic acid method has been compared with (a) the indigo-carmin method, and (b) the official method of the Board of Agriculture and Fisheries in which the reduction of the nitrate to ammonia is carried out by means of reduced iron and dilute sulphuric acid. The B.P. method is accurate between the limits 1.4–3.0% $\text{BiONO}_3 \cdot \text{H}_2\text{O}$, the main source of inaccuracy being the matching of the colours. Method (a) is essentially a time-reaction method, which under the specified conditions affords a good agreement with the B.P. method and with method (b), the last-named yielding consistent and accurate results.

C. C. N. VASS.

Action of catalysts on carbon monoxide-hydrogen mixtures. AUDIBERT and RAINEAU.—See II.
Detection of acetic acid. KRÜGER and TSCHIRCH.—See III.

PATENTS.

Concentration of nitric acid. A. K. CROAD. From CHEM. CONSTRUCTION CO. (B.P. 317,168, 11.6.28).—A mixture of dilute nitric acid with a dehydrating agent, *e.g.*, concentrated sulphuric acid, flows down a series of horizontal pipes connected by elbows and provided with baffles to maintain a shallow layer of the liquid in each pipe. The pipes are heated by a current of hot

liquid, *e.g.*, oil, flowing through jackets around the pipes in a direction opposite to that of the mixed acids inside the pipes. Concentrated nitric acid vapour passes out of the top pipe, and the residual sulphuric acid is withdrawn from the lowest pipe of the series.

L. A. COLES.

Production of hydrocyanic acid. CALIFORNIA CYANIDE CO., INC., Assees. of P. T. DOLLEY (B.P. 299,019, 19.10.28. U.S., 19.10.27).—Finely-divided material containing alkali or alkaline-earth cyanides, *e.g.*, calcium cyanide or the product obtained by fusing calcium cyanamide with sodium chloride, is treated in gaseous suspension, preferably by introduction tangentially into the lower end of a conical reaction vessel provided with suitable baffles etc., with a current of steam and, if desired, carbon dioxide; the hydrogen cyanide formed is separated from the solid residue.

L. A. COLES.

Manufacture of hydrogen peroxide by cathodic reduction of oxygen. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 316,919, 4.5.28).—The current of oxygen or gases containing it employed in the electrolysis is used also in the concentration and/or distillation of the hydrogen peroxide produced. Both electrolysis and concentration may be effected in the same vessel, water being removed from the gas stream before it is returned to the vessel, or separate vessels may be used for the processes of electrolysis, concentration, and distillation.

L. A. COLES.

Crystallisation of salts from brine. G. B. BURNHAM, Assr. to BURNHAM CHEM. CO. (U.S.P. 1,724,558, 13.8.29. Appl., 16.11.25).—Brine is caused to flow beneath a salt crust heated by rays of the sun.

H. ROYAL-DAWSON.

Production of potassium carbonate. KALI-IND. A.-G., C. T. THORSELL, and A. KRISTENSSON (B.P. 300,629, 5.10.28. Ger., 18.11.27).—Potassium chloride or other potassium salt is stirred, preferably in solid form, in liquid ammonia (containing little or no water) with the simultaneous introduction of carbon dioxide, the resulting potassium carbamate being converted in known manner into carbonate or bicarbonate. The ammonia and carbon dioxide thus liberated are employed afresh, ammonia is recovered by distillation from the liquid separated from the carbamate, and the salt remaining is heated to decompose any carbamate into ammonia and carbon dioxide, and is then treated with milk of lime to recover ammonia.

W. G. CAREY.

Production of nitrates of the alkalis and alkaline earths. KALI-IND. A.-G., C. T. THORSELL, and A. KRISTENSSON (B.P. 300,632, 18.10.28. Ger., 18.11.27).—A solution or suspension of an alkali or alkaline-earth hydroxide or carbonate containing only a small quantity of the nitrate is used as absorbent in the towers of a nitric acid plant, and the resultant solution, after conversion of the nitrite into nitrate, is cooled to crystallise out the greater part of the nitrate. After removal of the crystals, the mother-liquor is heated by the liquid leaving the towers, and is saturated with the hydroxide or carbonate for re-use.

L. A. COLES.

[Granular] compositions containing alkali peroxides [for use in respirators etc.]. IMPERIAL CHEM. INDUSTRIES, LTD., F. HOLT, and J. A. M. W. MITCHELL

(B.P. 317,966, 23.7.28).—Finely-divided sodium peroxide, alone or mixed with a catalyst or with potassium peroxide, is fritted at 300–400° in the presence of a flux, *e.g.*, sodium hydroxide, which may be added as such or produced *in situ*, *e.g.*, by grinding the material in air. The product is broken up into granules, the fines being separated for refritting.

L. A. COLES.

Manufacture of chloride of lime. A. CARUGHI and C. PAOLONI (B.P. 317,716, 15.5.28).—Chloride of lime containing more than 40% of available chlorine is made by chlorinating slaked lime suspended in a liquid inert to lime or chloride of lime, *e.g.*, carbon tetrachloride, the lime being wet-ground and chlorinated, and the solvent recovered by vacuum distillation in a single apparatus, or in a series of vessels, the chlorination taking place above 40°. The product becomes crystalline by reducing the temperature after the operation to 20° and continuing the mixing. The chloride of lime may be moulded by compression while wet, and then dried.

W. G. CAREY.

Manufacture of bleaching powder. J. W. MOORE, A. LAMBLE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 317,572, 9.6.28).—High-strength stabilised bleaching powder is produced by circulating a large volume of dilute chlorine through a rotary tube into which lime is introduced and from which bleaching powder is removed, water being abstracted by the gas heated by the heat of reaction. Removal of water from the gas is effected outside the reaction vessel, and after adjustment of the percentage of chlorine the gas is returned to the same or another reaction vessel.

W. G. CAREY.

Production of magnesia from dolomite. T. TWYNAM (B.P. 317,961, 19.7.28).—Dolomite is dissolved in nitric acid and just sufficient lime as burnt dolomite is added to precipitate the magnesium as hydroxide, which is separated from the calcium nitrate solution.

W. G. CAREY.

Production of aluminium chloride free from iron. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 317,259, 24.10.28).—Aluminium chloride vapour contaminated with ferric chloride etc. is brought into contact with molten lead at about 400°; ferrous chloride and lead chloride are deposited on the lead, and aluminium chloride vapour passes on to a condenser.

L. A. COLES.

Removal of ferric chloride during treatment of leucite and like silicates with hydrochloric acid. G. A. BLANC (B.P. 318,067, 11.12.28).—The strongly acid mother-liquor from which aluminium chloride has crystallised while hot (*cf.* B.P. 176,770 and 181,677; B., 1922, 812 A; 1923, 402 A) is allowed to cool before dilution with water, so that a quantity of ferric chloride is deposited together with a portion of the potassium chloride.

W. G. CAREY.

Manufacture of titanium compounds. I. G. FARBENIND. A.-G. (B.P. 290,174, 7.5.28. Ger., 6.5.27).—The mixture obtained by treating ilmenite etc. with sulphuric acid is treated hot with sufficient quantities of an alkali salt (preferably potassium sulphate) and water to yield a saturated potassium titanium sulphate solution, which is filtered and then cooled to crystallise

out the double sulphate; the crystals are removed, purified by washing with alkali sulphate solution or by crystallisation from dilute sulphuric acid, and worked up to give the desired titanium compounds.

L. A. COLES.

[Manufacture of] esters of silicic acid. M. MÜLLER-CUNRADI and G. LECHNER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,725,620, 20.8.29. Appl., 7.1.28. Ger., 18.1.27).—See B.P. 298,778; B., 1928, 893.

Oxidation of ammonia. F. G. LULJENROTH (U.S.P. 1,725,462, 20.8.29. Appl., 11.1.26. Swed., 30.1.25).—See F.P. 610,160; B., 1927, 251.

Manufacture of ammonium chloride crystals. J. W. MOORE and W. G. POLACK, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,725,292, 20.8.29. Appl., 1.6.27. U.K., 25.6.26).—See B.P. 273,093; B., 1927, 652.

Production of metallic [zinc] sulphate [from sulphide ores]. F. JOHANNSEN, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,725,865, 27.8.29. Appl., 6.7.27. Ger., 12.7.26).—See B.P. 297,597; B., 1928, 862.

Manufacture of cyanides. H. WIEDERHOLD, Assr. to N. V. NEDERL. MIJNBOUW EN HANDEL MAATS. (U.S.P. 1,727,261, 3.9.29. Appl., 18.11.26. Ger., 10.12.25).—See B.P. 262,802; B., 1927, 937.

Manufacture of chromium compounds. P. WEISE, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,723,536, 6.8.29. Appl., 25.10.26. Ger., 24.8.25).—See B.P. 259,447; B., 1926, 1013.

Generating hydrogen [by decomposition of hydrocarbons]. R. E. BATTIG (U.S.P. 1,726,877, 3.9.29. Appl., 18.5.27. Ger., 20.5.26).—See B.P. 271,483; B., 1928, 748.

Alloy resistant to nitric acid (B.P. 300,250).—See X. **Electrochemical treatment of solutions** (U.S.P. 1,726,236).—See XI.

VIII.—GLASS; CERAMICS.

Measurement of viscosity of glass at high temperatures by the rotating-cylinder viscosimeter. R. F. PROCTOR and R. W. DOUGLAS (Proc. Physical Soc., 1929, 41, 500–519).—Molten glass is contained in a cylindrical sillimanite vessel provided with a co-axial cylindrical stirrer which is caused to rotate, the viscosity of the glass being inferred from the angular velocity produced in the stirrer by a measured torque. The apparatus was calibrated by means of the viscosities of golden syrup and pitch, which had been determined with a falling-sphere viscosimeter and by a torsion method. The calibration factor is independent of the viscosity over a wide range. The viscosities of various glasses measured in this way show good agreement with the results of previous workers.

C. J. SMITHELLS.

PATENTS.

Circular kiln. O. BUSCH (U.S.P. 1,724,392, 13.8.29. Appl., 14.5.27. Fr., 10.6.26).—The chambers communicate with a common discharge flue extending round the kiln. Gas is fed from a central superstructure through a series of horizontal flues which communicate upwardly

with the atmosphere and downwardly with the kiln chambers.

J. A. SUGDEN.

Alkali silicate product. N. M. ELIAS (U.S.P. 1,724,185, 13.8.29. Appl., 6.7.27).—The alkali silicate material in the form of a glassy solid is heated at 200—500° and then coated with a waterproofing substance.

H. ROYAL-DAWSON.

Manufacture of transfers and their application for the decoration of pottery, glass, enamelled iron, jewellery, etc. L. ALLEN and E. C. DEERING (B.P. 317,411, 16.3.28).—Transfers printed in "liquid metal" (gold, silver, etc.) are obtained from copper plates, using commercial liquid gold preparations which have been heated to expel volatile oils and leave a tacky residue. The transfers are applied to surfaces heated at 50—70°, the paper backing is removed by water, and the design fixed by firing in the usual way.

A. R. POWELL.

Firing of ceramic ware. A. S. WATTS (U.S.P. 1,724,238, 13.8.29. Appl., 29.1.27. Renewed 8.11.28).—In a tunnel kiln provided with distinct zones for the various stages of biscuiting, vitrifying, glazing, and annealing, there is a temperature drop between each section so that each stage of the firing may take place independently of the heat treatment received in the preceding or succeeding zones.

J. A. SUGDEN.

Manufacture of refractory compositions. K. E. PEILER, Assr. to HARTFORD-EMPIRE Co. (U.S.P. 1,724,886, 13.8.29. Appl., 1.6.25).—Refractory grog is prepared by compressing in a mould powdered refractory clay containing only sufficient moisture for binding, then firing and grinding.

J. A. SUGDEN.

Casting refractory articles. CORNING GLASS WORKS, Assees. of G. S. FULCHER (B.P. 298,983, 28.9.28. U.S., 18.10.27).—A method for preventing the formation of "pipe" (voids due to shrinkage) during the casting of articles from molten refractory material is described.

J. A. SUGDEN.

Manufacture of abrasive articles. NORTON Co. (B.P. 295,292, 8.8.28. U.S., 8.8.27).—For successful bonding of abrasive grains, the bond and grains should have the same coefficient of expansion. In the case of aluminous abrasives the bond should mature below 1200° so that slag formation is avoided. Further, development of crystals in the bond during cooling should be avoided by rapid cooling through the devitrification range, followed by slow cooling until the bond is solid. Such a bond suitable for aluminous grains contains oxides of silicon 55%, aluminium 11.4%, iron 4.3%, magnesium 2.8%, calcium 5.9%, sodium 6.0%, potassium 2.6%, titanium 0.7%, and boron 11.3%. This bond matures at 1175° without detrimental frothing and can be annealed at 650—550°.

J. A. SUGDEN.

Production of sheet glass. A. E. WHITE. From MISSISSIPPI GLASS Co. (B.P. 318,430, 13.11.28).

Manufacture of spun glass. L. MATHIEU (B.P. 318,078, 26.1.29. Fr., 12.9.28).

Manufacture of plate glass. A. SCHILD (B.P. 318,047, 31.10.28).

Sealing metal to glass (B.P. 303,348 and U.S.P. 1,724,465).—See X. **Opacifying pigments** (U.S.P. 1,719,432).—See XIII.

IX.—BUILDING MATERIALS.

Steam-curing of Portland cement mortars. A new crystalline substance. T. THORVALDSON and G. R. SHELTON (Canad. J. Res., 1929, 1, 148—154).—An attempt has been made to elucidate the increased tensile strength and resistance to disintegration by sulphates of Portland cement mortars, which results from steam-curing (cf. B., 1928, 484). Resistance to sulphate action was studied on a 1 : 10 mix, by measuring the expansion produced by immersion in 2.1% and 6.7% solutions of sodium sulphate. Variations in the tensile strength and changes of crystalline form were studied in 2-day and 28-day briquettes (1 : 5 mix) after curing in steam for 6 hrs. at temperatures ranging from 100° to 200° and also at 150° for periods up to 48 hrs. No relationship between sulphate-resistance and tensile strength was established. Hydration increased with time of steaming and with temperature of the autoclave. Crystals of hydrated lime were formed at once and increased to a maximum at 175° and thereafter decreased, but at the same time a new crystalline form appeared to which the increased resistance to sulphate attack is thought to be due, since the crystals are not disintegrated by sodium sulphate and only slowly by magnesium sulphate. The crystals are very stable to heat, being unchanged after 24 hrs. at 400°, but darkening after prolonged heating at 650°. They are readily decomposed by very dilute acids, hydrochloric acid giving a skeleton of insoluble matter, and sulphuric acid producing a large amount of gypsum. The optical properties of the crystals are described and figured.

H. J. DOWDEN.

Effect of calcium chloride on cement. C. R. PLATZMANN (Chem.-Ztg., 1929, 53, 477—478, 494—496).—Cement or concrete mixed with a solution of calcium chloride sets more rapidly than when mixed with water, and, owing to the greater evolution of heat in the initial stages of setting, a more coarsely crystalline structure is obtained resulting in higher initial tensile and compression strength. During ageing for 1—3 months the compression strength of cement mixtures containing calcium chloride increases more slowly than that of chloride-free mixtures, but is always 5—20% greater. The tensile strength of the chloride mixtures, however, decreases appreciably after 1 month, and is finally less than that of water mixtures. Calcium chloride is a suitable addition agent for cement mixtures made in frosty weather, owing to the rapid setting and heat evolution produced.

A. R. POWELL.

Zinc cements. A. A. SCHTSCHUKIN (Ukraine Chem. J., 1929, 4, 285—291).—Zinc oxide is added to zinc chloride solution in such quantity that the total zinc present in solution is equal to that contained in the zinc oxide. The mixture is, for several days, alternately shaken and allowed to rest, and then filtered; a clear solution is obtained which finally solidifies. The solid is a mixture of the cement (oxychloride) and some of the original substances.

A. FREIMAN.

PATENTS.

[Electric kiln etc. for] manufacture of cement and lime. H. J. BENHAM (B.P. 316,715, 19.5.28).—Powdered raw material is delivered by plates, arranged inside a shaft, into a chamber, wherein it is fused between

electrodes. Waste gases from the chamber are baffled by the plates and pass through a rotary preheating tube into which the charge is initially fed, and which has holes at one end and around the circumference to allow preheated raw material to be discharged into the pulveriser.

J. S. G. THOMAS.

Manufacture of Sorel cement. H. S. LUKENS, Assr. to SOLIDON PRODUCTS, INC. (U.S.P. 1,724,945, 20.8.29. Appl., 30.6.26).—A mixture of a ballast material with magnesia and magnesium chloride is incorporated with an emulsion containing a water-proofing substance which does not affect the setting properties, and the emulsion is coagulated by the subsequent addition of a suitable reagent.

A. R. POWELL.

Production of cements, building, plastering, and coating materials from mud or the like. "KOLLOID-CHEMIE" STUDIENGES.M.B.H., J. B. CARPZOW, M. MARCH, R. LENZMANN, and H. SANDERS (B.P. 317,919, 30.5.28).—Mud containing unsaturated silicon compounds, or colloidal material recovered from the mud by washing or sedimentation, with or without pretreatment comprising prolonged suspension in water in the absence of air with the addition, if desired, of bacteria nutrients and anaerobic bacteria, or partial drying below 80° in the absence of air, is mixed, preferably in the absence of air, with metal oxides or metal compounds other than lime or chalk, *e.g.*, aluminium sulphate, chromium trioxide, bauxite, kaolin, furnace slag, calcium sulphate; when the reaction has ceased, the product is mixed with cement, colouring material, etc., or is calcined and ground for use as cement, or is moulded and baked. L. A. COLES.

Purifying fused cements. SOC. D'ETUDES CHIM. POUR L'IND. (B.P. 298,637, 6.10.28. Switz., 13.10.27).—Fused aluminiferous cement, obtained as a by-product in the electrothermic manufacture of phosphorus and its compounds, is treated with gases containing or yielding oxygen or with solid oxidising agents, *e.g.*, iron oxide or manganese dioxide, to remove deleterious constituents.

L. A. COLES.

Manufacture of plasters from anhydrite. V. LEFEBURE (B.P. 317,672, 22.10.28. Addn. to B.P. 236,695; B., 1925, 720).—By adding to anhydrite the component inorganic salts which constitute the double salt used in the prior patent, the setting time etc. of the plaster is modified. Thus up to 2% of zinc sulphate and up to 4% of potassium sulphate may be added.

J. A. SUGDEN.

Production of an artificial stone material. E. TELTSCHIK & Co. (B.P. 301,893, 15.10.28. Czechoslov., 9.12.27).—Vegetable ivory purified by treatment with acid or alkaline solutions and subsequent washing with water is mixed with magnesian cement, *e.g.*, by saturating the ivory with magnesium chloride solution and then adding magnesium oxide. Dyes and solutions of caoutchouc, celluloid, phenol-aldehyde condensation products, etc. may be added to the mixtures.

L. A. COLES.

Manufacture of building blocks, artificial stones, tiles, and other shaped objects. H. ENZIO GRAF VON PLAUE (B.P. 310,312, 27.9.28. Ger., 23.4.28).—Sawdust or other waste wood product, sand, peat, or other

filling material is combined with milk, magnesite, flowers of sulphur, and sodium carbonate or magnesium sulphate; talc and/or asbestos may also be added. The mixture is allowed to harden in air, during which the temperature rises to 70–80°.

J. A. SUGDEN.

Composition for rendering the surfaces of walls and other surfaces and for decorative purposes. W. TAYLOR (B.P. 317,902, 26.5.28).—The compositions claimed comprise calcium chloride, potassium soft soap (or a mixture of potassium hydroxide and oil), china clay or aluminium silicate, and coloured cement, worked up to the desired consistency with water.

L. A. COLES.

Surfacing materials for roadways, pavements, etc. A. H. TYLER (B.P. 317,527, 18.5.28).—A mixture of granite sand (32%), limestone dust (15%), granulated glass or silica of $\frac{1}{8}$ -in. mesh (22%), and peat or sawdust containing 5% of water (12%) is bonded with 20% of bitumen (m.p. 134°) at about 160°.

J. A. SUGDEN.

Cold asphalt (B.P. 317,496).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Finishing of converter-refined iron in the open-hearth furnace. A. JUNG (Stahl u. Eisen, 1929, 49, 1221–1224).—The results of a 3-day test in which iron refined in the Thomas converter was finished in the Siemens–Martin furnace are recorded, and the economics of the procedure discussed.

A. R. POWELL.

Solubility of carbide in ferrite. H. A. DICKIE (Iron and Steel Inst., Sept., 1929. Advance copy. 25 pp.).—The following physical properties have been measured in a wide range of steels including alloys containing up to 7% Ni, 2% Mn, 5.5% Cr, 4.5% Ni, and 1.5% Cr, and Armco iron:—(1) Izod impact value, (2) Brinell hardness, (3) sp. resistance, (4) sp. vol., (5) magnetic properties. The heat treatments were such as to indicate the effect of the small amount of carbon in solid solution in ferrite, and changes due to tempering effects such as spheroidisation were eliminated by first quenching the specimen from the temperature at which it was desired to measure, and then retempering at a lower temperature sufficiently high to redeposit the carbide out of solution without causing further spheroidisation. Dissolution of carbide in ferrite is shown up by marked decreases in remanence and permeability. As a general rule, the maximum induction is lowered and the coercive force raised slightly on dissolution. The solubility curves are estimated from the variations in the physical properties. The stability of the ferrite solid solutions and the solubility of carbon in pure α -iron are discussed.

W. HUME-ROTHERY.

Coalescence of pearlite. J. H. WHITELEY (Iron and Steel Inst., Sept., 1929. Advance copy. 8 pp.).—The conditions which affect the formation of continuous cementite borders around pearlite areas have been studied in hypoeutectoid steels containing 0.2–0.31% C and 0.04–1.10% Mn. In these steels the transformation of γ -iron solid solution into pearlite occurs over a short range of temperature immediately below the Ac point, and the formation of the cementite

borders takes place in this range before the γ -iron is completely transformed, the carbide necessary for the growth of the border being supplied by diffusion through the intervening ferrite, which can hold about 0.03% C in solid solution at 700°. When heated under conditions in which γ -iron is absent, marginal coalescence does not occur but the carbide films tend to break up into globules. The velocity of coalescence is diminished by the presence of manganese, and with 1.0% Mn the phenomenon can no longer be observed.

W. HUME-ROTHERY.

Iron-silicon-carbon alloys. Constitutional diagrams and magnetic properties. T. D. YENSEN (Iron and Steel Inst., Sept., 1929. Advance copy. 17 pp.; cf. B., 1925, 320).—Previous work on the magnetic properties of iron-carbon-silicon alloys is discussed with special reference to the equilibrium diagrams. A new diagram is given in which it is assumed that pure iron exists only in the body-centred cubic form, and that the face-centred cubic austenite appears only in the presence of carbon or other interstitial impurities, such as oxygen. The solubility of carbon in α -iron is about 0.008% at room temperature. Diagrams are also given for iron-carbon-silicon alloys, the general effect of silicon being to diminish, and finally to suppress, the γ -area. The magnetic properties are particularly sensitive to the formation of the solid solution in α -iron, a difference of 0.001% of dissolved carbon having the same effect on the magnetic hysteresis as 0.01% of sulphur. The variation of magnetic properties with composition and heat treatment is discussed on the basis of the suggested diagrams. W. HUME-ROTHERY.

Red-shortness [of steel]. A. NIEDENTHAL (Arch. Eisenhüttenw., 1929—30, 3, 79—97; Stahl u. Eisen, 1929, 49, 1227—1230).—The effect of sulphur and oxygen on the forging properties of steel at high temperatures has been investigated. With sulphur there are three distinct temperature zones; between 800° and 1050° there is a zone of red-shortness, and above 1100° a zone of hot-shortness, these two zones being separated by a more or less extensive zone of malleability. With a low sulphur content only the first zone is detectable, and with a high content (0.3% S) the two brittle zones merge into one, the intermediate malleable zone disappearing. Manganese tends to reduce the deleterious effects of sulphur and, to a smaller degree, of oxygen. The effect of oxygen in steel is to displace the zone of blue-brittleness towards higher temperatures so that the metal appears to be red-short, but above 900° the presence of oxygen does not produce hot-shortness. The red-shortness of steel containing sulphur, but not that due to oxygen, may be cured by prolonged annealing at 1150° followed by a short anneal at 1000° prior to forging; after this treatment a steel containing 0.36% S and 0.3% Mn was quite workable at a red heat. Microscopical examination showed that red-shortness was caused by the presence of a brittle constituent and hot-shortness by a molten constituent, both of which, by prolonged annealing, entered into solid solution.

A. R. POWELL.

Heat-resisting steels. E. HOUDREMONT and V. EHMCKE (Arch. Eisenhüttenw., 1929—30, 3, 49—60; Stahl u. Eisen, 1929, 49, 1265).—At temperatures

below 650° the steels which retain their strength most satisfactorily with rise of temperature are those having a stable martensitic structure, *i.e.*, steels containing chromium, molybdenum, tungsten, and vanadium. Molybdenum and vanadium steels especially retain their high yield point up to 500°; chromium, tungsten, and vanadium steels are the most satisfactory at 500—650°. Above 650° all steels tend to soften more or less rapidly, and the strongest steels are therefore those containing elements which tend to increase the m.p. and restrain the rate of recrystallisation, *i.e.*, tungsten, chromium, and molybdenum. Alloy steels with these three elements have the greatest strength of any steels at 800—900°.

A. R. POWELL.

Relative corrodibilities of ferrous and non-ferrous metals and alloys. II. Results of seven years' exposure to air at Birmingham. J. N. FRIEND (Inst. Metals, Sept., 1929. Advance copy. 4 pp.; cf. B., 1928, 410).—Fifty-four bars of ferrous and non-ferrous metals were fixed with putty into a frame, and exposed for seven years on the roof of the Birmingham Technical College. Results are given for the non-ferrous metals, which included tin, lead, nickel, zinc, aluminium, and various grades of copper and brass. The loss in weight due to the exposure was determined after cleaning with emery paper moistened with ammonia. Zinc was the only metal to show slight pitting, and all resisted corrosion much better than wrought irons and carbon steels. Brasses were less resistant than was copper, and arsenical coppers were generally slightly more resistant than was pure copper, but there is no advantage in increasing the arsenic content beyond 0.25%. Aluminium figured well in contrast to the sea-corrosion tests described earlier (*loc. cit.*), where it was virtually destroyed. The lead, tin, and stainless steels resisted atmospheric corrosion remarkably well, especially the antimonial lead.

W. HUME-ROTHERY.

Maurer's manganese steel in the development of non-rusting steel. E. MAURER (Stahl u. Eisen, 1929, 49, 1217—1220).—An historical survey of the development of non-rusting steels in England, Germany, and America.

A. R. POWELL.

Corrosion-resisting steel for laboratory use. G. A. STOKES (Analyst, 1929, 54, 538).—Total solids in milk and other foods may be satisfactorily determined in capsules of Hadfield's "Era, C.R.1" stainless steel, and 0.1N-solutions of fruit acids, vinegar, or sodium hydroxide do not affect the steel when evaporated to dryness on a water-bath.

D. G. HEWER.

Analysis of carbon in iron and iron alloys. N. A. ZIEGLER (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 8 pp.).—Improvements in manipulative details of the Yensen method for the determination of carbon in iron and iron alloys (B., 1920, 367 A) are described. The determination takes 44 min. and the results are reproducible to within 0.0001—0.0005%.

H. J. T. ELLINGHAM.

Determination of oxide inclusions in iron and steel by analysis of the residue obtained by treatment with chlorine. R. WASMUTH and P. OBERHOFFER (Arch. Eisenhüttenw., 1928—9, 2, 829—842; Stahl u.

Eisen, 1929, 49, 1260—1262).—For the determination of silica and alumina inclusions in iron and steel heating in chlorine at 500—600° followed by analysis of the residue gives good results. Under these conditions ferrous and manganous oxides are decomposed with the liberation of oxygen, but at lower temperatures (about 350°) all the oxygen is retained as the oxides Fe_2O_3 and Mn_2O_4 , so that an approximate idea of the oxygen content of the metal is obtained by determining the percentages of these constituents in the residue from chlorination. The method is not applicable to complex alloy steels.

A. R. POWELL.

Open-air corrosion of copper. Chemical study of the surface patina. W. H. J. VERNON and L. WHITBY (Inst. Metals, Sept., 1929. Advance copy. 15 pp.).—The chemical composition of the patina on copper roofs etc. in different localities has been studied, the periods of exposure to the atmosphere varying from 12 to 300 years. The typical green patina consists essentially of basic copper sulphate, except in purely marine atmospheres where basic copper chloride predominates; where both marine and urban conditions coincide the amount of basic sulphate greatly exceeds that of chloride. In contradiction to the general belief, basic copper carbonate is present only in small amount. A sufficiently long exposure to the atmosphere always produces a green patina, but usually, and in urban conditions always, there is an intermediate black stage which consists of sooty matter associated with oxides and sulphides of copper. The sulphur compounds are due to the products of combustion of coal, which are carried long distances by the wind, and the patina forms most readily on the side facing the prevailing wind. The impurities present in the copper have but little effect, except that lead favours the production of the preliminary black stage, and owing to the insolubility of its salts, may accumulate in the patina to such an extent that the green colour is suppressed. The results are confirmed by those of Hudson (B., 1929, 684) for much shorter exposures.

W. HUME-ROTHERY.

Creep of 80 : 20 nickel-chromium alloy at high temperatures. A. G. LOBLEY and C. L. BETTS (Inst. Metals, Sept., 1929. Advance copy. 20 pp.).—Wires of nickel-chromium alloy (0.018, 0.0625, and 0.25 in. diam.) were submitted to tensile stresses of 50, 100, 200, and 400 lb./in.² for 4000 hrs. at 700°, 800°, 900°, and 1000°, and the extension was measured at regular intervals. At a given stress the rate of creep increases with decrease in diameter of the wire below 0.0625 in., but with thicker wires the effect of diameter is less clear. The increase at small diameters agrees with Thompson and Millington's theory (B., 1924, 836) of the effect of free surfaces. At 400 lb./in.² the rate of creep increases continuously with temperature, but at the lower stresses no increase occurs until a temperature of about 900° is exceeded. The creep increases with the stress, being most rapid at the beginning of the test, and falling to a smaller rate after the first few days; the secondary creep is not uniform, but takes place in steps. Up to 900° the rate of secondary creep is unaffected by temperature, which increases only the initial creep. Above 900° there is no evidence of a

creep stress limit; between 700° and 900° there is no evidence of a creep stress limit with the fine wires, but the evidence for thick wires is inconclusive. The alloy used contained 21% Cr, 0.58% Mn, 0.63% Fe, 0.15% C, and the remainder nickel.

W. HUME-ROTHERY.

Corrosion of lead. K. SCHERINGA (Pharm. Weekblad, 1929, 66, 741—743).—The discovery of a piece of lead piping eaten completely through led to experiments which demonstrated that, in the presence of alkalis and moisture, lead is readily converted by air, in the absence of carbon dioxide, into the monoxide.

S. I. LEVY.

Use of tantalum as cathode for electrodeposition of copper. L. W. STROCK and H. S. LUKENS (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 10 pp.).—The possibility of using tantalum instead of platinum as the cathode material in the electro-analytical deposition of copper from nitric-sulphuric acid solutions has been investigated. In order to secure smooth, adherent deposits the tantalum surface must first be freed from any oxide film, and this is best effected by rubbing with fine carborundum paper. Chemical methods are unreliable and cathodic treatment causes brittleness of the cathode and leads to unsatisfactory deposits. Connections are made so that a current density of 3 amp./dm.² is produced on inserting the cathode into the solution. After electrolysis for about 5 min. at this high current density, which serves to produce an even deposit over the whole cathode surface, the current density is reduced to 1 amp./dm.² and maintained at this value until deposition is complete. Before making the next determination the copper deposit is dissolved from the cathode by means of a nitric-sulphuric acid mixture, but owing to the oxidising action of nitric acid coarsely crystalline deposits eventually appear and mechanical cleaning is then required.

H. J. T. ELLINGHAM.

Gravimetric electrodeposition of metals and its application to pharmaceutical chemicals. S. G. LIVEREDGE (Quart. J. Pharm., 1929, 2, 243—248).—Details are given for the rapid determination of mercury, bismuth, antimony, zinc, and silver in pharmaceutical preparations, based on Sand's method (A., 1929, 672).

C. C. N. VASS.

Metallurgical cokes. BRAUNHOLTZ and others. **Size of coke and combustion in tuyères.** MATHESIUS.—See II.

PATENTS.

Regenerative furnace used in steel making. G. H. ISLEY, Assr. to MORGAN CONSTRUCTION Co. (U.S.P. 1,724,656, 13.8.29. Appl., 17.1.27).—A charge to be used later in the furnace is inserted in a space behind the bag wall of the furnace to be preheated and conditioned. A travelling crane serves to transfer the charge through suitable openings in the furnace crown.

C. A. KING.

Smelting furnace. H. L. CHARLES (U.S.P. 1,724,340, 13.8.29. Appl., 27.11.26).—Hollow bricks are inserted in the roof and side walls at the firing end of a reverberatory furnace. A solid end to the bricks forms part of the crown, but the upper end is open, the bricks holding

metal containers which are fitted with cold-water inlets and hot-water headers. C. A. KING.

Smelting furnace. C. H. MACE (U.S.P. 1,724,490, 13.8.29. Appl., 30.1.25).—In a furnace of the cupola type, a tuyère entering at the bottom of the shaft is brought from the upper part of an air box, the lower part serving as a collector for material entering through the tuyère. Collected material may be removed through a normally closed opening in the air box. C. A. KING.

Roasting furnace. N. T. WELLMAN, ASSR. to GEN. CHEM. Co. (U.S.P. 1,721,265, 16.7.29. Appl., 25.6.25).—In a multiple-hearth, mechanically-rabbed roasting furnace for sulphide ores the feed hopper is provided with a second outlet which supplies a small proportion of raw ore to one of the lower hearths. A. R. POWELL.

Mechanical [ore-roasting] kilns. BALZ ERZRÖSTUNG GES.M.B.H., and G. BALZ (B.P. 317,341, 14.4.28. Addn. to B.P. 288,823; B., 1928, 451).—The arms of the kiln are composed of horizontal adjacent tubes (three) enclosed in a common insulating sleeve which may be spaced from the tubes to form additional air channels. The arms connect to a double concentric central shaft, and may carry vanes and devices for air-sealing the kiln compartments. C. A. KING.

Blast-furnace gas washer. S. STEWART (U.S.P. 1,718,988, 2.7.29. Appl., 25.4.25).—The washer comprises a cleaning chamber through which the gas flows, a transverse pipe opening into one side of the chamber and out of the opposite side, spraying devices on either side of these openings, and a vertically adjustable, V-shaped, spray-deflecting pipe mounted above the transverse pipe which can be moved into the path of the spraying jets to break them up and direct them through the gas stream. A. R. POWELL.

Apparatus for manufacturing iron and steel. P. J. PEYRACHON (U.S.P. 1,720,055, 9.7.29. Appl., 2.5.27. Fr., 13.7.26).—The apparatus comprises a vertical smelting furnace covered with a refractory hood into which opens a horizontal, rotary, reducing furnace so that the flames from the smelting furnace are directed through the reducing furnace where the hot gases effect a preliminary reduction of the ore. A. R. POWELL.

Heat treatment of [steel] bars. G. LANGFORD, ASSR. to MCKENNA PROCESS Co. OF ILLINOIS (U.S.P. 1,724,031, 13.8.29. Appl., 27.4.27).—The bar is heated to near its hardening temperature, then cooled to the critical point for the steel, from which point it is allowed to cool very gradually. H. ROYAL-DAWSON.

Pickling and cleaning metals [iron and steel]. G. D. CHAMBERLAIN, ASSR. to R. T. VANDERBILT Co., Inc. (U.S.P. 1,719,167—S, 1,719,649, and 1,719,650, 2.7.29. Appl., [A—C] 11.1.27, [D] 19.2.27).—The action of the usual hydrochloric or sulphuric acid pickling bath on iron or steel may be inhibited by addition to the bath of (A) a condensation product of a nitrogenous base and a ketone, *e.g.*, of ammonia and acetone, (B) hexamethylenetetramine, (C) a condensation product of an aldehyde with an amine, *e.g.*, of aldol and α -naphthylamine, or (D) an aromatic amine, *e.g.*, dibenzylaniline or dibenzylamine, with or without thiocarbonyl. A. R. POWELL.

Refining metals and alloys. S. WESTBERG (B.P. 317,180, 25.6.28).—Iron or steel scrap is heated at 1400—1500°, but below the m.p., in an atmosphere of hydrogen, with or without water vapour and/or hydrocarbon gases, to remove oxygen, sulphur, and phosphorus without formation of slag. The process may be operated under increased or reduced pressure according to the nature of the metal and the impurities to be removed. Copper may also be refined at 700° in a similar manner. A. R. POWELL.

Treatment of metals [iron]. F. C. KELLEY, ASSR. to GEN. ELECTRIC Co. (U.S.P. 1,718,563, 25.6.29. Appl., 17.4.25).—Iron is provided with a surface coating of an alloy resistant to oxidation by heating it in contact with powdered chromium and silicon at 1350° in a current of hydrogen. A. R. POWELL.

Preventing corrosion of submerged iron. P. LECHLER (B.P. 295,319, 13.7.28. Ger., 9.8.27).—The water covering the iron is itself covered with a layer of mineral oil containing dissolved bitumen to increase its viscosity. A. R. POWELL.

Protecting cables, pipes, and other metallic bodies against corroding influences. CHEMIEPRODUKTE G.M.B.H. (B.P. 293,835, 10.7.28. Ger., 13.7.27).—The cables etc. are wrapped with fabric or paper coated on both sides with material which does not harden nor become brittle, *e.g.*, a mixture of protoparaffins, m.p. about 60°, petroleum jelly, m.p. 25°, and bitumen, m.p. 30°, heated at 110° to remove moisture, and incorporated with fillers, *e.g.*, calcined china clay, talc, asbestos powder. L. A. COLES.

Manufacture of alloy steel. H. WADE. From INTERNAT. NICKEL Co. (B.P. 315,673, 2.2.28).—Steel scrap is melted in an electric furnace under oxidising conditions until the silicon content is reduced below 0.1%, preferably below 0.05%, so as to remove all occluded gases. The necessary alloying elements are then added immediately prior to casting. [Stat. ref.] A. R. POWELL.

[Iron-nickel] alloy. N. V. HYBINETTE (U.S.P. 1,726,489, 27.8.29. Appl., 12.10.21).—The proportion of iron to nickel in the alloy is not less than 5 : 2; up to 8% of chromium, molybdenum, and tungsten, together with a small amount of manganese, may also be present. M. E. NOTTAGE.

Recovery of zinc [from galvanised iron scrap]. W. G. HORSCH, ASSR. to VULCAN DETINNING Co. (U.S.P. 1,719,056, 2.7.29. Appl., 2.12.26).—The scrap is treated with a cold 20—40% solution of sodium hydroxide containing a little sodium nitrate, whereby the zinc dissolves as sodium zincate. The solution is diluted and boiled to precipitate zinc hydroxide, and the liquor is concentrated for further use. A. R. POWELL.

Separation of mixed sulphide ores by flotation. W. D. GREEN, ASSR. to COMBINED METALS REDUCTION Co. (U.S.P. 1,721,993, 23.7.29. Appl., 2.7.25).—A mixture of a sulphite and zinc sulphate is used as a modifying agent in the differential flotation of sulphide ores. A. R. POWELL.

Roasting of sulphide minerals. J. B. READ and M. F. COOLBAUGH (B.P. 317,665, 2.10.28).—In the roasting

of sulphide minerals of zinc, copper, and/or lead, the conditions are so maintained in the earlier stages that the maximum amount of sulphate is formed and a predetermined quantity of raw ore is fed into the furnace at the later stages, so as to decompose any desired proportion of the sulphates.

A. R. POWELL.

Production of ore briquettes. A. KNAFF, L. MAYER, and P. GREDT (B.P. 317,652, 8.9.28).—Finely-divided ores of manganese, chromium, tungsten, vanadium, molybdenum, titanium, nickel, cobalt, or phosphorus are mixed with carbonaceous material, iron compounds of low oxidation stages, finely-divided metallic iron, and a binder which accelerates the oxidation of iron, *e.g.*, waste liquor from etching of sheet metal, and are briquetted.

C. A. KING.

Purifying metals used for castings. A. KIRCHHOFF (B.P. 318,073, 28.12.28. Addn. to B.P. 278,164; B., 1927, 881).—Metals other than aluminium or cast iron may be purified by introducing powdered animal shell into the molten metal and removing the scum.

M. E. NOTTAGE.

Compound for use [as dressing] in casting metals. B. F. WALLACE (U.S.P. 1,717,820, 18.6.29. Appl., 15.7.25).—Precipitated calcium carbonate impregnated with 5–10% of an organic waterproofing substance, *e.g.*, wool grease, and with 7% of kerosene is claimed.

A. R. POWELL.

Production of copper-lead alloy with varying physical properties. A. H. ACKERMAN, Assr. to ACKERMITE Co. (U.S.P. 1,724,896, 20.8.29. Appl., 11.11.22. Can., 21.11.21).—In the production of bearing metal, molten copper and lead are combined in the presence of substantial amounts of sulphur, sulphides of arsenic and antimony, and arsenic oxide.

F. G. CROSSE.

Brazing alloy. A. E. AVERRETT, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,724,818, 13.8.29. Appl., 12.6.28).—The alloy contains about 54% Cu, 36% Zn, and not more than 10% Ag.

H. ROYAL-DAWSON.

Thermal treatment of non-cementable metals and alloys. CHEM. FABR. WEISSENSTEIN GES.M.B.H. (B.P. 302,642, 20.6.28. Austr., 19.12.27).—Copper, silver, and gold alloys are annealed in salt baths containing suspended carbonaceous material or reducing agents, such as cyanides.

A. R. POWELL.

[Nickel-chromium] metallic alloy [resistant to nitric acid]. BARBER ASPHALT Co., Assees. of A. B. DAVIS (B.P. 300,250, 16.4.28. U.S., 10.11.27).—The alloy comprises 50–60% Ni, 10–20% Cr, 3–5% Si, 1–2% Mn, 3–5% W, and the remainder iron, together with a small quantity of copper and cobalt such that the sum of these and the tungsten, manganese, and silicon does not exceed 10%. The preferred composition is 53% Ni, 23% Fe, 15% Cr, 4% W, 1.25% Mn, and 3.75% Si. [Stat. ref.]

A. R. POWELL.

Light metal [magnesium] alloy. J. A. GANN, Assr. to DOW CHEM. Co. (U.S.P. 1,718,642, 25.6.29. Appl., 18.10.22. Renewed 1.9.26).—The alloy comprises magnesium with 3–12% Cu, 1–4% Al, and 1–4% Cd; the preferred composition is 92% Mg, 4% Cu, 2% Al, and 2% Cd.

A. R. POWELL.

Improving the resistance to corrosion of magnesium and magnesium alloys. I. G. FARBENIND. A.-G. (B.P. 305,197, 22.11.28. Ger., 2.2.28. Addn. to B.P. 287,450; B., 1929, 60).—The articles are pickled in dilute nitric acid, then boiled for 1 hr. in a solution of sodium dichromate.

A. R. POWELL.

Process of treatment of oxidised lead ores. N. C. CHRISTENSEN (U.S.P. 1,726,258, 27.8.29. Appl., 4.12.22).—A solution of lead chloride, produced by lixiviating the ore with a concentrated solution of sodium chloride, is separated and electrolysed, using an iron anode, until about two thirds of the dissolved lead is precipitated and a solution of ferrous chloride is produced. The solution is further electrolysed, using an insoluble anode, whereby the remainder of the lead is precipitated and ferrous chloride is converted into ferric chloride, which is used to lixiviate more ore.

J. S. G. THOMAS.

Production of zinc. A. L. J. QUENEAU (U.S.P. 1,718,378, 25.6.29. Appl., 3.2.27. Renewed 20.11.28).—A mixture of 67% of quicklime and 33% of zinc oxide is suspended in 75% of its weight of a molten mixture of 5 mols. of sodium chloride, 2 mols. of potassium chloride, and 3 mols. of calcium chloride at 550°, and hydrogen is passed into the mass through the axis of a stirring paddle. Metallic zinc is formed and collects at the bottom of the vessel until the lime has combined with 31% of the moisture required to form the hydroxide. The operation is conducted in a cast-iron kettle lined with graphite.

A. R. POWELL.

Extraction of mercury from cinnabar ore. F. M. SCHAD (U.S.P. 1,718,491, 25.6.29. Appl., 11.3.26).—The finely divided ore is leached with a solution containing sodium sulphide and barium or calcium sulphhydrate, and the mercury is recovered by treating the liquors with zinc or dilute acid.

A. R. POWELL.

Removal of arsenic from ores, speiss, and other metallurgical products. E. KIRMSE and W. SCHOPPER, Assr. to AMER. METAL Co., LTD. (U.S.P. 1,718,825, 25.6.29. Appl., 29.10.27. Ger., 3.3.27).—The material is mixed with carbonaceous substances and iron pyrites or sulphur in such quantities as to form a self-combustible mixture, the mass is ignited, and air supplied to maintain an atmosphere of carbon oxysulphide in the combustion chamber until the arsenic is completely volatilised.

A. R. POWELL.

Manufacture of potassium. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,031, 9.5.28).—Potassium fluoride is heated with magnesium at 500–700° under reduced pressure in an inert atmosphere and the liberated potassium is condensed in a cooled receiver filled with paraffin oil.

A. R. POWELL.

Production of metallised [silvered] surfaces. A. ROWLAND-ENTWISTLE (B.P. 316,401, 29.6.28 and 1.1.29).—A plane glass surface is silvered by the usual method and the deposit is washed and dried at 40°. The warm surface is then coated thinly with a solution of 1 pt. of gelatin and 0.1 pt. of formalin in 8 pts. of 50% alcohol, the article to be silvered is pressed down evenly over the surface, and the whole allowed to dry completely at 40–42°. When dry the article covered

with a layer of silver may be readily stripped from the glass support. A. R. POWELL.

Production of metals [platinum from ores]. J. SAVELSBERG, Assr. to C. SCHLESINGER & TRIER KOMMANDITGES. AUF AKT. (U.S.P. 1,723,444, 6.8.29. Appl., 28.10.27. Ger., 29.11.26).—Ores containing platinum are heated in intimate contact with a reducing agent, and are then subjected to magnetic separation. A. R. POWELL.

Production of tarnish-resisting silver and silver plate. D. GRAY, R. O. BAILEY, and W. S. MURRAY, Assrs. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,719,365, 2.7.29. Appl., 3.4.24. Renewed 13.12.28).—Articles of silver or its alloys or of silver plate are exposed at 143–157° in a chamber filled with saturated mercury vapour until the required amount of mercury has been absorbed. A. R. POWELL.

Manufacture of aluminium-plated articles. D. GRAY, R. O. BAILEY, and W. S. MURRAY, Assrs. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,723,277, 6.8.29. Appl., 5.4.24. Renewed 26.10.28).—Aluminium is deposited from a bath consisting of a solution of an aluminium salt of an aminobenzenesulphonic acid in a non-aqueous solvent, using an aluminium anode. A. R. POWELL.

Re-working of finely-divided metal particles. J. SCHMELLER, SEN. (B.P. 317,378, 13.2.28).—Finely-divided metal, *e.g.*, aluminium, is melted in a furnace chamber and agitated by means of a vertical reciprocating plunger or grid. Portions of the molten metal are run off intermittently to a second chamber from which it is cast. C. A. KING.

Salt baths [for heat-treatment of metals]. A. E. BELLIS (U.S.P. 1,724,551—2, 13.8.29. Appl., [A] 10.9.25, [B] 18.3.29).—Baths containing (A) 79% of potassium chloride and 20% of anhydrous sodium borate, and (B) boron trioxide as an oxide solvent, are claimed. H. ROYAL-DAWSON.

Sealing metal upon glass. SIEMENS-REINIGER-VEIFA GES. F. MEDIZIN. TECHN. M.B.H. (B.P. 303,348, 11.10.28. Ger., 31.12.27).—The metal part at the point of sealing consists of a nickel-chromium alloy (60–90% Ni). C. A. KING.

Fusing metallic bodies to glass. R. ENGELS, Assr. to C. H. F. MÜLLER RÖNTGEN-ROHRENFABR. (U.S.P. 1,724,465, 13.8.29. Appl., 25.2.26. Ger., 30.1.26).—The part of metal to be fused to glass is first coated with an aluminium lacquer. H. ROYAL-DAWSON.

Electrolytic extraction of metals [nickel]. Soc. ANON. "LE NICKEL" (B.P. 299,375, 3.4.28. Fr., 13.4.27. Cf. B.P. 288,605; B., 1929, 214).—In the production of electrolytic nickel a number of wire cathodes are used instead of the usual sheets, whereby large, smooth, cylindrical ingots of high density can be obtained. A. R. POWELL.

Electrolytic deposition of chromium. L. H. R. GOWER, and S. O'BRIEN & PARTNERS, LTD. (P. 317,137—15.5.28).—The electrolyte comprises a solution of chromic acid, *d* 1.2–1.3, used in conjunction with an aluminium anode which may be a grid, a cage, or a

mass of granulated aluminium packed in a perforated container. A. R. POWELL.

Chromium plating of conducting bodies. Soc. NOUVELLE DE L'ORFÈVRENERIE D'ERCUIS (B.P. 303,884, 20.12.28. Fr., 12.1.28).—The electrolytic bath consists of (per 100 litres of aqueous solution) 25 kg. of sodium dichromate, 15 kg. of chromic acid, and 900 g. of chromic fluoborate, with or without 200 g. of fluoboric acid. M. E. NOTTAGE.

Annealing and heat-treating furnace. A. T. KATHNER (Re-issue 17,413, 20.8.29, of U.S.P. 1,669,902, 15.5.28).—See B., 1928, 527.

Manufacture of strong, machinable cast iron. F. GREINER (U.S.P. 1,726,433, 27.8.29. Appl., 2.11.26. Ger., 2.11.25).—See B.P. 260,619; B., 1928, 234.

Aluminium alloys. W. SANDER, Assr. to T. GOLDSCHMIDT A.-G. (U.S.P. 1,726,194, 27.8.29. Appl., 19.1.28. Ger., 20.1.27).—See B.P. 283,927; B., 1929, 134.

Converting tin ores into the form of pieces. P. HAEDRICH and O. KIPPE, Assrs. to METALLGES. A.-G. (U.S.P. 1,726,066, 27.8.29. Appl., 3.8.27. Ger., 9.8.26).—See B.P. 275,618; B., 1928, 269.

Surface treatment of metals. M. FOURMENT (U.S.P. 1,726,431, 27.8.29. Appl., 29.11.26. Fr., 5.12.25).—See B.P. 262,439; B., 1928, 338.

Electromagnetic body. R. SWINNE, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,725,026, 20.8.29. Appl., 15.4.27. Ger., 17.5.26).—See B.P. 271,106; B., 1928, 454.

Endothermic reactions (B.P. 309,942). Separation of minerals etc. (B.P. 316,922).—See I.

XI.—ELECTROTECHNICS.

Design and operation of vacuum furnaces with carbon resistor tubes. A. S. KING (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 14 pp.).—The development of electric vacuum furnaces for very high temperatures, such as are required in vaporising metals, is reviewed, and details of the construction and operation of the "hooded" vacuum furnace of the Mount Wilson laboratory are given. The charge is contained in a graphite tube which acts as a resistor to a low-voltage current of very high intensity. Temperatures above 3000° can be obtained. An outline is given of spectroscopic investigations of vapours of metals and metallic oxides heated in this type of furnace.

H. J. T. ELLINGHAM.

Tantalum as cathode for electrodeposition of copper. STROCK and LUKENS.—See X. Measuring the effect of water on varnish films. KOPP.—See XIII. Moisture determination in wheat. BURTON and PITT.—See XIX. Purification of water. ILLIG.—See XXIII.

PATENTS.

[Electric] muffle furnace. P. H. P. MONCKTON, Assr. to W. H. CROSS (U.S.P. 1,724,956, 20.8.29. Appl., 23.11.27. S. Afr., 16.9.27).—The furnace comprises a casing lined with refractory material and containing a muffle chamber spaced from the inside walls of the lining and heated internally by electric resistors. The

lower part of the heating chamber is provided with a series of holes through which air, preheated while passing through the above-mentioned space, is admitted to the chamber. A. R. POWELL.

Primary battery. M. L. MARTUS, E. H. BECKER, and J. G. ROSS (U.S.P. 1,725,716, 20.8.29. Appl., 21.12.26).—A copper oxide negative element is formed of a number of parts in contact with the oxide, one of these parts being electrically connected to a positive terminal of the battery, and another from this terminal until the battery has been discharged to a predetermined degree. The reduced copper oxide is automatically caused to bridge the insulation. J. S. G. THOMAS.

Galvanic battery. F. C. F. PORTAIL, Assr. to Soc. ANON. LE CARBONE (U.S.P. 1,716,461, 11.6.29. Appl., 14.6.27. Fr., 13.5.27).—The battery comprises a porous carbon anode rendered impermeable to liquids, but remaining permeable to gases, a zinc or galvanised iron cathode, and an electrolyte consisting of a solution containing 200 g. of zinc chloride and 50 g. of zinc bromide per litre. The anode consists of a mixture of 200 pts. of granulated charcoal and 100 pts. of granulated lead moistened with 20% zinc chloride solution and held around a carbon rod by means of a porous diaphragm. A. R. POWELL.

Making dry-storage-battery plates. B. HEAP, Assr. to ELECTRIC STORAGE BATTERY CO. (U.S.P. 1,725,734, 20.8.29. Appl., 5.1.25).—Wet, charged, negative plates are dipped into a solution of gelatin containing formaldehyde and, while still wet, are heated in a non-oxidising atmosphere, so that an oxidation inhibitor, soluble in electrolyte, is produced. J. S. G. THOMAS.

Electric discharge tube. G. L. HERTZ, Assr. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,726,107, 27.8.29. Appl., 18.2.25. Holl., 1.4.24).—A discharge tube, utilising the light from the positive column, is filled with argon and mercury vapour. J. S. G. THOMAS.

Electrical discharge device. K. H. KINGDON, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,725,281, 20.8.29. Appl., 26.2.23. Renewed 26.5.28).—Electrodes, including a thermionic thoriated cathode, are enclosed in an envelope filled with hydrogen at a pressure sufficiently high to reduce space-charge by positive ionisation. J. S. G. THOMAS.

Thermionic electrode. G. M. J. MACKAY, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,716,545, 11.6.29. Appl., 6.3.26).—An electrode for a thermionic valve comprises a sealed nickel tube containing caesium metal, which slowly diffuses through the walls during use, and thereby increases the emissivity of the valve. A. R. POWELL.

Thermionic cathode. WESTINGHOUSE ELECTRIC & MANUF. CO., Assees. of H. MCK. ELSEY and A. T. KROGH (B.P. 291,400, 25.5.28. U.S., 1.6.27).—A metallic core is coated with a mixture of one or more compounds of the alkaline-earth metals, preferably their hydrazides and/or carbonates, in a vehicle, e.g., organic alcohol, aldehyde, or ketone, especially methyl or ethyl alcohol, which is volatile below 200° and provides a reducing atmosphere when heated. The coated core is heated in carbon dioxide. J. S. G. THOMAS.

Electrochemical treatment of saline and alkaline solutions. H. C. PARKER (U.S.P. 1,726,236, 27.8.29. Appl., 26.9.28).—A solution containing sodium chloride and sodium carbonate is electrolysed in a cell having inlets and outlets for liquid and provided with a movable iron anode in such manner that iron hydroxide and sodium bicarbonate are formed and separated from the solution. J. S. G. THOMAS.

Manufacture of electrical insulating and filling materials, particularly liquid materials. BERRY, WIGGINS, & CO., LTD., and H. H. HOLMES (B.P. 317,717, 16.5.28).—The sp. gr. of a liquid or semi-liquid electrical insulating or filling material is increased by adding a solution either of a metallic salt of an organic acid in an organic solvent, or of halogen waxes or naphthalene in mineral oils. Thus a solution of barium oleate or resinate in oleic acid or of halogenated naphthalene in resin oil may be employed. J. S. G. THOMAS.

Measuring the humidity of gases or gaseous mixtures. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 317,306, 12.5.28).—The dew point is determined by means of a fluid-cooled mirror immersed in the gas, the presence or absence of dew being indicated by a beam of light reflected on to a selenium cell or other photo-electric device. The temperature of the mirror is recorded by an electrical thermometer. Immediately on the formation of dew, relays in circuit with the selenium cell cause the cooling to be stopped and electrical heat to be applied, also the current to the thermometer is cut off to prevent overshooting downwards. On disappearance of the dew the relays act in the reverse direction, allowing the thermometer to record again and starting a fresh cycle. B. M. VENABLES.

Electric lamp for use in mines. W. M. THORNTON (U.S.P. 1,727,185, 3.9.29. Appl., 24.9.28. U.K., 11.10.27).—See B.P. 298,767; B., 1928, 900.

Heating-furnace apparatus (U.S.P. 1,723,319).—See I. Liquid polymerisation products of hydrocarbons (B.P. 317,344).—See III. Hydrogen peroxide (B.P. 316,919).—See VII. Cement kiln (B.P. 316,715).—See IX. Lead ores (U.S.P. 1,726,258). Aluminium-plated articles (U.S.P. 1,723,277). Nickel (B.P. 299,375). Chromium (B.P. 317,137 and 303,884).—See X. Surface markings on rubber (B.P. 317,620).—See XIV. Impurities in water (B.P. 317,278).—See XXIII.

XII.—FATS; OILS; WAXES.

Laurel fat, especially its optical activity. G. WALLRABE (Arch. Pharm., 1929, 267, 405—412).—The fat was obtained as a clear, yellow, buttery mass by extracting the fruit, carefully freed from shells, with light petroleum. The following constants were determined for both the crude fat and the purified fat which had been freed from essential oils by distillation in steam. The values for the purified fat are given in parentheses; acid value 5.31 (6.06), saponif. value 210.85 (219.6), iodine value (Hübl) 67.94 (65.7), apparent acetyl value 15.27, unsaponifiable matter (5.0%), insoluble fatty acids (84.69%), $[\alpha]_D^{25} +11.21^\circ (+8.43^\circ)$. The fatty acids yielded 58.5% of liquid

fatty acids (mol. wt. 306) and 37.5% of solid fatty acids (mol. wt. 228.7, m.p. 38.5–39°) by Twitchell's method. The liquid fatty acids contained both oleic and linoleic acids and a higher member of the series. The unsaponifiable matter contained phytosterol, melissyl alcohol, laurane, and a liquid portion. The optically active constituent $C_{22}H_{30}O_4$ was isolated by fractional crystallisation of the crude fat from alcohol at temperatures from –15° to –50°. It loses water at 103–104° and blackens at 230–250°, has $[\alpha]_D^{25} +137.65^\circ$ in alcohol, iodine value (Hübl) 111.3, and is very unstable in solution. The essential oil had $[\alpha]_D^{25} -20.02^\circ$.

S. COFFEY.

Cacao butter. D. W. HORN and A. OSOL (Amer. J. Pharm., 1929, 101, 601–611).—Comparative tests on cacao butter, cacao-butter oleine and stearine, and cacao butter from "Dutched" nibs are recorded. No differentiation was possible from the specific gravities, saponification values, unsaponifiable matter, or liquid fatty acid contents. Very slight differences were shown in the refractive indices at 40°, surface tensions at 40°, and viscosities (Stormer) at 80°. Definite indications of differences were shown in the determinations of iodine value (Hanus), m.p. (Wiley), "transition points," and, possibly, of acidity. The "transition point" is the maximum temperature attained on crystallisation of a super-cooled fat. The surface tensions of the four products lay within the limits 34.9–35.7 dynes/sq. cm.

E. H. SHARPLES.

Differential halogen absorption of oils and fats. J. W. CROXFORD (Analyst, 1929, 54, 445–453).—The iodine, bromine, and chlorine values of a series of oils and fats have been determined, the first by the Wijs method, the second volumetrically, and also gravimetrically by Toms' method (B., 1928, 236), and the third by a modification of the gravimetric bromine vapour method. From the large number of values obtained it is concluded that the bromine vapour method compares very favourably with the Wijs method for determining the degree of unsaturation, requires a smaller sample, and is usually much more rapid, particularly with croton oil, but castor oil and its predominant (ricinoleic) acid readily undergo substitution. The difference between the absorption for 1 hr. with the reagents (Wijs' and bromine vapour) probably enables an opinion to be formed as to the position of the unsaturated linking in the fatty acids of the oleic series. Substitution occurs in most cases by the chlorine vapour method, but appears to reach a maximum. Other unsaturated organic substances such as petroselinic, tiglic, and cinnamic acids, cinnamyl alcohol, etc. gave results with bromine vapour and chlorine agreeing closely with the theoretical, but maleic and fumaric acids were found very inert under treatment. The method could be used to determine the amount of unsaturated, in admixture with saturated, compounds.

D. G. HEWER.

Emulsions of fats and hydrocarbons and their industrial application. L. MEUNIER (J. Soc. Leather Trades' Chem., 1929, 13, 309–321).—Information on the nature and industrial application of emulsions of petroleum distillation residues, bitumen, asphalt, rubber latex, mineral oil, aqueous petrol emulsions, water in crude petroleum, milk, butter, margarine, textile oils,

tannery emulsions, and emulsions formed during the commercial saponification of oils and fats, is summarised.

D. WOODROFFE.

Drying of [fatty] oils. R. S. MORRELL and S. MARKS (J. Oil Col. & Chem. Assoc., 1929, 12, 183–205).—Recent theories on the drying of oils are discussed. β -Elaeostearic glyceride has been oxidised in the solid state and in solution in carbon tetrachloride or benzene and the resulting gel examined by chemical methods and its peroxide nature established. On methylation, the solid gel yielded a polymerised peroxydihydroxy-methyl ester and a peroxyhydroxymethoxy-methyl ester of simple mol. wt., showing that the polymerisation in the gel is of an intramolecular character, which is associated with the modified peroxide group contiguous to the glyceryl radical. A difference in the behaviour of the peroxide groups in the gel is indicated: one group is definitely acidic, passing to $\cdot C(OH):C(OH)\cdot$ and the keto-form, and is the cause of the yellowing of drying oils; the other group, with which the initial gelation may be connected, has indications of basic properties, is much less stable, and gradually disappears with time. In addition, a soluble oxidation product could be separated from the gel by light petroleum, which on methylation yields products similar to those obtained from the gel, but of a simpler nature and devoid of peroxide properties; there is evidence of the existence of hydroxyl and carbonyl groups in these derivatives. In both oxidation products there is a persistence of the unsaturated character as shown by the iodine value. Piperidine (cf. Ruhemann and Browning, J.C.S., 1898, 23, 723) combined directly with β -elaostearic glyceride to the amount of 41% of the base at ordinary temperatures and 88% at 100° (calc. for one reacting weight of each substance), yielding rhombic plates, m.p. 71–72°, soluble in alcohol and warm light petroleum. The study of the action of piperidine on drying oils is being continued.

E. LEWKOWITSCH.

I. Marine animal oils. II. Influence of solute on the molecular depression of the freezing point in benzene and nitrobenzene. M. T. FRANÇOIS (Bull. Mat. Grasses, 1929, 189–202).—I. Investigations on the higher saturated and unsaturated alcohols in spermaceti and sperm oil are reviewed (cf. André and François, B., 1926, 247, 987; 1927, 706). Tetra-, hexa-, and octa-decyl alcohols (m.p., respectively, 38°, 49.5°, 61°) are to be found in both these substances; the occurrence in sperm oil of oleyl alcohol (cf. Toyama, B., 1924, 223) and of small amounts of a diethylenic ("erucyl") alcohol $C_{22}H_{42}O$ are noted. By hydrogenation of sperm oil over a nickel catalyst large quantities of octadecyl alcohol, identical with the reduction product of ethyl stearate, are obtained. By condensation of suitable Grignard reagents with the bromides of cetyl, octadecyl, and oleyl alcohols the author has prepared heptadecyl alcohol, m.p. 54°, nonadecyl alcohol, m.p. 63–64, and the monoethylenic hydrocarbons ethyloleylene, $C_{20}H_{40}$, and butyloleylene ("erucylene") $C_{22}H_{44}$.

II. Cryoscopic methods must be applied with reserve to certain substances: the "pseudo-constant" K for benzene and nitrobenzene with the higher fatty acids and alcohols with even numbers of carbon atoms

increases gradually to values considerably above the normal value as the chain lengthens. The suggested two steady values ("normal" and "abnormal") do not exist for these two solvents. E. LEWKOWITSCH.

Some analytical aspects of cod-liver oil. A. E. BRIDG, R. VAN WINKLE, A. E. JURIST, and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1929, 18, 771—778).—The following constants of an average sample of Newfoundland cod-liver oil have been determined: d_{25}^{25} 0.9221, iodine value (Hanus) 157.2, saponif. value 186.1, viscosity at 100° F. (Saybolt) 160 sec., unsaponifiable matter 0.88%, apparent acetyl value 11.1 (true value 8.7), oxidised fatty acids 0.50%, oxidisability value 12.2—18.7, iron content 1.3—4.7 pts. per 10,000,000, and sulphur content 0.03—0.2%. Sulphur has been found both in American and European oils, and the amount present appears to depend on the length of time which elapses before the oil is removed from the liver. No sulphide-sulphur is present. Of six recognised tests for the detection of peroxides, none was satisfactory, and a guaiacum-haemoglobin test has been devised which is very sensitive and gives a positive reaction with nearly all crude oils. E. H. SHARPLES.

Whiting and [discoloration of] linseed oil. R. G. BROWNING (J. Oil & Col. Chem. Assoc., 1929, 12, 211—219).—From experiments made with artificial whittings and linseed oil it appears that the amount of free lime present has little influence on the rapidity of discoloration. Since addition of ignited pure ferric oxide caused no abnormal action, but traces of a carefully dried hydrated iron hydroxide (precipitated in the presence of carbon dioxide) caused an immediate brown discoloration, it is concluded that such discoloration is due to the presence of iron in an active condition.

E. LEWKOWITSCH.

Influence of heat on the colour of soya-bean oil, and decomposition at 180—225° under the influence of nickel catalysts. H. I. WATERMAN and M. J. VAN TUSSENBROEK (Chem. Weekblad., 1929, 26, 410—413).—The effect of heating in air and *in vacuo*, with and without addition of nickel, has been examined. The increases in colour depth and viscosity caused by simple heating are intensified by the presence of nickel. The iodine value is unaffected by heating in air or in a vacuum, but is reduced considerably by heating with nickel. The thiocyanate value is little affected. S. I. LEVY.

Constituents of soya-bean [foots]. M. E. HOLZ (Seifensieder-Ztg., 1929, No. 13—17; Bull. Mat. Grasses, 1929, 208—213).—An account of the preparation and properties of soya-bean meal and oil is given. The foots from the oil contain 48.67% of lecithin, extraction of which in a pure state was attempted. By extraction with ether and precipitation with acetone a yellowish, soft, friable mass, insoluble in hot alcohol, and containing 98.5% of phosphatide, was obtained. This product and Merck's lecithin ("purissimum") contained, respectively, phosphorus 3.89, 3.94; nitrogen 1.22, 1.75; sulphur 0.54, 0%. The new product resembled lecithin in colour, consistency, odour, and taste, and appeared to be analogous to the phosphatides. Methods for the rapid precipitation of foots are referred to.

E. LEWKOWITSCH.

Pistachio oil. K. BEYTHIEN (Pharm. Zentr., 1929, 70, 551—558, 571—573).—Extraction with light petroleum of the kernels of *Pistacia vera* from the Levant gave 25.6% of a golden-yellow oil (d_{15}^{15} 0.91484) which produced an olive-green colour after dilution with carbon disulphide and addition of sulphuric acid. An expressed oil had d_{15}^{15} 0.91370, d_{19}^{19} 0.91355, n_D^{25} 1.4655, saponif. value 195.31, iodine value (Hübl) 87.90, Hehner value 93.7, Reichert-Meissl value 0.77, Polenske value below 0.3, acetyl value 19.19, unsaponifiable matter (contains phytosterol) 0.226%, and total fatty acids (m.p. 34—37°, solidification pt. 28—30°, iodine value 93.70, mol. wt. 274.85) 93.5—94.4%. The fatty acids were composed of 81.74% of liquid acids (α -linoleic 8.41—9.46%, β -linoleic 14.78—15.03%, oleic 70.28—70.70%, hydroxy-acids about 6.0%), and 17.92% of solid acids (apparently all palmitic acid; stearic, lignoceric, and arachidic acids could not be detected). A small amount of an essential oil having a terpentine-like odour was isolated by steam-distillation. E. H. SHARPLES.

Petroleum spirit test for purity of castor oil. T. T. COCKING and S. K. CREWS (Quart. J. Pharm., 1929, 2, 217—226).—Pure castor oil will not satisfy the requirements of the pharmacopoeial solubility test (B.P. 1914) unless, contrary to the pharmacopoeial description, the petroleum spirit contains some aromatic hydrocarbons. With pure hexane and genuine castor oils the clearing points of the different samples varied over such a range as to invalidate any such solubility test.

C. C. N. VASS.

Thiocyanogen value of strophanthus oil, and of oils of the chaulmoogra group. E. I. VAN ITALLE (Pharm. Weekblad, 1929, 66, 677—683).—The fact that only one double linking of acids and glycerides containing two ethylenic linkings is saturated by thiocyanic acid has been employed to deduce for strophanthus oil the composition: saturated acids 25—27, oleic acid 44—48, and linoleic acid 25—30%. Examination of chaulmoogra oil, hydnocarpus oil, and gorli fat (from the seeds of *Oncoba echinata*) by the same method indicates that no acid of the linoleic series is present. S. I. LEVY.

Effect of changes of hydrogen-ion concentration on emulsions of the water-in-oil type. J. C. KRANTZ, JUN., and N. E. GORDON (J. Amer. Pharm. Assoc., 1929, 18, 797—805).—The influence of changes in p_H on emulsions of water in olive and mineral oils, using magnesium oleate as emulsifying agent, has been examined and also the surface tension, viscosity, and particle size. The p_H range at which the emulsions were most stable was p_H 11—12.5, the stability decreasing with a lowering of p_H . The emulsions were also most stable when freshly prepared magnesium oleate was used. E. H. SHARPLES.

Solvents for waxes. O. A. PICKETT (Ind. Eng. Chem., 1929, 21, 767—768).—Solubility-temperature data for beeswax, candelilla wax, carnauba wax, and montan wax in the following solvents were obtained: "V.M. and P. Naphtha" (petroleum hydrocarbons, b.p. range 100—174°), wood turpentine, "No. 22 Thinner" (55% of terpene hydrocarbons, 45% of petroleum hydrocarbons), Solvenol, pine oil, sulphonated castor oil, and a 1:1 mixture of the last two oils. The method used was to observe the temperature of

precipitation on cooling hot wax solutions of known concentration. S. S. WOOLF.

Analysis of oils and fats. BURTON and ROBERT-SHAFF.—See XV.

PATENTS.

Production of solid [lubricating] greases. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 317,630, 3.8.28).—Liquid mineral oils, rape oil, train oil, etc. are incorporated with bleached montan wax (or its conversion products, *e.g.*, esters, salts) at about 100°, sufficient alkali (dissolved in a little water) being added to neutralise the free acids in the wax. E. LEWKOWITSCH.

Production of [vitamin-bearing fish-liver] oils. F. W. NITARDY, Assr. to E. R. SQUIBB & SONS (U.S.P. 1,725,964, 27.8.29. Appl., 29.1.24).—The oils are extracted without reduction in vitamin content by cooking the livers at not above 100° under reduced pressure, and cooling, separating, decanting, and storing the oil in an atmosphere of an inert gas. E. LEWKOWITSCH.

Manufacture of sulphonated linseed oil. ORANLIEN-BURGER CHEM. FABR. A.-G., Assees. of CHEM. FABR. MILCH A.-G. (B.P. 293,806, 12.7.28. Ger., 12.7.27).—A substantial proportion of oleic acid, or fatty matter containing it (*e.g.*, olive oil), is added to linseed oil before sulphonation in the usual manner, whereby (after washing and neutralisation as required) a clear, water-soluble oil is formed, suitable for oiling, smoothing, etc. in the leather and textile industries. E. LEWKOWITSCH.

Decolorising and refining crude cottonseed oil. W. S. BAYLIS, Assr. to FILTROL CO. OF CALIFORNIA (U.S.P. 1,725,895, 27.8.29. Appl., 1.11.24).—The oil is heated at not above 43° and intimately mixed with water, the water etc. removed from the oil, the oil mixed with activated clay, and the clay together with impurities carried by it is then separated. E. LEWKOWITSCH.

Modification of drying oils. J. E. BOOGE and C. COOLIDGE, Assrs. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,725,561, 20.8.29. Appl., 7.3.28).—The oils are heated at a moderate temperature in the absence of an oxidising gas, and the product is blown at a lower temperature with a gas containing oxygen. E. LEWKOWITSCH.

Separating impurities from oils (B.P. 308,752).
Soaps from mineral oil sludge (U.S.P. 1,718,335).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Titania and titanium white. C. P. VAN HOEK (Farben-Ztg., 1929, 34, 2828—2832).—The properties of titanium white pigments are discussed (with a full bibliography); it is concluded that these are unsuitable for paints as the coatings formed are too soft and chalk very rapidly in exterior use, and that the greatly increased covering power conferred by small additions of titania to other pigments constitutes its value to the industry. E. LEWKOWITSCH.

Pigment form. E. KLUMPF (Farben-Ztg., 1929, 34, 2612—2614).—A recapitulation of the author's view that oil absorption depends on interstitial volume at

close packing. When a pigment absorbs appreciably more than 66% of its own volume of oil it consists of secondary particles. Grinding such secondary particles will lower the oil absorption by producing a system more closely packed. The thickness of "oil sheath" around individual particles at close packing is shown to be so small as to have no appreciable effect on the observed oil absorption. Two types of secondary particles exist, *viz.*, those due to cohesion and adsorption, respectively. The former break down to primary particles on intensive grinding, giving the reduced oil absorption corresponding to close packing; the latter, however, owe their condition to adsorption of foreign material, *e.g.*, moisture, and retain their secondary structure even when intensively ground. The thickening of paints is probably due to formation of such adsorptive secondary particles, analogy being drawn to the thickening of a sol when precipitated to form a gel. S. S. WOOLF.

Course of the distillation and evaporation of solvents and thinners for nitrocellulose lacquers and their mixtures. H. JORES (Farben-Ztg., 1929, 34, 2886—2892).—The rates of evaporation and distillation for 65 lacquer solvents, thinners, and mixtures of these are recorded. No definite relation can be established between the two processes, although the generalisation may be made that, as a rule, the lower-boiling liquids evaporate the more rapidly; exceptions, however, *e.g.*, alcohol, b.p. 78—79°, and toluol, b.p. 97—142°, which evaporate at practically the same rate, are noted. The speed of evaporation of mixtures may be greater or less than that calculated from the observed value for the constituents. E. LEWKOWITSCH.

Determining the effect of water on varnish films. H. KOPP (Farben-Ztg., 1929, 34, 2892—2893).—A simple device is described which automatically records the time taken before the breakdown of insulating power occurs in a varnish film exposed to water; the value obtained is a measure of the susceptibility of the film to moisture. E. LEWKOWITSCH.

Use of Thénard's blue. DITMAR and PREUSZE.—See XIV.

PATENTS.

Production of white lead. F. T. BAILEY and W. AUSTIN (U.S.P. 1,720,196, 9.7.29. Appl., 28.1.26).—Corroded white lead is prepared by whipping into atmospheric suspension a mixture in water of lead oxide and acetic acid in a revolving closed container, through which air passes and carbon dioxide circulates at a definite rate. S. S. WOOLF.

Elimination of poisonous constituents from white lead. L. BEVILACQUA (B.P. 315,637, 11.10.28).—A continuous stream of sodium bicarbonate solution is introduced into the tanks in which white lead prepared by the Dutch process is softened; lead acetate is thereby converted into carbonate. The pulp is then washed in filter presses or by decantation until sodium acetate is completely eliminated. S. S. WOOLF.

Manufacture of coloured opacifying pigments. C. J. KINZIE, Assr. to TITANIUM ALLOY MANUF. CO. (U.S.P. 1,719,432, 2.7.29. Appl., 21.1.27).—Opacifying pigments for vitreous enamels are prepared by mixing

a colouring material, a zirconium compound, and a flux, heating the mixture until the ingredients are thoroughly incorporated, and cooling the product.

S. S. WOOLF.

Mixing colours with varnish, oil, and the like. KRAUSEWERK A.-G. (B.P. 306,926, 21.4.28. Ger., 29.2.28).—Residual water is expelled from washed pigments by means of a water-miscible liquid, *e.g.*, alcohol, which with the pigment forms a stirrable paste. The paste is mixed with a varnish or oil, non-miscible with the added liquid, so that minute pigment-varnish or pigment-oil aggregates are formed. These are coalesced by churning, nearly all the "added liquid" being expelled.

S. S. WOOLF.

Coating surfaces with cellulose varnishes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 317,987, 20.8.28).—To prepare surfaces of wood, metal, etc., or other cellulose coatings, for the reception of cellulose varnish, an intermediate coating of a solution of cellulose esters or ethers of the higher fatty acids (*e.g.*, in benzene, chlorobenzene) is applied; resins, rubber, fillers, etc. may be added.

E. LEWKOWITSCH.

Coating of solid surfaces [with synthetic resins]. DUNLOP RUBBER Co., LTD., and E. E. WHITE (B.P. 317,350, 14.5.28).—Impervious surfaces, *e.g.*, of metals, are partially or wholly coated with aqueous dispersions of synthetic resins, *e.g.*, of the phenol-formaldehyde type, with rubber and sulphur, the synthetic resin being present in the optimum proportion for adhesion; other dispersed substances may also be present. The deposits are dried and then heated in the presence of air.

D. F. TWISS.

Production of transfer pictures and their application for improving wood and other surfaces. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 317,871, 21.5. and 29.11.28).—The transferable adhesive layer of transfers (preferably made by printing etc. with lacquers or pastes containing cellulose esters or ethers on a gum-coated paper base) contains cellulose esters or ethers and/or resins which are both soluble in alcoholic solvents. The transfers are applied after moistening with alcohol, and the picture so affixed will withstand polishing after a very short time; extra priming layers comprising alcohol-soluble cellulose derivatives and resins and also protective polishing layers may be included in the transfer.

E. LEWKOWITSCH.

Refining wood rosin. H. E. KAISER and A. LANGMEIER, Assrs. to HERCULES POWDER Co. (U.S.P. 1,719,431, 2.7.29. Appl., 15.5.23).—Wood rosin is heated at 260–343° while under a vacuum corresponding to an absolute pressure of 3 in. of mercury or less, and a portion of the rosin is condensed at 177° or over.

S. S. WOOLF.

Manufacture of synthetic resins. G. T. MORGAN and A. A. DRUMMOND (B.P. 315,442, 13.2.28).—A mixture of a phenol, a solid polymeride of formaldehyde, a catalyst, and an organic solvent for the phenol which does not react so as to form part of the resin, is heated. The resin solution produced (after acidification, if an alkaline catalyst has been used) is washed, preferably with saturated sodium thiosulphate solution, and dried. The dried solution is then used directly in known

manner, or the resin is obtained by evaporation or by precipitation with a suitable solvent in which an inert pulverulent material may be suspended.

S. S. WOOLF.

Plastic composition. R. A. NORTON, Assr. to SELDEN Co. (U.S.P. 1,720,051, 9.7.29. Appl., 15.10.27).—A thermal-hardening phenol-aldehyde condensation product in the fusible state is mixed with a high-boiling ester of a polybasic, aliphatic acid and alcohols having less than three hydroxyl groups.

S. S. WOOLF.

Moulding compositions. K. RIPPER (B.P. 287,568, 22.3.28. Austr., 25.3.27).—Thiourea, or a mixture of thiourea and urea containing not less than 1 mol. of the former to 1 mol. of urea, is condensed with formaldehyde in boiling aqueous solution, the proportion of formaldehyde being not more than corresponds to 3 mols. to 1 mol. of thiourea in the first case, or to 2 mols. to 1 mol. of each of the other two starting substances in the second. The resinous condensation product is brought into intimate contact with cold water and thereby reduced to a fine powder suitable for moulding by hot-pressing. An opaque final product is obtained by condensation of the thiourea and urea simultaneously, or a transparent, clear article by initiating the condensation with thiourea before adding a solution of urea in formaldehyde.

E. LEWKOWITSCH.

Production of non-turbid aldehyde condensation products. R. A. NORTON, Assr. to SELDEN Co. (U.S.P. 1,720,052, 9.7.29. Appl., 12.3.28).—An aldehyde is condensed with a resinophoric compound, *e.g.*, phenol, in the presence of an alkaline condensing agent, a polycarboxylic acid compound is added, free water is substantially removed, and the product is hardened by heat.

S. S. WOOLF.

Coating compositions. W. P. DAVEY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,726,473, 27.8.29. Appl., 24.8.25).—See B.P. 257,243; B., 1927, 916.

Organic salts of dyes (B.P. 316,370). Azo dyes (B.P. 298,907 and 294,583).—See IV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Use of Thénard's blue in accelerated rubber mixtures. R. DITMAR and K. H. PREUSZE (Gummi-Ztg., 1929, 43, 2749–2750).—Thénard's blue withstands unaccelerated vulcanisation by sulphur, vulcanisation by sulphur chloride, and vulcanisation aided by ultra-violet radiation. It is also colour-stable to vulcanisation in the presence of most organic accelerators, but is affected by aldehyde-ammonia and certain aldehyde-aniline condensation products; it is also neutral to most accelerators, but activates a few, *e.g.*, the condensation product of butaldehyde and dimethyl-*p*-phenylenediamine. It is not satisfactory for colouring vulcanite.

D. F. TWISS.

Activating effect of various metal oxides on the accelerating action of piperidine pentamethylenedithiocarbamate, and cyclohexylethylamine dithiocarbamate in the vulcanisation of rubber. R. DITMAR and A. MATHIESEN (Chem.-Ztg., 1929, 53, 479).—Lime, strontia, and baryta have the greatest activating effect on the vulcanisation of rubber by colloidal sulphur in the presence of the above two catalysts. The oxides

of zinc, cadmium, arsenic, and antimony are almost as active, but their sulphides, stearates, and carbonates are inactive.

A. R. POWELL.

Emulsions of fats etc. MEUNIER.—See XII.

PATENTS.

Treatment of [rubber] latex. NAUGATUCK CHEM. Co., Assees. of J. MCGAVACK (B.P. 298,628, 1.9.28. U.S., 14.10.27).—A gel of uncoagulated rubber which can be redispersed in water or other non-solvent, is obtained by treating latex with a small proportion of a hydrosol of silicic acid so as to obtain subsequent gelation, *e.g.*, within a period between 30 sec. and 48 hrs. The hydrosol of silicic acid may be introduced as such or formed *in situ*, *e.g.*, from sodium silicate and boric acid.

D. F. TWISS.

Rubber latex-protein compound. W. W. CHRISTMAS (U.S.P. 1,724,906, 20.8.29. Appl., 27.3.24).—Casein is dissolved in concentrated ammonia solution at 70–95°, rubber latex is added, and the mixture dried and vulcanised.

A. R. POWELL.

Retarding deterioration of rubber. W. S. CALCOTT, W. A. DOUGLASS, and O. N. HAYDEN, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,725,564, 20.8.29. Appl., 19.11.27).—Two or more substances are incorporated into rubber, one primarily retarding the deteriorating influence of heat and the other primarily retarding deterioration by oxygen.

D. F. TWISS.

Application of surface markings to vulcanised rubber. CALLENDER'S CABLE & CONSTRUCTION Co., LTD., and J. BOWYER (B.P. 317,620, 27.7.28).—Markings are made with copying ink or other material not containing rubber, on a layer of suitable material, *e.g.*, transfer paper; this is secured against the rubber, which is then submitted to vulcanisation, during which the markings become transferred to the rubber surface. The process is particularly applicable to marking the insulation rubber of electric conductors.

D. F. TWISS.

Production of rubber substitutes. J. W. MOUNSEY, W. T. GALBRAITH, and H. G. BAILEY (B.P. 317,814, 23.5.28).—Seaweed, after agitation with hydrochloric acid and washing with alcohol, is mixed with gummy substances; the mass is then admixed with such starchy materials as vinal residuals which have been subject to fermentation in a closed vessel by *Granulobacter butylicus* and *Bacillus orthobutylicus*. The mass is then mixed with sodium and treated with such gases as carbon dioxide-chlorine, ammonia, or ether vapour; resin, fish oil, and other ingredients are then added, also casein and a vegetable oil. After heating for 1 hr. at 120° a rubber-like product is obtained.

D. F. TWISS.

Manufacture of plastic and elastic polymerisation products of diolefines. I. G. FARBENIND. A.-G. (B.P. 292,103, 8.6.28. Ger., 13.6.27).—Polymerisation is greatly accelerated and the product improved by emulsifying the diolefines and polymerising in the presence of hydrogen peroxide. With a mixture of 30 c.c. of cow's milk, 20 c.c. of isoprene, and 3 c.c. of 3% hydrogen peroxide at 95°, polymerisation in a closed vessel occupies 4–5 days.

D. F. TWISS.

Vulcanisation of rubber. C. O. NORTH and C. W.

CHRISTENSEN, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,726,713, 3.9.29. Appl., 19.5.24).—See Can. P. 258,626; B., 1927, 393.

Coating of solid surfaces (B.P. 317,350).—See XIII.

XV.—LEATHER; GLUE.

Tannins and non-tannins of tanning extracts.

New theory of tanning. V. S. SADIKOV (Biochem. Z., 1929, 210, 296–333).—The behaviour of tannins and non-tannins to hide powder and to ammonium carbonate using oak, valonia, quebracho, pine bark extracts, and extracts of the rhizomes of *Saxifraga* or *Bergenia crassifolia* is investigated. Tannins are divided into K-tannins which are, and K₀-tannins which are not, absorbed by hide powder (the K₀ fraction representing the analytical non-tannin). The K-tannins are further divided into L-tannins (oxidisable by Lowenthal's method) and the L₀-tannins (not oxidisable). A series of tables give the values for dried residues, soluble and insoluble substances, tannins, and non-tannins in each type of extract, and the variation of each fraction of the tannins (K, L, etc.) on treatment with a series of increasing concentrations of ammonium carbonate. It is suggested that under the action of ammonia and amines, liberated by enzymic hydrolysis of the hide material, the tannins are converted into insoluble substances which are deposited in a thin layer over the collagen fibres.

P. W. CLUTTERBUCK.

Precipitation figures of basic chrome alum [tanning] liquors. W. SCHINDLER and K. KLANFER (Collegium, 1929, 282–300).—One-bath chrome tanning liquors are controlled better by using the "precipitation quotient" (*Q*), which is the change in *p_H* value required to diminish the precipitation figure by 1 c.c. of 0.1*N*-sodium hydroxide in 100 c.c. of a chrome tanning liquor containing 1 g. of Cr₂O₃ per litre, and is given by: $Q = (p_H \text{ at precipitation point} - p_H \text{ of liquor}) \times \text{precipitation figure}$. The value of *Q* is often a better measurement of the astringency than is the precipitation figure; the former value was not much affected at low temperatures by the concentration of the sodium carbonate solutions used to render the chrome alum solutions basic. Small differences disappeared on keeping. Greater and more permanent differences were found with liquors of 45% basicity at 40°, and lower *Q* values were obtained by ageing the chrome alum solution before rendering it basic. Only small variations in *Q* and in the precipitation figures were observed on varying the temperature, the speed of addition and concentration of the sodium carbonate solutions, and on ageing the chrome alum solutions in preparing liquors of 25% basicity. These precipitation-control figures were determined for liquors of different basicities shortly after they had been rendered basic and also 24 hrs. later. The precipitation figures diminished proportionately with increasing basicity. The "precipitation quotient" remained practically constant to about 30% basicity, then increased slowly at first and more rapidly later. The tanning properties of liquors of different basicities were found to be more closely allied to the changes in *Q* than to the precipitation figures. Small additions of iron caused a considerable increase in the *Q* value. D. WOODROFFE.

[Qualitative] analysis of [vegetable] tanning materials by luminescence. V. KUBELKA and V. NĚMEC (Collegium, 1929, 300—303).—The tannin solution (20 c.c.) is shaken with 10 c.c. of ethyl ether or ethyl acetate, allowed to separate, and the upper layer poured off into a beaker and examined by the light of a quartz lamp. Cotton impregnated with the solution sometimes intensifies the fluorescence. Characteristic colours have been observed and also colour changes with certain tanning materials on rendering the solution alkaline or acid, respectively. A specific reaction is given for gambier; when 5 c.c. of ethyl alcohol are mixed with 5 c.c. of this tannin solution, a characteristic greyish-violet fluorescence can be seen in ultra-violet light; this changes to green on the addition of 1 c.c. of 10% sodium hydroxide solution. On extracting this solution with 10 c.c. of petroleum spirit, and separating the spirit layer, the latter shows an intense bright greenish fluorescence in ultra-violet light. Small amounts of gambier can be detected in other extracts by this method. D. WOODROFFE.

Acidity of vegetable-tanned leather. W. R. ATKIN and F. C. THOMPSON (J. Soc. Leather Trades' Chem., 1929, 13, 300—309).—Each of three different quantities, *e.g.*, 1, 2, and 4 g. or 1, 3, and 9 g., of the leather is extracted with 100 c.c. of cold 0.1N-potassium chloride for 24 hrs. with occasional shaking, the p_H values of the extracts are determined electrometrically, the results plotted against the logarithms of the dilution of the 14% of water assumed to be present in the leather, and the p_H value of that water is determined by extrapolation. The value obtained is termed the "acid figure" of the leather, and is considered to be more accurate than that given by Innes' method (B., 1928, 721), which represents the p_H of an aqueous extract of the leather. Comparison of the Procter-Searle value and the "acid figure" for various commercial leathers shows that, in general, a high Procter-Searle value corresponds with a low p_H value. Leathers containing less than 0.5% of free mineral acid (Procter-Searle) had $p_H > 2.5$. Leathers containing much free sulphur or which have been dyed with acid dyes are shown to give high Procter-Searle values, but not low acid figures. Innes' method is specific for sulphuric acid; certain other acids interfere. D. WOODROFFE.

Limitations of present methods used in analyses of oils and fats in the leather industry. Report of a Committee of the Society of Leather Trades' Chemists. II. D. BURTON and G. F. ROBERTSHAW (J. Soc. Leather Trades' Chem., 1929, 13, 285—297).—Ammonium salts in an oil are titrated as fatty acids in the determination of the acid value. The Hehner value is subject to errors through incomplete removal of soluble fatty acids, oxidation of the highly unsaturated fatty acids, and the production of anhydrides from the hydroxylated fatty acids. The unsaponifiable matter cannot be determined accurately owing to the retention of moisture, the possibility of oxidation and decomposition, and difficulties in the choice of solvent and separation of the solution of the unsaponifiable matter from the soap solution. Opinions differ on the best method of determining the iodine value. Fahrion's method for the determination of the oxidised fatty acids is generally

accepted. Water in fats can be determined in a water-oven. The toluene distillation method is best for sulphonated oils, but it requires standardisation in every detail. The purity of an oil is doubtful since the variation in the composition of natural oils and fats has not been studied. D. WOODROFFE.

Emulsions of fats and hydrocarbons. MEUNIER.—See XII. **Tannery effluent.** THEIS and LUTZ.—See XXIII.

PATENTS.

Tanning of hides. W. S. SHAW (U.S.P. 1,722,398, 30.7.29. Appl., 23.1.28).—Hides are treated in stages first in a solution of low tannin concentration and then in a more concentrated solution. The former solution is prepared by treating spent liquors from the latter stage with a reagent which converts a portion of the non-tans into tans and then adding a swelling agent for hides. D. WOODROFFE.

Tanning [of hides]. A. ROGERS and R. Y. H. LEE (U.S.P. 1,722,594, 30.7.29. Appl., 18.4.27).—Hide is treated with a material containing a simple non-condensed α -naphthol group in presence of a labile metal salt. D. WOODROFFE.

Preparation of substances composed in part of protein-containing cells for the manufacture of adhesives. G. DAVIDSON, Assr. to I. F. LAUCKS, INC. (U.S.P. 1,724,695, 13.8.29. Appl., 27.6.27).—In order to facilitate its dispersion in aqueous alkali, vegetable material containing protein, after removal of its natural oil, is ground until 95% passes a screen having openings of 0.074 mm. F. G. CLARKE.

Tanning agent. H. SCHÜTTE, Assr. to I. G. FARBE-ENIND. A.-G. (U.S.P. 1,727,135, 3.9.29. Appl., 29.6.27. Ger., 6.9.26).—See B.P. 291,245; B., 1928, 617.

Treatment of skins of animals containing calcified formations. R. A. GERMAIN (U.S.P. 1,725,629, 20.8.29. Appl., 26.5.27. Fr., 3.6.26).—See B.P. 272,199; B., 1928, 723.

Impregnation of solids by liquids (B.P. 317,427).—See I. **Sulphonated linseed oil** (B.P. 293,806).—See XII.

XVI.—AGRICULTURE.

Field trials and the examination of soils by the methods of Mitscherlich, Neubauer, and König. H. VAGELER (Z. Pflanz. Düng., 1929, 8B, 459—470).—Results are recorded of comparative trials of these methods for determining the fertiliser requirements of soils. Best agreement is shown in the figures for potash and least for nitrogen. The value of the various methods in establishing the nature and quantity of fertilisers to be used in practice is discussed. A. G. POLLARD.

Soil survey of Block E (Renmark) and Ral Ral (Chaffey) irrigation areas. J. K. TAYLOR and H. N. ENGLAND (Council for Sci. and Ind. Res., Australia, 1929, Bull. No. 42. 51 pp.).—Mechanical and chemical analyses are recorded and the various soil types discussed from the point of view of irrigation and drainage. A. G. POLLARD.

Utilisation of increasing applications of nitrogen by different varieties of oats. J. WEIGERT and F. FÜRST

(Z. Pflanz. Düng., 1929, 8B, 426—458).—The effects of nitrogenous manuring on oats are most apparent from the middle to the end of May. Only slight differences in the development of different varieties of oats were observed. Nitrogen fertilisers slightly accelerated the formation of shoots and ears, but did not appreciably alter the periods of blossom or ripening. Injury to the crops by the frit fly was most serious where little or no nitrogen had been applied. Increased yields per unit nitrogen used were greatest with the smallest application in the case of grain, and with the next larger application in the case of straw. Relative differences in the yields of the different varieties following the manuring with nitrogen were smaller than those in the case of the other cereals. The weight per unit volume of the grain increased with the smaller treatments, but decreased with the larger ones. A general summary and discussion of the results of trials with this and other cereals (cf. B., 1929, 654) are recorded.

A. G. POLLARD.

Value of sulphur for fertilisation of lucerne.

B. W. DOAK (New Zealand J. Sci. Tech., 1929, 11, 25—29).—Manuring of lucerne with sulphur compounds, *e.g.*, gypsum, superphosphate, and elementary sulphur, produced significant increases in the yield and sulphur content of the crop. The value of the latter which corresponds to a sufficient supply of sulphur appears to be greater than 0.25%, as suggested by Alway (Proc. 1st Internat. Congress Soil Sci.).

A. G. POLLARD.

Determination of manganese in plant materials by the periodate method.

J. DAVIDSON and R. G. CAPEN (J. Assoc. Off. Agric. Chem., 1929, 12, 310—311).—In the determination of manganese by the periodate method (cf. Willard and Greathouse, B., 1918, 41 A), to avoid the tedious removal of hydrochloric acid by boiling with sulphuric and nitric acids the plant ash is dissolved directly in sulphuric, nitric, or phosphoric acid. As iron is present in plant ash in very small amounts, the addition of ferric nitrate is unnecessary, but access of alcohol vapour must be prevented. The plant material, ashed in platinum vessels in an electric furnace, is treated with 5 c.c. of sulphuric, nitric, or phosphoric acid and 20 c.c. of water. To the solution, heated to boiling, is added 0.3 g. of potassium periodate and heating continued until the permanganate colour is fully developed, which in the case of phosphoric acid requires 2—3 min., but longer for the other two acids. The results are in good agreement with those obtained by the official method.

H. J. DOWDEN.

Hydrocyanic acid from calcium cyanide. YOUNG.—See VII.

PATENTS.

Grain-immunising media.

A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,813, 29.5.28).—Fungicidal solutions contain an alkali-soluble complex organic mercury compound, *e.g.*, mercuri *o*-cresoxide, and an iodide, *e.g.*, potassium iodide, or a derivative of cyanamide, *e.g.*, sodium cyanamide.

L. A. COLES.

Agent for control of pests injurious to vegetation. M. B. PATTESON, Assr. to CALIFORNIA FRUIT GROWERS' EXCHANGE (U.S.P. 1,726,364, 27.8.29. Appl., 17.3.26).—See B.P. 267,878; B., 1928, 102.

XVII.—SUGARS; STARCHES; GUMS.

Resistance of wheat starch to diastatic activity.

J. G. MALLOCH (Canad. J. Res., 1929, 1, 111—147).—After a lengthy study of the factors influencing diastatic activity, as evidenced by the amount of reducing sugar produced by taka-diastase, a method has been evolved for measuring the resistance of wheat starch to enzymic hydrolysis. A 10-g. sample of flour, suspended in 100 c.c. of water, is rendered slightly alkaline and then treated with 3 c.c. of 15% sodium tungstate solution to inactivate the natural diastase. After centrifuging and washing, the flour is suspended in a citrate-hydrochloric acid buffer solution (p_H 4.7) at 27°, and to the suspension are added 5 c.c. of buffer solution containing 0.03 g. of taka-diastase, the mixture being then digested for 60 min., diluted to about 175 c.c., and inactivated by adding 3 c.c. of *N*-caustic soda, 3 c.c. of 15% sodium tungstate solution, and 1 c.c. of sulphuric acid, in succession. Having been diluted to 200 c.c., allowing an extra 7 c.c. for the volume of the flour, the suspension is centrifuged for 4 min. and reducing sugar determined in an aliquot portion of the solution. The method has advantages over Rumsey's method (cf. B., 1923, 470 A). It was found that either very fine grinding or extraction with ether for 24 hrs. lowered the starch resistance. Both diastatic activity and starch resistance are influenced by the locality in which the wheat is grown, by the variety of the wheat, and by the maturity at time of cutting. Allowing the grain to sprout produces a marked continuous increase in diastatic activity, whereas the starch resistance at first decreases and then rises again as the sprouting progresses.

H. J. DOWDEN.

Test for invert sugar in honey. GREENLEAF and BROWNE; also NELSON.—See XIX.

PATENTS.

Purifying raw sugar juices. H. SCHREIBER (U.S.P. 1,724,376, 13.8.29. Appl., 21.5.28).—Raw sugar juice is maintained at a temperature below the b.p. until no further separation of non-sugars occurs, the juice is removed from the sediment, and, after adjustment of its p_H by lime, is heated until clear.

E. B. HUGHES.

Maltose products. F. C. WEBER and W. H. RANDALL, Assrs. to FLEISCHMANN Co. (U.S.P. 1,722,775, 30.7.29. Appl., 20.2.26).—Improved yields of maltose products are obtained by diastatically saccharifying starch in the presence of yeast of the true saccharomycetes type.

C. RANKEN.

Removing albuminous substances from saccharine juices, molasses, etc. J. POHLMANN and J. B. F. RASSERS (U.S.P. 1,726,451, 27.8.29. Appl., 13.3.28. Holl., 15.3.27).—See B.P. 287,119; B., 1928, 832.

XVIII.—FERMENTATION INDUSTRIES.

PATENTS.

Manufacture of yeast. J. HASLING, JUN., Assr. to FLEISCHMANN Co. (U.S.P. 1,722,746, 30.7.29. Appl., 30.12.24).—The yeast in a dilute nutrient solution is aerated until the seed yeast is increased approx. 50%.

when one third of the propagating liquid is withdrawn and the yeast separated therefrom. An amount of fresh nutrient solution equal to that withdrawn is added to the fermenter and the yeast reproduction continued.

C. RANKEN.

Manufacture of yeast. L. J. HOWELLS (U.S.P. 1,725,583, 20.8.29. Appl., 20.2.25. Austral., 3.3.24).—The wort is divided into two parts, *A* and *B*, *A* having a high ratio of fermentable carbohydrate to assimilable nitrogen, and *B* a low ratio of fermentable carbohydrate to assimilable nitrogen. The wort *B* is seeded with yeast, and portions of wort *A* are added so that the yeast is constantly supplied with only just sufficient fermentable carbohydrate to maintain unrestricted growth. During the fermentation at 29.4° vigorous aeration is maintained.

C. RANKEN.

Manufacture of butyl and isopropyl alcohols [by fermentation]. A. IZSAK, ASSR. to EASTERN ALCOHOL CORP. (U.S.P. 1,725,083, 20.8.29. Appl., 8.11.27).—A carbohydrate medium is fermented with a bacillus isolated from incubated malt wort. The bacillus is characterised by having the form of round-ended rods 6–20 μ in length and 2–3 μ in diam., and by its property of rapid formation of spores.

C. RANKEN.

Production of fusel oil by fermentation. F. A. McDERMOTT, ASSR. to EASTERN ALCOHOL CORP. (U.S.P. 1,725,084, 20.8.29. Appl., 8.11.27).—A sterile mash of acid molasses or other carbohydrate-protein medium is cooled to the optimum temperature and inoculated with a culture of *Willia anomala*, and added culture of regular distillery yeast. The medium is aerated and allowed to ferment.

C. RANKEN.

Production of glycerol, and of alcohol and glycerol, by fermentation. E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. [A] 316,567 and [B] 316,597, 27.4.28).—(A) During the fermentation of a sugar by yeast the addition to the mash at intervals in successive small doses is made of a sugar solution of alkaline salts, such as sodium and potassium carbonate, which are capable of promoting the formation of glycerol. The concentration of sugar in the added solution approximates to that of sugar in the mash undergoing fermentation. (B) The alkaline salts used in (A) are replaced by the incinerated ash of a fermented mash or by the potassium salts extracted from that ash. The added potassium salts should amount to 3–7% of the weight of the mash, and the total potassium salts present should be 4.5–8.5% of the weight of the mash.

C. RANKEN.

Manufacture of gluconic acid. H. T. HERRICK and O. E. MAY, ASSRS. to U.S.A. (U.S.P. 1,726,067, 27.8.29. Appl., 28.7.27).—An aqueous solution or suspension of carbohydrate material is inoculated with the spores and mycelium of one of the fungi from the group consisting of *Penicillium citrinum*, *P. divaricatum*, and *P. luteum purpurogenum*.

C. RANKEN.

Preparation of lactic salts. S. K. ROBINSON, ASSR. to K-P-C. Co. (U.S.P. 1,726,768, 3.9.29. Appl., 24.4.25).

—Fermented whey is treated with alkali to form the particular lactic salt desired, and unwanted solids are removed from solution by filtration after evaporation.

C. RANKEN.

Fermentation process. J. VAN LOON, ASSR. to NOVADEL-AGENE CORP. (U.S.P. 1,727,223, 3.9.29. Appl., 18.2.26. Ger., 25.2.25).—See F.P. 611,663; B., 1927, 589.

Production of glycerol by fermentation. F. A. McDERMOTT, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,725,363, 20.8.29. Appl., 7.11.22).—See B.P. 316,567; preceding.

Rubber substitutes (B.P. 317,814).—See XIV. **Maltose products** (U.S.P. 1,722,775).—See XVII. **Milk for infants** (U.S.P. 1,722,710). **Foodstuffs for cattle** (U.S.P. 1,725,969).—See XIX.

XIX.—FOODS.

Rapid determination of moisture in wheat. E. F. BURTON and A. PITT (Canad. J. Res., 1929, 1, 155–162).—In a specially arranged oscillatory radio circuit (cf. A., 1928, 712) the value of the current generated is influenced by the presence of foreign materials within the inductances, and the change of current can be measured by an ammeter. When wheat of varying moisture content was introduced into the electric field it was found that a linear relationship existed between the current and the moisture content of the wheat, as measured by the Brown-Duvel method, within the range of commercial interest, viz., 12–22%, and hence the ammeter could be calibrated to read moisture content.

H. J. DOWDEN.

Determination of moisture in milk powder. S. H. MEIUIZEN (Chem. Weekblad, 1929, 26, 417–421).—Treatment with carefully dried air in the Meihuizen apparatus for 3 hrs. at 100° gives accurate results. With undried air low results are obtained.

S. I. LEVY.

Fiehe's test [for commercial invert sugar in honey]. C. A. GREENLEAF and C. A. BROWNE (J. Assoc. Off. Agric. Chem., 1929, 12, 319–323).—A comparison of the official resorcinol test, the aniline acetate test, and Fiehe's test (cf. B., 1908, 1127) has been made on samples of pure honey and on honey with 5% and 10% additions of invert sugar. The last test was found to be the most sensitive but difficult of interpretation. Application of the resorcinol test to a series of natural honeys indicated that moderate heating will not cause the honey to give the colour reactions for hydroxymethylfurfuraldehyde, but increasing the acidity before heating favours the production of positive results in Fiehe's test.

H. J. DOWDEN.

Modified Fiehe test for detection of artificial invert sugar in honey. E. K. NELSON (J. Assoc. Off. Agric. Chem., 1929, 12, 323–324).—To avoid the formation of a persistent emulsion and to facilitate extraction of the hydroxymethylfurfuraldehyde, a smaller quantity of honey is advocated. A 2-g. sample of honey in 10 c.c. of water is extracted with ether for 30 min., the ether being then separated and concen-

trated to about 5 c.c. The extract is shaken with 2 c.c. of resorcinol reagent (0.2 g. of resorcinol in 20 c.c. of concentrated hydrochloric acid) and the colour noted at the end of 5 min. Pure honey gives a faint pink, honey with 10% of invert sugar a deep pink, and honey with 20% of invert sugar a dark red coloration.

H. J. DOWDEN.

Corrosion-resisting steel. STOKES.—See X. **Cacao butter.** HORN and OSOL.—See XII. **Resistance of wheat starch to diastatic activity.** MALLOCH.—See XVII.

PATENTS.

Bread-making. C. J. PATTERSON and C. G. HARREL, ASSRS. to BAKERIES SERVICE CORP. (U.S.P. 1,722,677, 30.7.29. Appl., 13.3.22).—Sufficient of a non-toxic organic acid is incorporated with a dough ingredient to bring the gluten of the flour to optimum condition during dough formation.

W. J. BOYD.

Treatment of milk and its products. O. F. HUNZIKER, ASSR. to PFAUDLER CO. (U.S.P. 1,723,860, 6.8.29. Appl., 4.10.23).—The milk is brought to a predetermined acidity and then rapidly circulated continuously and repeatedly at a high temperature and in a state of fine subdivision through a partial vacuum. Undesirable flavours and odours are removed by maintaining a current of air through it in the vacuum. The milk is cooled with continuous agitation to churning temperature.

W. J. BOYD.

Preparation of milk for infant feeding. L. A. ROGERS, ASSR. to U.S.A. (U.S.P. 1,722,710, 30.7.29. Appl., 15.3.28).—Milk is heated at 62.5° for 30 min. and homogenised at 2800 lb. pressure. After cooling to 42.5°, the milk is inoculated with a lactobacillus culture and maintained at 42° until its p_H value reaches 3.5.

C. RANKEN.

Substitute for mothers' milk. T. C. MANCHESTER (U.S.P. 1,723,188, 6.8.29. Appl., 3.7.23).—A volume of pasteurised cow's milk is divided into two portions consisting of 85% and 15% of the whole. From the larger portion cream to the amount of 10% of the original volume, 2.59% of protein, and 0.18% of ash are removed, leaving 72.23% of whey. Cream to the amount of 5.75% of the original volume and the smaller portion of the milk are added to the whey, and the mixture is concentrated to produce a product containing approx. fat 3.52, protein 1.58, lactose 6.50, ash 0.74, solids 12.34%.

W. J. BOYD.

Canning of meats. J. W. RICHARDSON and P. W. WOOLLETT (U.S.P. 1,723,710, 6.8.29. Appl., 16.11.26. Austral., 16.3.26).—Sulphur compounds are eliminated from meat prior to packing by subjecting it to the action of flowing steam at a temperature (up to 113°) sufficient to convert such compounds into a gaseous form.

B. M. VENABLES.

Preservation of fish and fish derivatives. D. A. HANSEN (B.P. 290,292, 10.5.28).—The fish are treated by brushing with or immersion in a dilute solution of sodium diborate containing a much smaller proportion of boric acid; other preservatives may be added.

Before use the product is washed with water to remove the preservatives.

W. J. BOYD.

Controlling the sulphur dioxide content of dried fruit. F. J. PROWSE, R. D. ROBINSON, E. M. HALL, and C. F. FLEMING, ASSRS. to CALIFORNIA PACKING CORP. (U.S.P. 1,724,514, 13.8.29. Appl., 6.5.25).—Sulphured dried fruit is subjected to the action of air at an elevated temperature in the presence of moisture.

E. B. HUGHES.

Sterilisation and preservation of fruit juices. L. R. CLEVELAND, ASSR. to NAT. ACAD. SCI., WASHINGTON, (U.S.P. 1,725,956, 27.8.29. Appl., 21.12.25).—The juices are clarified and subjected to the action of oxygen under pressure for a sufficient time to inhibit the growth of bacteria.

E. LEWKOWITSCH.

Treating cocoa beans to obtain food products therefrom. E. C. R. MARKS. From POSTUM CO., INC. (B.P. 317,335, 10.2.28).—The beans are heated, in presence of moisture, at 49–54° for about 20 min. and then dried *in vacuo* at about 76°. The dried beans, containing 1–2% of moisture, are then roasted at 106–114°, nibbed, and finely milled at 32–38°. Products having improved flavour and keeping qualities are claimed.

E. B. HUGHES.

Extracting vitamin substances from cottonseed. C. SCHMITT (B.P. 317,554, 25.5.28).—Nutritive substances with an increased vitamin content are obtained by adding extracts prepared from peeled cottonseed after removal of the oil by leaching (without boiling) with water or aqueous liquids, e.g., milk, wine, etc.

E. LEWKOWITSCH.

Conversion of foodstuffs for cattle. H. ROBINSON, ASSR. to PIERCY CO. (U.S.P. 1,725,969, 27.8.29. Appl., 22.6.27. Renewed 28.12.28).—Vegetable materials are converted into more readily digestible feeding-stuffs by treatment in a moist state with approx. 8% of various enzymic substances in the presence of 5% of sodium bicarbonate, 32% of sodium chloride, 36% of lime, 15% of malt, and 4% of anise.

C. RANKEN.

Sterilisation and activation of [liquid] food. J. W. D. CHESNEY, ASSR. to CHESNEY PROCESS, INC. (U.S.P. 1,723,603, 6.8.29. Appl., 17.2.28).—To the pasteurised liquid not more than 1% of an organic acid is added and the product is exposed to ultra-violet rays of not less than 2000 Å. in the absence of air.

W. J. BOYD.

Destruction of insect pests [in food products etc.]. A. M. KOBIOLE (U.S.P. 1,725,650, 20.8.29. Appl., 27.7.27. Austral., 10.8.26).—See B.P. 300,529; B., 1929, 72.

Cheese packing [in tin foil]. E. SUTTER (B.P. 317,968, 24.7.28).

[Honey-butter] food product. F. B. DEHN. From HONEY-BUTTER CO. (B.P. 318,388, 31.8.28).

Separation of cereals etc. (B.P. 316,922). **Blending or mixing** (B.P. 317,128).—See I. **Odourless containers** (U.S.P. 1,724,778). **Packing of fruits etc.** (B.P. 317,513).—See V.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Oxidation of ether: effect of certain gases. F. C. HYMAS and G. MIDDLETON (Quart. J. Pharm., 1929, 2, 278—280).—The effect of passing air, oxygen, carbon dioxide, nitrous oxide, or mixtures of these gases through ether has been investigated. In general, the amount of peroxide in the ether increased and the amount of aldehyde decreased. Oxidation is reduced by avoiding exposure to light, the accumulation of ether in the apparatus, and the use of a strip of copper in the bubbler (cf. Hewer, Lancet, 1929, 215, 770). C. C. N. VASS.

Cause of irritation of 8-hydroxyquinoline compounds. Effect of p_H caused by acid molecule. R. VAN WINKLE and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1929, 18, 794—796).—The sulphate, salicylate, β -resorcyate, tartrate, and citrate of 8-hydroxyquinoline have been examined. The antiseptic action of the compound is only slightly affected by changing the acid molecule, the sulphate, citrate, and β -resorcyate being the best and equally efficient. The sulphate and citrate have also the highest germicidal activity. No definite relation exists between irritation and the p_H variation caused by the different acid molecules; the irritation is probably a factor of the 8-hydroxyquinoline nucleus itself. E. H. SHARPLES.

Determination of 2-phenylquinoline-4-carboxylic acid in the presence of salicylic acid, acetylsalicylic acid, and hexamethylenetetramine. E. SCHULEK and G. VASTAGH (Pharm. Zentr., 1929, 70, 533—537).—The sample (1.5 g.) of phenylquinolinecarboxylic acid is dissolved in 5% sodium hydroxide solution, the mixture is boiled to hydrolyse acetylsalicylic acid, cooled, and made up to 100 c.c. An aliquot portion (20 c.c.) is diluted to 40 c.c., acidified with 50% sulphuric acid, and the liberated acids are collected. The filtrate is basified, evaporated down, and again acidified, and the acids are removed until the filtrate measures only 10 c.c. The salicylic acid is removed by washing the precipitate on a Gooch crucible with a saturated solution of phenylquinolinecarboxylic acid. When the filtrate no longer gives a positive test with ferric chloride, the contents of the crucible are dried at 120° and weighed, or, alternatively, the acid may be dissolved in alcohol and titrated with sodium hydroxide solution, using phenolphthalein as indicator. Bromothymol-blue gives results which are 1.2% too low. S. COFFEY.

Decomposition of alkaloids in aqueous solution, particularly during sterilisation: atropine, hyoscyamine, scopolamine, yohimbine, hydrastine, and hydrastinine. R. DIETZEL, F. SCHLEMMER, and R. FISCHER (Arch. Pharm., 1929, 267, 468—484).—The stabilities of aqueous solutions of the above alkaloids have been studied by means of their ultra-violet absorption spectra at various dilutions (cf. Dietzel and Huss, B., 1929, 147). The three related alkaloids, atropine, hyoscyamine, and scopolamine, afford very similar absorption spectra showing linear absorption at high concentrations (about 1%), the limit being situated at 3704 Å., but this is displaced to 4200 Å. at concentrations in the neighbourhood of 0.1%. On heating such

solutions (0.1%), a general displacement is observed towards the region of shorter wave-lengths. This appears to be due to changes in the tropic acid part of the molecule. Yohimbine solutions become coloured on heating and develop entirely new bands in the region of longer wave-lengths. Solutions of hydrastine (1%) on heating to 100° for 1 hr. develop a new absorption, 2500—2900 Å. Higher temperatures produce highly coloured solutions showing entirely different absorption spectra. Hydrastinine is far more stable in aqueous solution than hydrastine, and changes in the absorption spectra on heating are very slight. S. COFFEY.

Nicotine in tobacco. III. A. HEIDUSCHKA and F. MUTH (Pharm. Zentr., 1929, 70, 517—520. Cf. B., 1927, 570; 1928, 501).—A large number of analyses of cigar and cigarette tobacco, and of the gases produced by combustion, are given in tabular form.

S. I. LEVY.

Microchemical reactions of nicotine. M. WAGENAAR (Pharm. Weekblad, 1929, 66, 773—776).—The precipitates obtained with the ordinary reagents are described. Picric acid and cadmium iodide give precipitates with 1 mg. at a dilution of 1 in 1000.

S. I. LEVY.

Microchemical reactions for coniine. M. WAGENAAR (Pharm. Weekblad, 1929, 66, 757—760).—A crystalline precipitate may be obtained with as little as 0.1 mg. at a dilution of 1 in 5000, with phosphomolybdic acid, but the most characteristic reactions are those with potassium bismuth and potassium antimony iodides, by which 0.5—1.0 mg. may be detected at a dilution of 1 in 1000.

S. I. LEVY.

Determination of the water content of strychnine sulphate. W. SCHNELLBACH (Amer. J. Pharm., 1929, 101, 587—596).—The water content of commercial, crystallised strychnine sulphate is variable and will sometimes exceed the 11% allowed by the U.S.P. X. One specimen consisted of a mixture of the two hydrates, and preparations with water contents deviating from the theoretical values of the penta- or hexa-hydrate are considered to be mixtures of the two.

E. H. SHARPLES.

Reaction of "barbaloin" and aloes. L. ROSENTHALER (Pharm. Zentr., 1929, 70, 573).—Two drops of *o*s-phenylmethylhydrazine are added to a solution of 0.1 g. of aloes in 10 g. of ethyl alcohol and the mixture is observed periodically for 24 hrs. and then at 48 hrs. Colour reactions given by different brands of aloes are as follows: Barbadoes: first red, then greenish, blue-green, green; Curaçao: dark, greenish, blue-green, green; Zanzibar: similar to Curaçao; Natal: darkens very slowly; Cape: darker, then tinged with green, green. Phenylhydrazine gives none of the above reactions. Using a concentrated solution of "barbaloin" the reaction gives a greenish-blue powder sintering at 200° and decomposing at 240°. A faint green colour is still noticeable with 5 g. of a 1:20,000 "barbaloin" solution. E. H. SHARPLES.

Assay of hyoscyamus leaves and extract of hyoscyamus, B.P. C. M. CAINES (Quart. J. Pharm.,

1929, 2, 271—275).—A combined gravimetric and volumetric method for the assay of both the leaves and the extract of hyoscyamus, based on the recommendation for the assay of the tincture of belladonna, B.P., is described.

C. C. N. VASS.

Quillaia tincture. K. A. KARSMARK and L. KOFLER (Arch. Pharm., 1929, 267, 424—433).—Thirteen tinctures were prepared according to different official recipes and tested for dry residue, ash content, and saponin content. Tinctures prepared with concentrated alcohol contained less ash and less saponin than those prepared with dilute alcohol. The foam values for the various tinctures appeared to be roughly proportional to the saponin content. Tinctures prepared with dilute spirit are most suitable for the preparation of liquor carbonis detergens.

S. COFFEY.

Assay of jalap. L. E. WARREN (J. Assoc. Off. Agric. Chem., 1929, 12, 324—332).—Six methods of assay have been studied, viz., Jenkins' method (B., 1914, 885), Dale's method (B., 1927, 955), and the United States, Dutch, French, and German pharmacopœial methods. None of the last three methods ensures complete extraction, whilst the U.S.P. X. method and the Jenkins method are identical, each giving somewhat high results, since the fractions represented by the differences between the values obtained by these two methods and that from the Dale method were physiologically inactive. A shortened form of the Dale method is proposed, in which 10 g. of the powdered drug are heated on the water-bath for 30 min. with 50 c.c. of alcohol and then percolated with warm alcohol until 95 c.c. have been collected. After dilution to 100 c.c., 25 c.c. of the tincture are evaporated to dryness, the residue is washed 2—3 times with 15 c.c. of boiling water, the resultant resin being then dissolved in 15 c.c. of warm alcohol and filtered. The filter is thoroughly washed with warm alcohol and the combined filtrate and washings are evaporated to dryness, the residue being dried at 100° until constant in weight.

H. J. DOWDEN.

Galenical preparations of broom. Determination of sparteine and total alkaloids. J. HIRT (J. Pharm. Chim., 1929, [viii], 10, 111—124, 145—161).—The silicotungstic acid method of the French Codex for the determination of aconitine has been modified and adapted to the determination of sparteine in broom preparations. Accurate results are obtained in 3—4 hrs. The total alkaloid contents of 19 different galenical preparations of broom and the reactions of sparteine and scoparine have been determined.

E. H. SHARPLES.

Determination of [organic] arsenic compounds adapted to their study in the organism. A. BERAT (J. Pharm. Chim., 1929, [viii], 10, 49—69).—From a critical examination of the methods of destruction of organic material and the losses of arsenic involved, modifications of existing methods are suggested. With the exceptions of sodium methylarsinate and sodium and manganese cacodylates, which are only imperfectly oxidised, satisfactory results are given with organic arsenic compounds in presence of other organic matter.

E. H. SHARPLES.

Determination of formaldehyde in certain pharmaceutical preparations. O. HEIM (Analyst, 1929, 54, 537—538).—Formaldehyde may be determined (in the absence of sugars) in a substance such as a mouth-wash by treating 10 c.c. (containing about 0.2% of formaldehyde) with 2 c.c. of concentrated hydrochloric acid and 10 c.c. of *N*-silver nitrate, shaking, adding 4 c.c. of 30% sodium hydroxide solution, shaking, and leaving for 15—30 min. with occasional shakings. A black coloration forms in presence of formaldehyde, and, after filtering, the precipitate is washed with hot water, the filter perforated and rinsed with 1:3 nitric acid, the reduced silver being thus dissolved. After dilution, and removal of the residual silver chloride by filtration, the silver in the filtrate is determined as chloride in the usual way and calculated as formaldehyde ($2\text{AgCl}=\text{CH}_2\text{O}$). The method was successful when the hydrogen peroxide, iodine, and ammonia methods failed.

D. G. HEWER.

Examination of spirit of camphor, especially for the determination of camphor and alcohol. W. MEYER (Pharm. Ztg., 1929, 74, 1035—1036).—The following method is used: 50 g. of spirit are treated with 50 c.c. of water and 50 c.c. of light petroleum (b.p. range 40—60°), and the two layers are separated. The alcohol content of the lower layer is ascertained from its density, and the camphor is determined in the upper layer by careful evaporation at 65°. Petrol, ligroin, sulphuric ether, and pentane are unsuitable substitutes for the light petroleum specified above. The aqueous layer can be examined for methyl and isopropyl alcohols and acetone by the usual means.

S. COFFEY.

Anti-leprosy oils from the genus *Caloncoba* from the Cameroons. Peirier (J. Pharm. Chim., 1929, [viii], 10, 124—128).—Oils from the seeds of *Caloncoba Welwitschii* and *C. glauca* are described. The seeds have, respectively: oil content, decorticated 51.0%, 47.5%, undecorticated 33.58%, 40.24%; water 5.62%, 4.10%; proteins 19.56%, 21.17%; glucosidic material 9.43%, 9.58%; mineral matter 3.17%, 3.66%; cellulose 26.64%, 21.25%; and the oils, which are both brown and solid, have: butyric odour, none; m.p. 40°, 38—40°; solidification pt. 36.4°, —; d_{15}^{45} 0.9386, 0.928; n_D^{45} 1.4719, 1.4685; $[\alpha]_D^{25} +47.7^\circ$, $+40^\circ$; saponif. value 194.88, 187.08; ester value 189.06, 184.06; acid value 5.82, 3.02; Hehner value 95.6, 90.5; Ferrier value 17.6, 16.4; Reichert-Meissl value 2.5, 2.2; Polenske value 1.1, 0.1; unsaponif. matter 2.36, 1.80; iodine value (Hanus) 99.06, 84.3; acetyl value 10.6, 12.3. The free fatty acids have: titre 47.5, 53.8; mean mol. wt. 284, 304; iodine value 99.48, 93.21; hexabromide value nil, nil; $[\alpha]_D^{25} +50.0^\circ$, $+47.5^\circ$. A cyanogenetic glucoside is present in the oil cake from the seeds of *C. glauca*, but is absent from that of *C. Welwitschii*.

E. H. SHARPLES.

Pine-needle extract and pine-needle bath extract. C. A. ROJAHN (Pharm. Ztg., 1929, 74, 978—980).—It is suggested that bath extracts made with sulphite-liquor containing large amounts of tannins are harmful and should be classed as adulterated. Representative

samples (18) have been examined by means of the analytical quartz lamp and capillary methods, and it is suggested that these methods may be used for detecting adulterated extracts. S. COFFEY.

Pine-needle extract and pine-needle bath extract. P. BOHRISCH (Pharm. Ztg., 1929, 74, 1078—1079).—A reply to Rojahn (cf. preceding abstract). Purified sulphite-liquor is not to be considered as an adulterant in a bath extract. The methods of analysis are criticised and considered unsatisfactory. S. COFFEY.

[Pine-needle extract and pine-needle bath extract.] C. A. ROJAHN (Pharm. Ztg., 1929, 74, 1079).—A reply to Bohrisch (cf. preceding abstract). S. COFFEY.

Electrodeposition of metals and its use in pharmacy. LIVERSEDGE.—See X. Laurel fat. WALL-RABE. Pistachio oil. BEYTHIEN. Castor oil. COCKING and CREWS.—See XII.

PATENTS.

Manufacture of preparations having an action resembling that of tuberculin. I. G. FARBERIND. A.-G. (B.P. 302,226, 19.11.28. Ger., 12.12.27. Addn. to B.P. 285,087; B., 1929, 661).—Acid-resisting rod bacilli, e.g., *Mycobacter lacticola* (Rabinowitsch) or species of *Streptothrix*, are treated as described in the prior patent. L. A. COLES.

Production of highly active substances from male internal secretory organs. SOC. CHEM. IND. IN BASLE (B.P. 298,089, 1.10.28. Switz., 30.9.27. Addn. to B.P. 265,567; B., 1928, 107).—Extracts from male organs, e.g., testicles, or distillates from such extracts, are treated as described in the prior patent.

L. A. COLES.

[Manufacture of] basic phenol alkyl ethers. H. MAHL, ASSR. to WINTHROP CHEM. CO., INC. (U.S.P. 1,725,136, 20.8.29. Appl., 22.3.28. Ger., 11.4.27).—See B.P. 288,555; B., 1929, 835.

Extracting the cardio-active substance of *Bulbus scillae*. W. KREIS, ASSR. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,725,652, 20.8.29. Appl., 7.3.28. Ger., 16.3.27).—See B.P. 287,147; B., 1928, 874.

Separating impurities from oils (B.P. 308,752).—See II.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Interaction of silver halides in emulsion form. O. BLOCH (Proc. 7th Internat. Congr. Phot., 1928, 194—201).—A study of the characteristics of emulsion mixtures made by co-digestion of separately prepared silver chloride, bromide, iodide, and iodobromide emulsions demonstrates conclusively that different silver halides will react with one another after precipitation, yielding complexes having quite different photographic properties from those of their constituents. The mechanism of the reaction appears to be mainly one of adsorption, since the interaction is apparently confined to the surface. No change in grain-size distri-

bution or grain shape could be detected in any of the cases where the amount of reacting halide added was small. J. W. GLASSETT.

Silver iodide in ammonia-made emulsions. A. STEIGMANN (Proc. 7th Internat. Congr. Phot., 1928, 256—258).—The advantages derived from the presence of silver iodide in ammonia-made emulsions are most marked if quick-ripening gelatins rich in sensitisers are used. Increased sensitivity with freedom from fog is readily obtained when iodide is present, whereas a pure bromide emulsion rapidly fogs. With slow-ripening gelatins iodide is not necessary to obtain maximum sensitivity before occurrence of fog, although it appears to stabilise conditions at precipitation. Highly dispersed silver iodide has the same action as, and may be substituted in equivalent quantities for, potassium iodide in the preparation of ammonia-made emulsions, provided that more than 3% of silver iodide is used, that the mixing is slow, and that a gelatin poor in sensitiser is used. J. W. GLASSETT.

Action of mercuric salts and of mercury on the photographic plate and on silver bromide-gelatin emulsions in a liquid medium. A. REYCHLER (Bull. Soc. chim. Belg., 1929, 38, 241—247).—The latent image in a photographic plate can be destroyed by dipping the plate prior to development in a dilute solution of a mercuric salt, a minimum concentration of the latter being necessary. The same effect is obtained by placing the plate on a layer of the dry mercuric salt or on mercury, and then immersing in water before development. Treatment of the plate with a 2.5% solution of sodium sulphite instead of with water destroys the effect of the mercuric salt, development being normal. Similar results are obtained with a suspension of silver bromide in a 1% aqueous solution of gelatin. In the absence of mercuric salts addition of the developer in the dark produces immediate blackening of the bromide suspension, but in presence of small quantities of mercuric chloride the suspension remains milky. An explanation of these results is offered. O. J. WALKER.

Observations and measurements of the photo-sensitivity of silver halide sols. A. LOTTERMOSER (Proc. 7th Internat. Congr. Phot., 1928, 43—44).—Some indication of the sensitivity of silver halide sols towards light is afforded by the extent of the depression of the potential curve towards more negative values during the electrometric titration of soluble halides with silver nitrate. The greater the sensitivity the more negative is the solution; this is explained by the lowering of the silver-ion concentration owing to the formation of halide ions by the hydrolysis of the halogen liberated by the light. Contrary to other observations, the silver halide sols appear to be more sensitive in presence of a slight excess of halide ions than with an excess of silver ions. J. W. GLASSETT.

Effect of temperature on the sensitivity of photographic plates. T. T. BAKER and W. A. BALMAIN (Proc. 7th Internat. Congr. Phot., 1928, 42).—Plates of low, moderate, and extreme sensitivity show with increase in temperature during exposure a definite rise in

gamma, which reaches a limiting maximum between 77° and 100°. With the last-named type of emulsion this change is accompanied by an increase in sensitivity, as shown by greater densities in the lower exposures, although the actual H. and D. speed decreases owing to the rise in gamma. No increase in fog was obtained with any of the plates, even at 100°. J. W. GLASSETT.

"Reversal-like" phenomena of the discharge-figures impressed on photographic plates. T. TERADA (Mem. Coll. Sci. Kyoto, 1929, A 12, 217—222).—Experiments are described dealing with the reversal phenomena produced on photographic plates when the latter are illuminated by a spark simultaneously with the production of the discharge figure. The mechanism of the phenomenon is discussed. C. W. GIBBY.

Comparison of some developers for sensitometric standardisation. S. E. SHEPPARD and A. P. H. TRIVELLI (Proc. 7th Internat. Congr. Phot., 1928, 174—192).—Comparative tests were made on emulsions of widely varying characteristics with *p*-aminophenol hydrochloride, pyrogallol, and metol-quinol developers containing the same molecular equivalents of the active constituents and the same concentrations of sodium sulphite and carbonate. The *p*-aminophenol developer showed less tendency to fog, and gave a latitude for the same gamma equal to or even greater than that of the other developers. No marked differences were shown in the speed values recorded, and at equal degrees of development the covering power of the silver deposit was the same. It is concluded that the *p*-aminophenol developer, consisting of *p*-aminophenol hydrochloride 7.25 g., sodium sulphite (anhyd.) 50 g., sodium carbonate (anhyd.) 50 g., and water to 1 litre, is well suited for sensitometric work. J. W. GLASSETT.

Fine-grain developers and their application to spectrography. E. VITERBI (Proc. 7th Internat. Congr. Phot., 1928, 365—367).—The Lumière and Seyewetz physical developer (B., 1924, 579) gave negatives with a very fine grain, but contrast was lacking and there was a tendency to fog. A *p*-phenylenediamine-sulphite developer gave very similar results, but the contrast was improved by addition of borax or sodium carbonate. The most satisfactory results were obtained with a developer containing metol 2 g., sodium sulphite (anhyd.) 100 g., quinol 5 g., borax 2 g.

J. W. GLASSETT.

Chemical study of desensitisers. I. F. M. HAMER (Proc. 7th Internat. Congr. Phot., 1928, 92—104).—A general account of known desensitisers is given, together with a full bibliography.

J. W. GLASSETT.

Intensification of photographic latent image. E. P. WIGHTMAN and R. F. QUIRK (Proc. 7th Internat. Congr. Phot., 1928, 235—343).—Organic peroxides will produce intensification of the latent image in the same manner as hydrogen peroxide, and this effect is shown to be independent of the developer used. No intensification could be obtained after X-ray exposures; this is explained by the absence of latent image specks of

intermediate size, the energy of the X-ray quantum being sufficiently great to produce only specks above the size required to induce developability. Acetone-semicarbazone, a bromine acceptor, lessens the intensification by the peroxide, but at the same time produces intensification itself. It is concluded that only those substances which act on the latent image specks produce intensification, whilst those which form new specks on the silver halide produce fog.

J. W. GLASSETT.

Intensification of autochromes by dye-toning. R. J. GARNOTEL (Brit. J. Phot., 1929, 76, Colour Suppl., 23, 29—30).—The autochrome is immersed in a mordanting bath containing copper sulphate 40 g., potassium citrate 60 g., glacial acetic acid 30 g., ammonium thiocyanate 20 g., and water to make 1 litre. After washing, it is transferred to the dye bath composed of 1% aqueous dye solutions in the following proportions: methylene-blue 287 c.c., rhodamine-D 333 c.c., phosphine-M 380 c.c., with addition of glacial acetic acid 10 c.c. When a sufficient degree of intensification has been reached the autochrome is washed for 2 min. and dried. Reduction may be carried out by means of a dilute acid permanganate solution, and correction for any predominant tint is made by bathing the autochrome in a 1% solution of a suitable acid dye, *e.g.*, tartrazine extra-concentrated (for blue) and acid-green NJ, or naphthol-green NB (for red).

J. W. GLASSETT.

PATENTS.

Photographic papers. F. AHRLE (B.P. 317,173, 15.6.28).—Special sheen effects are obtained by coating the emulsion upon a support consisting of a thin layer of tin foil affixed, by means of a special size, to a paper base which has been impregnated with a waterproof varnish to prevent the penetration of the developing solutions etc. into the layer of tin foil. The metal surface is coated with a substratum of gelatin hardened with chrome alum to facilitate the application of the emulsion.

J. W. GLASSETT.

Production of photographic images. H. D. MURRAY, and NORTON & GREGORY, LTD. (B.P. 317,199, 14.7.28).—The stability of prints from diazo papers is improved by treatment, subsequent to exposure, with an inorganic reducing agent such as an alkali hypophosphite or thiosulphate, which may be applied in the solution of the coupling agent, or after development, as a separate bath or in a varnish which is brushed over the surface of the print. Further, the alkalinity of the developing solution is neutralised by an acid bath containing either oxalic, tartaric, or citric acid, or sodium metabisulphite. The preferred combined developing bath contains β -naphthol 0.6, trisodium phosphate (hydrated) 4.5, sodium hypophosphite 7, sodium thiosulphate 5, and water 100 pts. by wt.

J. W. GLASSETT.

Manufacture of silver halide photographic emulsions. J. REITSTÖTTER, Assr. to AGFA ANSCO CORP. (U.S.P. 1,725,934, 27.8.29. Appl., 16.9.25. Ger., 31.1.25).—See B.P. 246,800; B., 1926, 722.

XXII.—EXPLOSIVES; MATCHES.

Beating of nitrocellulose. JUPEAU (Mém. Poudres, 1928, 23, 183—197).—The power consumed in beating nitrocellulose makes up about 50% of the total consumption during manufacture. To control the efficiency of the beating process a quicker and more accurate method for judging the fineness was desirable. At Toulouse a device for this purpose, called a finometer, has been introduced. It consists of a lead sphere weighing 350 g. which is suspended by a thin steel wire 47 cm. long. This bob when placed in the beater in a moving stream of nitrocellulose suspended in water forms an angle with the vertical, the tangent of which is a measure of the fineness. For the Horne beater a reduced charge of 450—525 kg. according to the desired fineness increased the output from 3.5 to 4.5 to 5.6 metric tons per day. A charge containing 88.5% of water required the minimum duration of beating.

S. BINNING.

Heating *in vacuo* of nitroglycerin powders. MARQUEYROL (Mém. Poudres, 1928, 23, 178—182).—Samples of various nitroglycerin powders were heated *in vacuo* at 50° for 1834 days and determinations made at intervals of the amount of gas evolved and the loss in weight. Bo powder gave 225 c.c. of dry gases and lost 3.55% in weight. An AD₁ powder and Na powder evolved 31.7 and 170.5 c.c. and lost in weight 2.55 and 3.22%, respectively. BM powder taken as an example of a nitrocellulose powder gave under the same conditions 11.44 c.c. of gas and lost 3.53% in weight.

S. BINNING.

[Smokeless powder] stabilisers. MARQUEYROL (Mém. Poudres, 1928, 23, 158—177).—Samples of BF powder containing various percentages of diphenylamine, nitrosodiphenylamine, or carbazole were heated at 75°, 60°, and 40°. At intervals samples were taken, extracted with chloroform, and the nitrogen content of the extracted nitrocellulose was determined. In this way the denitrating effect of the stabiliser on the hot powder was determined. Diphenylamine attacks nitrocellulose at 75°, but very feebly at 60°. The velocity of denitration is initially considerable, but then diminishes. Diphenylamine appears to be converted into nitrosodiphenylamine, which is itself an excellent stabiliser. Nitrosodiphenylamine appears to combine with the nitrous compounds formed when nitrocellulose decomposes and thus prevents their autocatalytic action. Carbazole is less efficient than nitrosodiphenylamine.

S. BINNING.

Loss in weight of B powders on storing at 50°. DESMAROUX (Mém. Poudres, 1928, 23, 230—235).—Experiments over 684 days show that the loss in weight when BSP and BM powders are stored at 50° is less than that of SD (a French nitroglycerin powder). The German RPC powder was similar in behaviour to SD. Two Italian nitroglycerin powders lost considerably more than the SD or RPC powders. S. BINNING.

PATENTS.

Detonators and the like. G. A. ASHCROFT, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 317,023, 8.3.28).

—About 3% of graphite or aluminium powder is added as a lubricant to the priming charge of lead trinitroresorcinate or lead trinitroresorcinate-lead azide mixture in detonators. S. BINNING.

Percussion fuses for bombs or similar projectiles. E. GALASSINI (B.P. 301,517, 1.12.28. It., 2.12.27).

XXIII.—SANITATION; WATER PURIFICATION.

Purification of water. F. DILLER (Papier-Fabr., 1929, 27, 521—523).—For the manufacture of fine papers, colourless and crystal-clear water, free from iron and manganese, and of not too great hardness, is necessary. Three methods of treatment of moor-water in order to obtain these results are described, involving treatment with (1) aluminium sulphate, (2) lime and aluminium sulphate, and (3) sulphuric acid and aluminium sulphate. In the first method addition of excess of the reagent should be avoided. The precipitate formed settles quickly, has a decolorising action, carries down with it dissolved colloidal substances (including iron combined with organic matter), but does not remove manganese. In the second method treatment with saturated milk of lime and aluminium sulphate, followed by filtration and neutralisation of the treated water with acid, results in removal of the manganese and of the objectionable colour, but strict chemical control of the process is necessary. By the third process the p_H of the water is controlled, the time required is short, but manganese is not completely removed. The most satisfactory method from the point of view of decolorisation and removal of manganese is the second, although a relatively long time is required for the precipitation.

B. P. RIDGE.

Purification of water by base exchange. F. DIÉNERT (Chim. et Ind., 1929, 22, 249—258).—Water of zero hardness is essential to many industries and is best produced by the base-exchange process. When the mineral content of the raw water is high it is cheaper, and results in a water of lower total solid content, if this process be preceded by a lime-soda treatment, though steps must be taken to prevent incrustation of the zeolite granules. The base-exchange material must be chosen with regard to its physical properties as well as to its capacity to exchange sodium for calcium and the ease with which it can be regenerated. It is suggested that the base-exchange method might be used for the manufacture of sodium carbonate or the preparation of oxygen by decomposition of sodium hypochlorite. C. JEPSON.

Electro-osmotic purification of water and the preparation of "distilled" water by electro-osmosis. K. LLIC (Chem. Listy, 1929, 23, 408—416).—Apparatus and methods used for the electro-osmotic purification of water are described. R. TRUSZKOWSKI.

Laboratory experiments with a foaming boiler water. A. S. BEHRMAN (Ind. Eng. Chem., 1929, 21, 817—818).—A boiler water, which foamed so badly that the highest concentration of dissolved solids which could be carried was 150 grains/gal. (2550 p.p.m.), was derived from a highly coloured surface supply which had been

treated, after rough filtration, in a zeolite softener and corrected with sulphuric acid to conform with the carbonate-sulphate ratio recommended by the Amer. Soc. Mech. Eng. boiler code. Examination of the blow-down water indicated only alkalinity and coloured organic matter as likely causes. A copious and lasting foam could be produced by shaking in a stoppered bottle even when the alkalinity had been reduced by 90%. When the sample was given an acid reaction and the colour was removed by coagulation or aqua regia, the tendency to foam was prevented; this result was confirmed when the samples were boiled. On the basis of these experiments a plant has just been completed to remove colour and suspended matter by coagulation with aluminium sulphate and sulphuric acid prior to entering the boiler, but results of treatment are not yet available. C. JEPSON.

Chemical proportioning of internal water feed treatment. E. M. PARTRIDGE (Ind. Eng. Chem., 1929, 21, 819—821).—The addition of small amounts of organic matter, *e.g.*, tannin, to boiler-feed water increases the proportion of carbonate in the deposited scale with the result that the deposits are softer, less likely to build up to any considerable thickness, and more easily removed when washing out the boiler. Its action in postponing the deposit of carbonate also removes a source of trouble in injectors etc., which are likely to be blocked thereby. This treatment has proved successful and economical when applied to locomotives, the chemicals being made up into 1-lb. balls and dissolved in part of the raw water, which is by-passed through a suitable container before mixing with the remainder in the storage tank. C. JEPSON.

Mechanism of formation of calcium sulphate boiler scale. E. P. PARTRIDGE and A. H. WHITE (Ind. Eng. Chem., 1929, 21, 834—838).—Microscopical observations of the effects produced by heating a highly polished chromium plate in solutions of calcium sulphate indicate that crystals are deposited at the solid-liquid-vapour interfaces formed by the surface, solution, and bubble. If the substance so deposited has a negative solubility slope the crystals will continue to grow by contact with the supersaturated liquid film at the heating surface. If the solubility slope is positive the crystals will re-dissolve provided this tendency is not overbalanced by the rate of deposition of new crystals by the process of bubble evolution. The rate of scale formation depends largely on the degree of supersaturation of the liquid with which it is in contact, and this is probably a function of the rate of heat transfer across the boiler surfaces and of the slope of the solubility curve of the scaling substance. C. JEPSON.

Thermal effects of boiler scale. E. P. PARTRIDGE and A. H. WHITE (Ind. Eng. Chem., 1929, 21, 839—844).—Determinations of the heat conductivity of calcium sulphate scales have been made in an experimental boiler at pressures between atmospheric and 150 lb./in.², with results varying between 0.93 and 2.06 B.Th.U. per sq. ft. per hr. per ft. thickness of scale per ° F. The effect of scale on heat utilisation is small, for with scale

of average conductivity and thicknesses up to 2 mm. the loss may not exceed 3%. In modern high-pressure boilers its effect is likely to be considerable, for in the case of front-row tubes and water walls where the rate of heat input by radiation is very high the increased temperature of the metal may cause failure. It is considered that a boiler operating at a pressure of 1800 lb. would probably be endangered by a normal quality scale of 1.5 mm. in thickness or with much thinner deposits if the scale formed were porous. C. JEPSON.

Effect of nitrate oxygen on tannery effluent. E. R. THEIS and J. A. LUTZ (Ind. Eng. Chem., 1929, 21, 763—766).—Under anaerobic conditions the oxygen of dissolved nitrates is available for oxidation of organic matter, the carbonaceous material being first attacked, with liberation of carbon dioxide, and the sulphur compounds later. Most of the nitrogen is liberated in the free state, but some remains as ammonia, the proportion depending on the amount of nitrate present in relation to the amount required for complete oxidation. The amount of nitrite produced is always small and quickly suffers further reduction. C. JEPSON.

PATENTS.

Preparation of water-softening material. G. W. PRATHER (U.S.P. 1,724,679, 13.8.29. Appl., 15.7.27. Cf. U.S.P. 1,636,942; 'B., 1927, 766).—A mixture of a non-acid, soluble compound of an alkali metal and a suitable mineral is heated until the alkali compound fuses, and the resulting mass is rendered sufficiently hard to resist disintegration when used for filtering and softening water. The excess alkali is removed by washing. F. G. CLARKE.

Treatment of feed water for boilers, condensers, etc. D. W. KENT (U.S.P. 1,725,925, 27.8.29. Appl., 3.5.24).—Water is passed in succession through a series of shallow superimposed vessels in countercurrent to a stream of gas containing hydrogen and adapted to replace the oxygen dissolved in the water with a gas inert in respect to the formation of scale. The gas is diffused through the water by means of a porous medium forming the bottom of each vessel. Any oxygen removed is burned with hydrogen and, after any losses have been made good, the mixed gases are re-circulated. C. JEPSON.

Indicating the presence of saline, alkaline, acid, or other impurities in water or other fluids. W. C. CROCKATT, and W. CROCKATT & SONS, LTD. (B.P. 317,278, 12.12.28).—For automatically recording the proportion of impurities in water, an apparatus, dependent on variations in electrical conductivity, is fitted with devices which give visible or audible warning of any abnormal proportion of impurity, and by short-circuiting the recording instrument prevent possibility of damage due to excessive currents. C. JEPSON.

Centrifugal separator (U.S.P. 1,723,495).—See I.
Compositions for respirators (B.P. 317,966).—See VII.
Sterilisation of liquids (U.S.P. 1,723,603).—See XIX.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOV. 8, 1929.

L—GENERAL; PLANT; MACHINERY.

Extractor. JALADE.—See XX.

See also A., Oct., 1161, **High-temperature technique** (RUFF). 1162, **Volumenometer** (FRANCIS and OXNARD).

PATENTS.

Furnace. M. APPEL (U.S.P. 1,726,606, 3.9.29. Appl., 28.10.21).—A reversing, regenerative, reverberatory furnace is provided with adjustable deflectors adjacent to the mouths of the gas and air ports.

B. M. VENABLES.

[Rotary] kiln. M. S. PRICE (U.S.P. 1,727,217, 3.9.29. Appl., 17.3.26).—The kiln is shaped as follows, working downwards from the outlet for gases: a relatively small and short cylindrical section, a quickly expanding conical part, a long slowly converging part, a cylindrical part. The rate of travel of the material in the slow taper which comprises the major part of the kiln is slower than in the final cylindrical part.

B. M. VENABLES.

[Vertical] kiln. W. D. MOUNT and I. WARNER (U.S.P. 1,725,763, 27.8.29. Appl., 4.4.21).—The kiln is supplied with gaseous fuel at an intermediate point and with air at the bottom, the products of combustion being drawn off by suction at the top of the kiln proper, but below the feed hopper. The latter is of large capacity and discharges into the kiln by gravity without control. The finished and cooled material is withdrawn at the bottom by continuous mechanical means, the rate of withdrawal determining the rate of feed.

B. M. VENABLES.

Determining the amount of heat given off by a heating body. SIEMENS & HALSKE A.-G. (B.P. 299,880, 29.10.28. Ger., 3.11.27).—The difference in temperature between two points in a heat-conducting substance attached to the heating body is measured and integrated with respect to time. In the case of a domestic radiator, one end of a metallic rod is attached to the narrow face of the radiator at the top, the rod being insulated radially, but the free end is exposed to the atmosphere and may carry a surface having heat-emitting properties similar to those of the radiator. Both junctions of a thermocouple are embedded in the rod, the hot junction being nearer the radiator than the cold junction; alternatively either two thermocouples with a common cold junction or an electrical resistance thermometer may be used. The leads of the thermocouple are connected to an integrating device, e.g., an electrolytic counter.

B. M. VENABLES.

Drying apparatus. H. D. MILES, Assr. to BUFFALO FOUNDRY & MACHINE Co. (U.S.P. 1,726,751, 3.9.29. Appl., 18.6.25).—A drying drum is supported (and

rotated) at one end only, and the same support carries the means for supplying material to and removing it from the drum. A vacuum-tight casing enclosing the whole can then be easily slipped on from the other end.

B. M. VENABLES.

Centrifugal drying machine. G. GRAUSS (B.P. 303,120, 28.12.28. Fr., 28.12.27).—A centrifugal machine with quickly detachable basket (as described in B.P. 212,551; B., 1924, 622) is made completely airtight so that it can be used with pressures and temperatures different from those of the atmosphere. The solid matter may be collected in a filter bag which, when running, fits closely to the basket, but when stopped springs away because of whalebone insertions.

B. M. VENABLES.

Fire-extinguishing media. A. E. WHITE. From Fyr-Fyter Co. (B.P. 318,302, 5.4.28).—Aqueous solutions for spraying on to fires comprise potassium, rubidium, or caesium carbonate considerably in excess of that required to react with the acid added to liberate carbon dioxide for generating pressure, or, alternatively, the whole or a part of the excess carbonate may be replaced by other alkali salts with high fire-extinguishing properties, e.g., sodium lactate or nitrate, potassium lactate, formate, acetate, chloride, chlorate, etc.; f.p. depressants, e.g., glycol, and about 0.8–2% of a chromate to counteract the corrosive action of the carbonate, may also be added.

L. A. COLES.

Pulverisers. W. A. WHITE (B.P. 318,375, 21. and 31.8.28).—An impact pulveriser has oppositely-rotating blade-carrying elements, and both the inlet and outlet for material are nearer the axis than is the pulverising zone, the ground material being withdrawn by a current of air produced by a fan in a separate compartment from the pulveriser.

B. M. VENABLES.

Single-zone pulverising apparatus. D. K. BEACH, Assr. to RILEY STOKER CORP. (U.S.P. 1,724,895, 20.8.29. Appl., 5.5.26).—In an impact pulveriser the inlet for material (and air) is at the periphery and the outlet at the axis.

B. M. VENABLES.

Rotary hammer mills and crushers. J. Y. JOHNSON. From PENNSYLVANIA CRUSHER Co. (B.P. 317,208, 24.7.28).—In a mill of the type where the material is flung by rotating hammers against a crusher plate or plates, each plate is pivotted at one end, and the other end is mechanically agitated to free it from adhering material. The crusher plate may be subdivided and each section independently agitated.

B. M. VENABLES.

Fine-grinding mills. H. JOHANSON and G. KÖPPEL (B.P. 318,392, 4.9.28).—In a mill in which the material

(e.g., graphite for pencils) is fed to the grinding elements under pressure, the feed pump is of the screw-thread type, and a similar pump of about equal capacity is used to return the partly ground material back to the reservoir, the net amount of material delivered being regulated by controlling the delivery of the return pump. Both pumps are driven by the same shaft as is the grinding mill.

B. M. VENABLES.

High-speed mixer. R. M. GREENLEAF and O. L. ROUTH, Assrs. to PLASTOID PRODUCTS, INC. (U.S.P. 1,725,186, 20.8.29. Appl., 7.2.27).—A vertical shaft rotating at high speed carries horizontal arms from which the material is flung to the wall of the containing vessel and by which it is scraped from the wall.

B. M. VENABLES.

Separation of dry materials. K. C. APPLEYARD, C. W. H. HOLMES, I. L. BRAMWELL, and BIRTLEY IRON CO., LTD. (B.P. 317,802—4, 6.2.28).—These three patents refer to the arrangement of riffles on tables for the dry concentration of material such as coal.

B. M. VENABLES.

Production of dispersions of solids in liquids. W. H. WHATMOUGH (B.P. 317,981, 9.8.28).—A relatively coarse suspension is treated first in a high-speed mill (e.g., as described in B.P. 293,510 or 304,178; B., 1928, 658; 1929, 268) until only a small percentage of solid remains undispersed, and then in a roller mill having smooth reciprocating rollers.

B. M. VENABLES.

Centrifugal filter. C. G. HAWLEY, Assr. to CENTRIFIX CORP. (U.S.P. 1,725,712, 20.8.29. Appl., 4.5.25).—The material enters downwardly through an expanding conduit within which is a conical baffle; at the base of the cone is situated a fluid-whirling element, and below that an axial outlet and bulbous collecting pocket for heavy material.

B. M. VENABLES.

Clarifier. P. J. CHAPPELLE (U.S.P. 1,725,618, 20.8.29. Appl., 1.8.27).—The apparatus comprises a number of concentrically arranged, basket-shaped elements provided with openings and engaging with one another by flanges at the upper ends. The side and bottom walls of the baskets are covered with a fibrous filtering material which is retained in position by means of the stiff lower walls of the baskets. The outer basket is adapted to revolve.

A. R. POWELL.

Liquid sprayers. DORMAN, LONG & CO., LTD., and M. R. KIRBY (B.P. 318,061, 28.11.28).—Compressed air is led through a tapered (converging) inlet nozzle which abuts against a diverging outlet nozzle; on the abutting surface of the inlet nozzle radial grooves are formed through which the liquid is admitted from a pipe at the side.

B. M. VENABLES.

Distillation apparatus. M. KONTER (U.S.P. 1,725,528, 20.8.29. Appl., 15.4.27).—The conduit leading from the still to the condenser is bent downwards and up again. An opening is left at the apex of the V thus formed and a jar is attached in a gas-tight manner for collection of products of distillation.

B. M. VENABLES.

Bubble still. C. F. BRAUN (U.S.P. 1,725,052, 20.8.29. Appl., 7.9.26).—In a bubble still the drain pipes from successive plates are in vertical alinement, but interposed is a device for mixing downwardly-flowing liquid and

rising vapour and projecting both in a turbulent manner into the liquid on the bubble plates.

B. M. VENABLES.

[Metal rings for] fractionation apparatus. R. H. VAN SCHAAK, JUN. (U.S.P. 1,725,429, 20.8.29. Appl., 8.9.27).—Each ring is formed, as a hollow cylinder with diametral partition, from one strip of sheet metal bent somewhat like a letter S closed up.

B. M. VENABLES.

Evaporators. H. L. GUY, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (B.P. 317,772, 19.5.28).—The horizontal tubes of an evaporator are of oval cross-section, and have the long axis vertical, and are evenly spaced over the wetted cross-section of the containing vessel. To allow freer passage for rising bubbles of vapour the upper ranks of tubes may be of smaller horizontal dimensions, or some may be omitted.

B. M. VENABLES.

Vaporising apparatus. L. V. BARNABE (B.P. 318,123, 25.5.28).—The apparatus comprises a freely supported tube coil which is heated from an external source and to which the liquid to be vaporised is supplied under pressure from a reservoir above. Between the vaporising coil and the reservoir is situated an auxiliary heating coil which is connected at both ends to the reservoir and serves to maintain the pressure therein by heating the liquid.

B. M. VENABLES.

Purification of furnace and like gases. O. RADULESCU (B.P. 292,118, 2.5.28. Roum., 14.6.27. Addn. to B.P. 289,825; B., 1929, 839).—The device described in the original patent is preceded by a dry-cleaner for removal of coarser particles, comprising centrifugal discs rotating between annular baffles.

B. M. VENABLES.

Separation of dust from boiler flue gases. J. T. BARON and J. B. CLARKE (B.P. 318,082, 22.12.28).—An uptake, with a cross-section not smaller than that of the flue from the boiler, is provided with one set of steam jets above the junction with the flue, and with another set below the flue in a downward extension of the uptake which serves as a settling chamber.

B. M. VENABLES.

Refrigeration. E. B. MILLER, Assr. to SILICA GEL CORP. (U.S.P. 1,729,081, 24.9.29. Appl., 3.8.29. U.K. and Irish Free State, 25.10.24).—See B.P. 225,191; B., 1925, 483.

Mill for homogenising, emulsifying, etc. W. EPPENBACH (U.S.P. 1,728,178, 17.9.29. Appl., 24.12.27).—See B.P. 306,502; B., 1929, 497.

Preparing emulsions of liquid or dissolved substances. B. REDLICH (U.S.P. 1,729,185, 24.9.29. Appl., 18.10.27. Ger., 13.7.26).—See B.P. 302,761; B., 1929, 308.

Furnace walls [comprising water-tubes and metal blocks]. FULLER LEHIGH CO., Assees. of (A) R. SHELLENBERGER, (B) E. G. BAILEY (B.P. 292,959 and 294,876, 27.6.28. U.S., [A] 27.6.27, [B] 30.7.27).

Furnace walls [comprising water-tubes and tiles]. A. P. THURSTON. From FULLER LEHIGH CO. (B.P. 318,695, 27.6.28).

Absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING

SYSTEM AKTIEBOLAG (B.P. 301,323, 26.11.28. Swed., 25.11.27).

Evaporator units for [absorption] refrigerating apparatus. ELECTROLUX, LTD. From PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 319,050, 15.6.28 and 28.2.29).

[Automatic conveyor] apparatus for filling solid articles [tablets, pills, etc.] into receptacles. I. G. FARBENIND. A.-G. (B.P. 299,017, 18.10.28. Ger., 19.10.27).

Distributing plate for gas washers and the like. C. STILL (B.P. 318,812, 28.12.28).

II.—FUEL; GAS; TAR; MINERAL OILS.

Evaluation of gas coal. G. A. BRENDER A BRANDIS and H. J. A. DE GOEY (Het Gas, 1929, 49, 91—103; Chem. Zentr., 1929, i, 1771—1772).—"Specific values," Ω (calorific value of gas \times volume/quantity of volatile substance, computed on the dry material), are: cellulose 29.1, brown coal 34.2, bituminous coal 47.7—84.3, anthracite 129.1. Curves illustrate the relationship of Ω to volatile matter, oxygen, water and carbon dioxide, oxygen content of coal, and to the ratio of oxygen to hydrogen. A. A. ELDRIDGE.

Rate of burning of individual particles of solid fuel. H. K. GRIFFIN, J. R. ADAMS, and D. F. SMITH (Ind. Eng. Chem., 1929, 21, 808—815).—The times taken for the complete combustion of particles of fuel when dropped down a vertical furnace maintained at a definite temperature have been measured. The furnace was provided with a narrow, vertical, silica window through which the incandescent particles were photographed on a film fixed to a rotating drum. The time of burning for particles of a gas coal (45—50-mesh) increased from 0.258 sec. at a furnace temperature of 720° to 0.578 sec. at 1000°. Similar figures were obtained for a semi-coke. Particles of activated charcoal required a considerably longer period to burn than particles of coal of the same size, but the rate of burning of the former showed much less variation with the temperature. Results obtained with a beehive coke were somewhat indefinite, due probably to difficulty of ignition, and to selective burning of the smaller particles. The time of burning for all the fuels decreased with the particle size. Possible explanations of the negative temperature coefficient of the rate of burning of coal and semi-coke particles are discussed.

A. B. MANNING.

Fire and explosion risk with acetylene. W. RIMARSKI (Z. angew. Chem., 1929, 42, 933—936).—An enquiry into the cause of the recent disaster at Borsigwalde, and into the conditions under which explosive decomposition may be initiated and propagated in acetylene. The findings of earlier workers on the effects of high temperature and pressure, and the adequacy of the normal safeguards, are confirmed.

S. I. LEVY.

Determination of carbon dioxide in gases containing acetylene. H. FRIEDRICH (Chem.-Ztg., 1929, 53, 706—708).—Acetylene and carbon dioxide are appreciably soluble in caustic soda and fuming sulphuric

acid, respectively, so that the determination of either by absorption in these reagents is inaccurate. In an improved method the carbon dioxide is measured by removal with soda-lime, and then both acetylene and carbon dioxide are absorbed by treatment with fuming acid followed by caustic soda. The final traces of acetylene are removed by a solution of mercury cyanide containing a little alkali. R. H. GRIFFITH.

Designing flash-distillation equipment for petroleum refining. R. S. PIROOMOV and N. E. LOOMIS (Chem. Met. Eng., 1929, 36, 472—475).—The term "flash distillation" is applied to any operation in which a mixture of hydrocarbons, after being heated above its initial b.p., is discharged into a zone where separation of vapours from residual liquid can occur. Distillation takes place under conditions of substantial equilibrium between vapour and liquid in the separating zone. The equilibrium single-flash curve of a stock starts at a higher and ends at a lower temperature than either the true b.p. or the Saybolt 10% distillation curves, crossing the last two somewhere in the lower-middle range. It is possible to establish a simple relationship for the slopes and points of intersection of flash curves with the slopes and mean b.p. of the corresponding true b.p. or Saybolt 10% distillation curves. This relation holds only for the distillation curve at 760 mm. If the true b.p. distillation of a stock is available, the point of intersection and the slope of the flash curve may be read off, and the straight line drawn to represent it will approximate very closely to the actual equilibrium curve at atmospheric pressure. Experimental results indicate that the slope of the flash curve and the percentage off at its intersection with the true b.p. curve are practically independent of pressure. Consequently, to estimate the flash curve of a stock at any other pressure than 760 mm., the temperature at the intersection of the two curves at 760 mm. must be extrapolated to the desired pressure by using the Cox chart (B., 1923, 699 A) for the vapour pressure of hydrocarbons. Examples are given showing how steam consumption, and the temperatures at the top and bottom of the bubble tower etc., can be obtained from these curves. The bearing of these principles on the proper design of the overhead separating equipment and of the pipe-still itself is indicated. W. S. E. CLARKE.

Recent development in the art of cracking [of petroleum hydrocarbons] in the vapour phase. C. R. WAGNER (J. Inst. Petroleum Tech., 1929, 15, 484—492).—A review of the scope and development of vapour-phase cracking, with especial reference to the Gyro process in which charging stock is pumped through heat economisers to an evaporator, and the vapours pass through a separator to remove entrained liquid and are mixed with superheated steam prior to passing through parallel convertor tubes fitted with catalytic cores. The plant is equipped with modern fractionating and gas-recovery plant. Carbon formation is reduced to a minimum and the non-condensable gas production, although double that made by liquid-phase processes, is produced at the expense of the fuel oil fraction and not by reduction of the yield of motor fuel. The distillate contains 45—50% of olefines and 40—45% of

aromatics, the balance being naphthenes; paraffins are almost wholly absent. Methods have been evolved for removing colour and odour from the motor spirit, whilst the gum-forming tendency is reduced by special treatment. By blending equal volumes of straight-run gasoline and vapour-phase cracked motor fuel, there is produced a motor fuel having an anti-knock rating of 40–50% of benzol. The finished motor fuel is richer in low-boiling fractions than ordinary gasoline, its sp. gr. is greater by about 0.03 than that for a straight-run spirit of the same distillation range, whilst its f.p. is considerably below -73° . The fixed gas is a valuable material for synthetic chemical manufacture, its composition varying little except in hydrogen sulphide content, which is dependent on the sulphur in the charging stock, the greater part being liberated in this way.

H. S. GARLICK.

Flame characteristics of "pinking" and "non-pinking" fuels. II. G. B. MAXWELL and R. V. WHEELER (J. Inst. Petroleum Tech., 1929, 15, 408–415).—Former research (B., 1928, 511) has been continued with slightly modified apparatus. Increase in temperature of pentane-air mixtures slightly widened the range in which pinking occurred, and with benzene-air mixtures a narrow range giving faintly audible explosions was found. Previous observations, that the addition of benzene, the combustion of which is continuous throughout the explosion, to pentane reduces the likelihood of a pinking explosion by reducing the amount of chemical energy available to maintain a shock-wave when the initial wave is arrested, were confirmed. Addition of "pro-knock," e.g., ethyl ether and amyl nitrite, reduces the amount of afterglow in the wake of the flame and gives a correspondingly more intense glow at the moment of pinking. When carbon dioxide and steam were added to pentane-air mixtures it was found that, with the former, the range of mixtures giving pinking explosions was narrowed, and, with the latter, the spread of the flame and its luminosity were progressively decreased with increase in amount of water vapour added, the audibility of the pinking was greatly reduced, and the afterglow nearly eliminated. With carbon disulphide-air mixtures combustion was continuous and long-continued behind the flame front. No mixture of hydrogen and air was found which gave a pinking explosion similar to that with pentane-air mixtures. Two conditions were established under which a pinking explosion may occur in a closed vessel: (1) the shape and size of the vessel, the nature and strength of the mixture, and the magnitude and rate of heat liberation in the burning gases must be such that a stationary wave could be set up in the column of gases before the initial flame has travelled throughout the vessel; (2) the nature of the fuel and its concentration must be such that there is sufficient residual energy available to maintain a shock-wave when the flame, accelerated by the stationary wave, is arrested by the wall of the vessel. Anti-knocks render the combustion continuous behind the flame fronts. Turbulence tends to prevent the formation of a stationary wave and accelerates the combustion in the wake of the flame.

H. S. GARLICK.

Measurement of detonation in internal-combus-

tion engines. R. O. KING and H. MOSS (Engineering, 1929, 128, 219–221, 272–274).—The tendency of a fuel to detonate is usually determined by one of the following methods: (1) The running conditions of any available engine are set to produce continuous detonation, the amount of the latter occurring in a given time being measured. The relative anti-knock values of fuels are thereby obtained by their measured amounts of detonation under carefully standardised conditions; it is usual to determine the benzol or dope addition required to make an inferior fuel equal in performance to some fuel, provisionally adopted as a standard. (2) Some factor in engine running such as ignition timing is varied by being set at a value to avoid detonation with the fuel under test, and is then gradually altered until continuous detonation is just audible; the ignition timing to produce detonation is determined for the standard fuel, and the relative anti-knock values of inferior fuels may then be obtained in terms of the dope or benzol additions required to bring about the same ignition timing, with audible detonation, as the standard fuel. (3) The relative fuel values are obtained in terms of the highest useful compression ratio (H.U.C.R.), which is made a variable and is the controlling factor used to produce detonation; it is obtained by running trials with the Ricardo variable-compression engine; the anti-knock value of a fuel can then be expressed in terms of the compression ratio with which detonation is just appreciable. The two fuels selected for examination by methods (1) and (3) described above were a straight-run California spirit, highly resistant to detonation, and a straight-run naphtha possessing less resistance to detonation than any fuels in general use. An addition of 8 c.c. of "ethyl fluid" per gal. was required to make the naphtha equal to the undoped California spirit, but if 2 c.c. of ethyl fluid per gal. were added to the spirit the naphtha required 17 c.c. per gal. for equality. The H.U.C.R. approached a maximum value for the larger additions of dope, but it would be impossible to use enough dope to enable the engine to run at full power at the ideal compression ratio of 8:1, even if the dope were added to superior fuels. The rate at which metallic dope can be supplied to an engine is at present limited by considerations of cleanliness, and 8 c.c. per gal. is the maximum that can be used. On this basis a poor fuel could be used at a compression ratio of 5.5 instead of 4.5, but a good fuel such as the California spirit could be used at nearly 7 instead of 5.5.

C. B. MARSON.

Measurement of detonation in internal-combustion engines. F. B. THOLE (Engineering, 1929, 128, 386).—The use of the bouncing-pin test for detonation, which is described, is particularly suitable with a special Armstrong engine on account of the cylinder design, but the method cannot be applied indiscriminately to any engine. The accuracy of measurement is always greater than that attainable by the audibility test, but the two give results in good agreement. The experiments of King and Moss² (preceding abstract) are reviewed, and suggestions are made as to an improved procedure for testing blended and doped fuels, which involves standardisation with chemically pure liquids of known values.

R. H. GRIFFITH.

Determination of paraffin hydrocarbons in motor fuels by sulphonation. G. KUHN (Chem.-Ztg., 1929, 53, 701—702).—The methods of Heilingötter (B., 1928, 512) and of Pritzker and Jungkunz (B., 1923, 483 A) for the determination of benzene in benzene-benzene mixtures are considered unsatisfactory. The author's method is to use 1 pt. of fuel to 2 pts. of acid, as in Pritzker and Jungkunz's method, and fuming sulphuric acid containing 4.8% SO_3 (free). The sulphonation is carried out in a 150-c.c. flask fused to a burette 60 cm. long and reading to $\frac{1}{10}$ c.c. The opening of the burette can be closed by means of a ground glass stopper. The initial substances consisted of pure benzene (d^{15} 0.8833) and normal benzene (d^{15} 0.7005), and in each experiment 10 c.c. of benzene-benzene and 20 c.c. of fuming sulphuric acid were used. Results so obtained for benzene-benzene mixtures containing up to 30% of benzene are greater than those given by the Pritzker-Jungkunz method. The differences between the two increase when the benzene content is above 30%, rise to a maximum at 50% of benzene, and then gradually decrease with greater amounts. It is possible to ascertain the benzene content from the sulphonation residues of fuels of any composition with an accuracy of about 2%; greater accuracy is obtained by using increased quantities of reagents.

W. S. E. CLARKE.

Determination of aromatic hydrocarbons in gasolines produced by straight distillation. M. D. TILITSCHEEV and A. I. DUMSKAYA [DUMSKI] (J. Inst. Petroleum Tech., 1929, 15, 465—483).—The aniline and nitrobenzene critical solution temperature as well as the densitometric and refractometric methods were critically examined, and attempts made to determine the variation of the experimental constant, k , in the equation $x = kd$, where x is the percentage of aromatic hydrocarbons by wt., and d is the difference between the gasoline constants before and after separation of the aromatics. Artificial mixtures of benzene, toluene, xylene, and cymene were made with pure paraffin and naphthene hydrocarbons, and with petroleum fractions, and the basic constants, i.e., the critical solution temperatures in aniline and nitrobenzene, the sp. gr., and the refractive index, were determined both for the petroleum fractions (or hydrocarbons) freed from aromatics and for their respective artificial mixtures with aromatic hydrocarbons. For different solutions containing the same aromatic hydrocarbon in the same concentration, k is a function of the constants of the non-aromatic portion of the fraction. Values of k are plotted against the values of one or other of the constants of the non-aromatic portion of the fraction (e.g., the aniline point) to obtain a curve showing the functional dependence of k . Similar curves were obtained in connexion with other methods. It was found, in general, that the simplest and quickest method is the refractometric, in which very small quantities of each fraction suffice, and the results are accurate to $\pm 0.3\%$. Where greater accuracy is required, the densitometric method is little inferior to the others for fractions boiling below 150° , but above this temperature the aniline and nitrobenzene methods are preferable, since these are less affected by the varying composition

of the aromatic hydrocarbons in this range. The nitrobenzene method possesses the advantages of lower critical solution temperature, and hence more rapid determination, smaller fluctuations of nitrobenzene coefficients due to variation of chemical composition of the non-aromatic portion, and greater stability of the nitrobenzene.

H. S. GARLICK.

Determination of unsaturated hydrocarbons in benzene with bromine. J. ZDÁRSKÝ (Chem. Obzor, 1929, 3, 165—168, 205—210; Chem. Zentr., 1929, i, 1772).—Bromine values differ according to the method employed for their determination. Even when the conditions are constant, only relative values can be obtained.

A. A. ELDRIDGE.

See also A., Oct., 1168, **Destructive distillation of lignin from corn cobs** (PHILLIPS). **Lignin colouring matters** (PODBREZNIK).

PATENTS.

Apparatus for carbonising coal and the like. F. C. GREENE and I. F. LAUCKS, Assrs. to OLD BEN COAL CORP. (U.S.P. 1,723,932, 6.8.29. Appl., 24.10.25).—Preheated coal is discharged into the bottom of a vertical screw-retort comprising a tubular casing, which is suspended from its upper end and contains a screw-conveyor, the width of each thread of which is one third of the space between adjacent threads. The casing tapers slightly downwards, and is free to expand into an enlargement at the bottom, provided with a steam outlet.

F. G. CLARKE.

Treatment of coking coal and the like with smoke and dust from reduction furnaces. P. L. J. MIGUET (B.P. 292,931, 25.6.28. Fr., 25.6.27).—The fine dust from furnaces of the reduction type, other than blast furnaces, which is collected, e.g., by electrostatic precipitation, is utilised by its incorporation in the coke used for the reduction, by mixing it with the fines of the coking coal before compressing and placing the latter in the coke oven.

A. B. MANNING.

Hydrogenating and treating carbonaceous materials. M. HOFSSÄSS (U.S.P. 1,711,499, 7.5.29. Appl., 30.11.26. Ger., 2.12.25).—Carbonaceous material is hydrogenated under pressure in presence of anthracene or a homologue above the decomposition temperature of hydrogenated anthracene. Anthracene etc. may be recovered by fractional distillation of a part of the product.

R. BRIGHTMAN.

Recovery [from hydrogenated coal products] of organic substances which are volatilisable at an elevated temperature and apparatus therefor. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 317,506, 30.4.28).—The mixture of oleaginous liquors and solid matter obtained by destructive hydrogenation of coal etc. is fed in a thin stream on to a revolving vertical cylinder or cone down the surface of which it flows. The cone is heated by burner gases either from the outside or through a chimney within the cone, the gases passing in the latter case down the inside of the cone and up over the outer surface. The volatile products are carried off by the heating gases, and the solid residue is removed by suitably placed scrapers.

C. HOLLINS.

Activation of carbon. J. C. MORRELL (U.S.P. 1,713,347 and 1,712,930, 14.5.29. Appl., [A] 19.5.23, [B] 4.3.26).—(A) Carbon is treated with an aqueous solution of a halogen (*e.g.*, chlorine), (B) in presence of a neutralising agent, *e.g.*, a metallic carbonate or hydrogen carbonate, selective for hydrochloric acid and not hypochlorous acid. R. BRIGHTMAN.

Carburetted water-gas apparatus. R. M. SEARLE (U.S.P. 1,726,317, 27.8.29. Appl., 23.7.24).—The combined carburetter and dust catcher comprises a vertical cylindrical shell having a spray device depending from the centre of the roof. The water-gas enters by a tangential inlet, and the carburetted gas passes to a superheater through a vertical outlet pipe, the mouth of which is immediately below the spray.

F. G. CLARKE.

Manufacture of water-gas. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,016, 17.9.28. Addn. to B.P. 214,544; B., 1924, 549).—The process described in the main patent is modified by the provision of a heat accumulator above the fuel bed in the producer. At the end of the blow period steam is passed downwards through the heat accumulator and fuel bed in succession. The steam may be preheated by passing through the hollow bars of the grate and through a heat accumulator in the dust separator before entering the producer. A good water-gas can thus be obtained from a brown-coal semi-coke with relatively low temperatures in the fuel bed. A. B. MANNING.

Suction gas generator for distilling and gasifying inferior fuel and its operation. F. A. G. DANIEL and J. VAN VLAARDINGEN (B.P. 318,013, 14.9.28).—The distilling shaft of the generator and the feeding hopper are coned, becoming wider in the direction of their bases. The hopper has no valve and projects some distance into the interior of the shaft, leaving a small annular space for the gases to pass. A cylindrical wall is mounted outside the conical wall of the shaft, the intervening space containing water over which the entering air supply is drawn. The air and steam supplies are so adjusted that the highest temperature 25–30 cm. above the grate of the generator varies from 300° to 400°. A. B. MANNING.

Apparatus for producing synthetic hydrocarbons and alcohols [from natural gas]. H. A. WEBSTER, Assr. to E. T. SCHULER (U.S.P. 1,711,913, 7.5.29. Appl., 1.10.26).—Natural gas, *e.g.*, methane, mixed with steam is injected at 400 lb./in.² into a retort initially at 1260°, and the mixture of carbon monoxide and "nascent" hydrogen is passed at 325–475° through a catalysis chamber containing, *e.g.*, iron calcined with potash. The resultant mixture of alcohol and hydrocarbon vapours passes through a relief valve to cooling coil and receiver. Alternate use of a similar retort in parallel with the first and connected with the same catalyst chamber gives a continuous process. R. BRIGHTMAN.

Effecting chemical reactions in [hydrocarbon] gases by means of electrical discharges. P. H. HULL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 317,558, 31.5.28).—Hydrocarbon gases are fed turbulently (at 800–900 ft./sec.) into the zone of electrical

discharge of a high-tension arc, and, after treatment, are led rapidly away from that zone to a surrounding chamber, whence they re-enter the zone of electrical discharge and pass through a hollow electrode.

J. S. G. THOMAS.

Effecting chemical reactions in [hydrocarbon] gases by means of electrical discharges. P. H. HULL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 317,920, 31.5.28).—Acetylene is produced (*a*) by treating hydrocarbon gases with high-tension electric arcs while passing between the opposing rims of two co-axial tubes, the gas passing in through one tube and out through the other; or (*b*) by creating an electric discharge between a hollow conducting tube, having its mouth shaped convergently in the direction of gas flow, and an opposing rod electrode, and causing the gas to enter the discharge and travel along the hollow tube at high velocity, thereby sucking the electric discharge into the hollow tube. The electrodes are made of or tipped with carbon. H. S. GARLICK.

Oxidation of hydrocarbons. G. EGLOFF and J. C. MORRELL, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,710,155, 23.4.29. Appl., 20.7.22).—Hydrocarbon vapours are mixed with oxidising gases under pressure and subjected to a high-potential electrical discharge in presence of a catalyst. R. BRIGHTMAN.

Distillation of tar. BARRETT Co., Assees. of S. P. MILLER (B.P. 289,832, 21.4.28. U.S., 3.5.27).—The hot gases from part of a coke-oven battery are subjected to regulated cooling, and are then cleaned by electrical precipitation before they are passed to the condensing system. The tar which is separated in the electrical precipitator, either alone or together with the tar which separates in the collector main, is distilled by being brought into intimate contact with the hot gases from other coke ovens (*cf.* B.P. 282,367; B., 1929, 507). The enriched gases from the latter are also cleaned by electrical precipitation before passing to the condensing system. The gases from both treatments are combined for the subsequent recovery of ammonia therefrom.

A. B. MANNING.

Distillation of [tar] oil. BARRETT Co., Assees. of S. P. MILLER (B.P. 287,900, 19.3.28. U.S., 29.3.27; *cf.* B., 1929, 633).—Tar oils are brought into intimate contact with hot coke-oven gases, *e.g.*, by spraying them into the uptake pipes and collector main, in sufficient quantity to cool and scrub the gases and supply an excess of undistilled oil to dissolve the separated tar constituents and flush them from the main. The vapours, free from pitch, pass to a suitable condensing system. The temperature of the gases in the collector main may be regulated in part by the addition of a spray of water or ammonia liquor.

A. B. MANNING.

Production of lubricating oils and phenols from coal tar. A. WEINDEL, Assr. to ZECHE M. STINNES (U.S.P. 1,726,638, 3.9.29. Appl., 12.12.24. Ger., 27.12.23).—Primary tar is distilled to about 240° and benzene is added to the residue in order to precipitate asphaltic substances. After separation of these the benzene is distilled off and the lubricating oil material remaining is taken up in a solvent which is also miscible

with water; by then adding water to the mixture the oil separates out and the phenols remain in solution.

A. B. MANNING.

Apparatus for distilling mineral oil. A. E. PEW, JUN., and H. THOMAS, ASSRS. to SUN OIL Co. (U.S.P. 1,714,812, 28.5.29. Appl., 29.5.26).—The oil is vaporised by rapidly flowing in a thin stream in heat exchange with a mercury-vapour condensing chamber. Vapours pass out to a condenser and receivers, the unvaporised oil passing on to successive similar vaporising units in which the mercury vapour is maintained at progressively higher temperatures and pressures. R. BRIGHTMAN.

Apparatus for distilling [cracking] oil. A. SCHWARZ (U.S.P. 1,713,149, 14.5.29. Appl., 6.11.25).—The oil is heated in a still with a movable cover carrying hollow heating tubes which dip into the oil and through which hot gases are passed out of direct contact with the oil. Carbon from the cracking process deposits on the heating elements and is periodically removed. The still may be provided with a sleeve to promote circulation of the oil. Vapours from the still pass to a condenser or receiver, or a number of such stills may be connected in series, reflux condensate from the first stills being heated and cracked in successive units.

R. BRIGHTMAN.

Apparatus for cracking [petroleum] oil. A. SCHWARZ, ASSR. to COAL & OIL PRODUCTS CORP. (U.S.P. 1,714,454, 21.5.29. Appl., 20.5.25).—Petroleum oil, *e.g.*, pressure-still tar, is sprayed into the top of a gas producer on to the relatively cool bed, *e.g.*, at 430°, while superheated steam at 315° and a small amount of air are admitted to the incandescent bed. Waste-heat gases escape from the top of the producer and oil vapours *etc.* pass out to a settling box and fractionating tower, vapours from which are condensed. Condensate from the upper part of the fractionating tower may be returned to the base of the tower or again injected with fresh oil. An alternative process, in which the waste gases from the producer are used to heat oil pumped to the injector is described. R. BRIGHTMAN.

Prevention of substantial corrosion in hydrocarbon oil-treating [cracking] apparatus. J. C. MORRELL and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,715,095, 28.5.29. Appl., 3.9.25).—Ammonia solution is injected into the pipe conducting vapours from the dephlegmator to the cooling coil.

R. BRIGHTMAN.

Cooling the heating coils of an oil cracking apparatus. G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,725,067, 20.8.29. Appl., 14.1.25).—In the type of cracking apparatus in which oil is continuously supplied to a heating coil, where it is raised to cracking temperature, and thence delivered to a chamber, provision is made for cooling the coil by discontinuing the application of heat, ceasing the delivery of oil from the coil to the chamber, and cyclically circulating cool oil from a bulk supply.

H. S. GARLICK.

Oil cracking. M. J. TRUMBLE (U.S.P. 1,724,982, 20.8.29. Appl., 10.12.25).—Successive portions of a revolving member pass through a heat-receiving zone, where direct flame contact occurs, and then through

an oil-receiving and vapour-evolving zone where oil is delivered on to the heated portion and the evolved vapours are conducted therefrom. H. S. GARLICK.

Cracking of oil. J. C. BLACK (U.S.P. 1,715,980, 4.6.29. Appl., 20.10.26).—Petroleum oil, *e.g.*, fuel oil, is heated at 425° in a primary coil and maintained at 425° for 20–25 min. in a secondary cracking coil under, *e.g.*, 500 lb./in.², whence the oil passes after heat exchange with incoming oil to atmospheric pressure at 290° to an evaporator and bubble tower. Gasoline and other light petroleum vapours after scrubbing with fresh oil are condensed, whilst the phlegms pass to a cooler and receiver as gas oil, or may be injected after passing through the heat exchangers into fresh oil in the primary or secondary heating coil. Alternatively, the oil passing from the primary to the secondary coils may be cooled to 25–50° by the injection of cold oil.

R. BRIGHTMAN.

Converting heavy combustible oils into light combustible oils. P. L. HAHN (B.P. 289,482, 27.4.28. Ger., 30.4.27. Addn. to B.P. 235,625; B., 1925, 661).—The preliminary vaporisation of the oil to be treated is effected by passing it through a bath of molten metal provided with a circulating conduit which is heated to reheat the molten metal. Further, the vapours are mixed with superheated steam only, or the constituents thereof, at 350–600° to effect the necessary chemical changes.

H. S. GARLICK.

Conversion [cracking] of petroleum oils. W. M. CROSS, ASSR. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,717,007, 11.6.29. Appl., 14.10.22).—The hydrocarbon oil is raised to cracking temperature in heating coils under high pressure and passed at 370–510° and under 400–900 lb./in.² for mid-continent gas oil into an insulated conversion chamber, into which air or other combustion-supporting gas is injected to agitate the oil and maintain a temperature above that attained in the heating coils. The converted oil is drawn off and cooled under pressure and the carbon separated by settling.

R. BRIGHTMAN.

Distillation [cracking] of [heavy petroleum] oil. A. E. MILLER, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,714,097, 21.5.29. Appl., 11.6.27).—Heavy petroleum oil is topped, *e.g.*, by heating in successive shell-stills with gravity flow so that vapours leave the dephlegmators at 205° and 230°, and the topped oil is pumped to the bottom of a vertical still beneath a relatively deep bed of fuller's earth at 300–340°. The earth is supported by screens to prevent contact with the hot still bottom, and vents are provided to avoid gas or vapour pockets beneath the screen. Vapours from the still pass to a reflux tower and are condensed, whilst the reflux is returned for re-use.

R. BRIGHTMAN.

Treatment [cracking] of mineral oils. H. BLUMENBERG, JUN. (U.S.P. 1,713,252, 14.5.29. Appl., 24.10.27).—Dehydrated mineral oil is treated with 2–10% of dry hypochlorite (*e.g.*, of aluminium) and cracked at 110–345° for 1–2 hrs. Hypochlorite of iron or copper is preferred with oils of high sulphur content.

R. BRIGHTMAN.

Pyrogenetic cracking process [for mineral oils]. R. W. HANNA, ASSR. to STANDARD OIL Co. OF CALIFORNIA

(U.S.P. 1,724,476, 13.8.29. Appl., 15.10.27).—Vapour evolved by heating the oil under pressure is separated from the liquid, and vapour and oil, subjected to separate pressures, re-combine in such manner that the temperature of the oil is raised to the temperature of decomposition. The oil is then passed into a reaction zone. J. S. G. THOMAS.

Converting heavy mineral oils into lower-boiling products. W. G. LEAMON (B.P. 317,868, 14.3.28).—Mineral oil to be cracked passes through a pipe still where it is raised to a temperature below that of cracking but sufficient to vaporise a substantial portion, leaving the high-boiling portions, including the tarry constituents. The vapours are discharged into a dephlegmator, and the separated vapours are rapidly superheated, to avoid extensive cracking, to 530–550° while travelling at high velocity in a stream of relatively small diameter. The superheated vapours are delivered to a heat-insulated cracking chamber containing a mass of inert, refractory, porous, absorptive contact material, and maintained there sufficiently long for cracking to be effected wholly by the continued superheat, the cracked vapours then passing to a condenser. The fixed gases from the condenser pass to a flow-meter and to means sensitive to differential gas pressure for automatically regulating either the supply of fuel to the furnace or the rate at which the oil to be cracked is fed into the system. H. S. GARLICK.

Manufacture of low-boiling and gaseous hydrocarbons from those of higher b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,270, 27.4.28).—Olefines, diolefines, and butadienes are obtained from tars, mineral and vegetable oils, bituminous coals, etc., which, maintained in a vapour or finely-divided state, are subjected to high-temperature electrical discharges at temperatures not exceeding 700–800°.

C. B. MARSON.

Thermal decomposition of hydrocarbons. ELECTRO METALLURGICAL CO., Assees. of S. M. NORWOOD (B.P. 298,556, 19.6.28. U.S., 11.10.27).—Hydrocarbons are passed at cracking temperatures through a tubular member consisting of an alloy containing 15–40% Cr, 2–15% Ni, 0.7–3.0% Si, 0.7–3.0% Mn, and not more than 1.0% C, the balance being principally iron.

H. S. GARLICK.

Catalytic cracking of hydrocarbons. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,722,042, 23.7.29. Appl., 7.1.21. Renewed 12.5.27).—Oil is passed continuously through a cracking zone of high temperature and superatmospheric pressure into an expansion chamber where vaporisation occurs and the pressure is reduced though still maintained above atmospheric. The vapours are led through a catalytic bed consisting of a relatively thin layer of a metallic oxide supported in the vapour space of the expansion chamber before passing to a condenser for collection as pressure distillate.

H. S. GARLICK.

Treatment [cracking] of hydrocarbons. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,722,043, 23.7.29. Appl., 14.11.21. Renewed 14.9.28).—Portions of unvaporised oil are continuously withdrawn from a supply of oil subjected to cracking conditions of

temperature and pressure, diluted with an oil of lower sp. gr., passed through a bed of filtering material, and the cleansed oil is injected into the lower portion of the bulk supply of oil being cracked. H. S. GARLICK.

Cracking of hydrocarbons. E. W. ISOM, Assr. to SINGLAIR REFINING CO. (U.S.P. 1,711,351, 30.4.29. Appl., 21.7.27).—Hydrocarbon oils are cracked, by passing the oil vapours from the heating conduit (540–650°) successively through digesting drums (510–650°), scrubbing tower (260–290°), reflux tower (180–230°), and fractionating tower (150–190°), vapours from the fractionating tower passing to the usual condenser, receiver, and gas separator, direct or after treatment with absorbent earth or clay. R. BRIGHTMAN.

Conversion [cracking] of hydrocarbons. W. M. COLONY, Assr. to PETROLEUM CONVERSION CORP. (U.S.P. 1,715,341, 28.5.29. Appl., 3.1.27).—Hydrocarbon oil vapours are passed into a conversion chamber at 390–425° fitted with chequer work, together with steam and hydrocarbon gases etc. from a regenerative hot-blast stove at, e.g., 815–980°, the temperature of the hot gases being regulated by previous admixture with cold gas, e.g., stripped gas from the final receiver. Vapours from the converter, after heat exchange with feed oil, are condensed, the uncondensed vapours being then stripped in an absorber and the gas finally recycled through the blast stove, a part being used to cool the hot gas feed to the converter. R. BRIGHTMAN.

Conversion of hydrocarbons. L. J. WALSH, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,725,434, 20.8.29. Appl., 28.11.23).—Hydrocarbon material leaving the heating coils enters a reaction drum where tarry material is allowed to separate during conversion and is withdrawn from near the bottom. Vapours are withdrawn from near the top of the drum, and both streams are combined and conveyed to a region of reduced pressure. H. S. GARLICK.

Treatment [cracking] of hydrocarbon compounds. A. BALMER. From PETROLEUM CONVERSION CORP. (B.P. 317,508, 11.5.28).—The process of B.P. 207,276 (B., 1924, 84) for the treatment of petroleum oils so as to produce motor fuels of lower mol. wt. and b.p., is modified by the following improvements: (1) A stream of heat-carrier gas of uniform temperature is produced by mixing hot gas from regenerative furnaces with a regulated supply of relatively cold gas. (2) The oil is vaporised and the vapour mixed with the hot carrier gas to bring about the conversion reaction. (3) The heat in the products of the reaction chamber is used to raise the temperature of the carrier gases prior to their admission to the heating element employed and to raise the temperature of the incoming oil. (4) The products of the reaction are scrubbed with a high-boiling oil to remove carbon before passing to the rectifying and condensing columns. (5) Advantage of the scrubbing step is taken to top the oil used as scrubbing medium, and the vapours are added to the products of the process. H. S. GARLICK.

Preparation of hydrocarbon products. F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,727,303, 3.9.29. Appl., 25.4.24).—A fraction having a relatively high b.p. is condensed out of vaporious

hydrocarbons, which have been obtained by absorption, and is cooled out of contact with the uncondensed gases. The latter are compressed and rectified so as to obtain a fraction having a relatively low b.p. The two fractions are then mixed.

F. G. CLARKE.

Cracking of hydrocarbons and hydrocarbon oils. E. C. HERTHEL and H. L. PELZER, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,714,090—1, 21.5.29. Appl., 11.6.27).—Petroleum oil, *e.g.*, gas oil, is circulated at 125–300 lb./in.² through a heating coil to and from a main supply still. A gaseous heating medium (at, *e.g.*, 425–450°) is forced into the still beneath a bed of absorbent filtering material, *e.g.*, fuller's earth or pulverised coke, supported on perforated plates. In (A) vapours from the still ascend to a reflux tower, condensate from which is returned to circulate direct to the heating tubes, and thence to condenser and receiver. Uncondensed gases discharged from the latter may be used as heating medium. Raw oil may be fed in either at the top of the reflux tower with fresh absorbent material to replace that lost in the periodic discharge of pitch-laden oil or tar, or to the circulating pump in excess of that required to cool the bearings. In (B) the vapours ascend through a reflux tower in countercurrent with crude-oil feed containing a little fine fuller's earth in suspension, and thence to condenser and receiver. Phlegms from the tower with stripped feed oil are passed direct to the circulating pipe to the heating tubes. R. BRIGHTMAN.

Cracking of [hydrocarbon] oils. E. W. ISOM, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,722,222—3, 23.7.29. Appl., [A] 1.4.27, [B] 21.5.27. Renewed 31.12.28).—A quantity of oil is maintained in a reservoir at cracking temperature by circulating a portion of the oil through heating tubes, where it is raised to a cracking temperature, and back to the main body of the oil. A supply of fresh oil is passed through a reflux condenser in indirect heat exchange with the vapours generated in the cracking operation, and is then introduced into the oil in the reservoir. The uncondensed vapours from the first refluxing operation are then submitted to further refluxing in direct contact with fresh oil, and the condensate from both refluxing operations together with unvaporised fresh oil from the second refluxing operation is returned to the heating conduit, while a portion of the unvaporised residual oil is withdrawn from the reservoir.

H. S. GARLICK.

Conversion of hydrocarbon oil. G. EGLOFF, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,707,349, 2.4.29. Appl., 25.6.23).—The crude oil passes from the heating coil to the inner container of the expansion chamber, whence it ultimately overflows into the outer chamber. Residuum may be drawn off periodically from the expansion chambers, while the vapours pass to a dephlegmator, to which crude oil may be supplied as reflux medium, and is condensed. The phlegms are returned to the outer shell of the expansion chamber.

R. BRIGHTMAN.

Cracking of hydrocarbon oils. G. W. GRAY, ASSR. to TEXAS Co. (U.S.P. 1,724,760, 13.8.29. Appl., 28.10.24).—Oil is cracked in a pressure zone, and the high-boiling fractions of the evolved vapours are condensed from the low-boiling fractions, and, while still hot, subjected to

a higher cracking temperature and pressure. At the same time an independent quantity of oil is distilled and the evolved vapours are fractionated, the condensate thus formed being admixed with the oil in one of the pressure zones.

H. S. GARLICK.

Treatment of [hydrocarbon] oils. G. D. WHITE, ASSR. to TEXAS Co. (U.S.P. 1,726,280, 27.8.29. Appl., 15.3.20).—Connected with a still are a mixing chamber for aluminium chloride and oil, and a pair of recovery stills adapted to receive sludge from the main still and return aluminium chloride vapours thereto.

H. S. GARLICK.

Heat treatment of hydrocarbon oils. R. C. OSTERSTROM and C. R. WAGNER (B.P. 317,507, 9.5.28).—Topped hydrocarbon oil or gas oil is forced through a pipe still in which it is raised to 315°, and in passing through the outlet line under a pressure of 15–25 lb./in.² is mixed with superheated steam before being discharged into an evaporator where the sudden decrease in pressure causes complete vaporisation of the lighter constituents. The condensed oil is removed from the system, while the vapours, maintained at 315–427°, pass through a separator, to trap any unvaporised or condensed particles, to the converter, which comprises a modified pipe still in which the upper coils are exposed to intense radiant heat developed within the combustion chamber. Conversion temperatures are above 537°, the vapours in the pipe line being raised from 315–427° to 537° in the shortest possible time. The converter elements are provided with cores of catalytic material, *e.g.*, ferrous or ferric oxide, of cylindrical form, and tapered to provide means for regulating the rate of flow of the vapours. The vapours leave the converter and pass to a cooler, where they immediately meet and intimately mix with a fine spray of liquid make-up stock or other cooling medium, so that almost instantaneous cooling from 537° to 315° occurs. Uncondensed vapours are led to a fractionating tower and condenser for final separation, whilst the oil from the bottom of the cooler is circulated by a pump back into the line leading to the pipe still.

H. S. GARLICK.

Pressure distillation of heavy hydrocarbon oils. R. E. WILSON, ASSR. to STANDARD OIL Co. (U.S.P. 1,712,187, 7.5.29. Appl., 29.6.25).—The oil, *e.g.*, distillate of initial b.p. 260–288°, is brought to 370–400° under 4–6 atm. in a bulk-supply drum by circulation through heating tubes and headers. Vapours escape to a reflux tower and are condensed, the reflux being returned to the drum and crude oil fed either direct to the heating tubes or to the top of the reflux tower. Unvaporised oil containing about 0.1% of asphaltene is withdrawn continuously from the drum and heated under 200–600 lb./in.² to 420–460° in a coil whence the oil passes to conversion chambers in series. Vapours pass to a second reflux tower, condensate from which is returned to the heating coil while the vapours pass to the usual condensers and receiver. R. BRIGHTMAN.

Manufacture of lubricating oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,311, 5.6.28).—Highly viscous lubricating oils, containing only traces of hydrocarbons of low b.p., are prepared by treating mineral and tar oils of b.p. range 200–300° with

anhydrous aluminium chloride and finely-divided metals and/or metalloids at 100–200°. In examples, zinc dust, a mixture of copper bronze and powdered nickel (1 : 1), powdered brass, zinc dust and selenium, and powdered tellurium are used. C. B. MARSON.

Vacuum distillation of hydrocarbons. J. C. BLACK (U.S.P. 1,717,002, 11.6.29. Appl., 2.2.24).—The hydrocarbon oil, *e.g.*, lubricating oil stock, is circulated thermally or by steam ejection or pumping through heating coils to and from an evaporator under a vacuum of at least 24 in. Dry steam is injected, and the vapours pass into a separating chamber and baffle tower, and thence to condenser and receiver. Paraffin condensate from the baffle tower in part returns to the evaporator, and in part flows through a cooler to a separate receiver, whence a part is returned to the top of the baffle tower to promote the condensation of heavy distillates, light distillates and steam passing into the condenser and receiver. Paraffin or lubricating distillate is thereby condensed in the tower practically free from water.

R. BRIGHTMAN.

Apparatus for treating emulsified oil. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,722,041, 23.7.29. Appl., 1.9.20. Renewed 28.5.28).—Oil is supplied to the upper section of a still composed of vertical parallel sections, in which it is heated by external horizontally-placed burners, positioned and independently controlled so that heat is supplied to the surface of the oil continuously as the bulk of the oil in the still decreases and the surface lowers. H. S. GARLICK.

Treatment of emulsion oils. E. H. PAYNE and S. A. MONTGOMERY, Assrs. to STANDARD OIL Co. (U.S.P. 1,726,309—10, 27.8.29. Appl., 1.3.22).—(A) Spent caustic soda which has been used for washing hydrocarbon oils or refinery gases is mixed with the emulsions. On continued heating, stratification takes place. (B) A small quantity of a spent alkaline solution containing materials extracted from sour oils is used for the same purpose.

F. G. CLARKE.

Treatment of [petroleum-water] emulsions. Reclamation of oil. C. FISCHER, JUN., and W. T. REDDISH, Assrs. to KONTOL Co. (U.S.P. 1,727,164—5, 3.9.29. Appl., [A] 7.9.26, [B] 30.10.26).—(A) The emulsions are heated with a homogeneous reagent containing the sodium salts of mineral-oil sludge sulphonic acids, alcohol, and a bituminous substance having m.p. 35–95°; the resulting oil and water are separated. (B) The reagent used consists of an aqueous solution of bitumen and an oil-soluble mineral-oil sulphonate.

F. G. CLARKE.

Emulsion product. L. KIRSCHBRAUN (U.S.P. 1,725,648, 20.8.29. Appl., 15.4.27).—Bitumen pitch particles are dispersed in water containing not more than 1.5% of alcohol and small quantities of a deflocculating electrolyte, these quantities being insufficient to prevent freezing of the dispersion at temperatures below 0°, but sufficient to prevent injury of the emulsion at such temperatures.

H. S. GARLICK.

Revivifying spent [mineral oil] clarifying and decolorising agents. M. L. CHAPPELL, Assr. to CONTACT FILTRATION Co. (U.S.P. 1,715,535, 4.6.29. Appl.,

24.3.26).—The material, *e.g.*, acid-treated montmorillonite, Ventura or Utah clays, is agitated with a combined colour and oil solvent (*e.g.*, a mixture of acetone, ethyl alcohol, and gasoline) containing less than 3% of sulphuric acid, and then with a solvent free from sulphuric acid. The revived agent is filtered, pressed, and dried. The washings and vapours passing to still and condensers are heated for recovery and recirculation of the solvent and separation of the petroleum oil, colouring matter, and acid. R. BRIGHTMAN.

Obtaining high-boiling organic acids from their mixtures with high-boiling oils. STANDARD OIL DEVELOPMENT Co., and S. P. COLEMAN (B.P. 294,892, 30.7.28. U.S., 30.7.27).—The mixture of naphthenic acids and oil, *e.g.*, Gulf Coast crude petroleum heavy fractions, is washed with hot sodium hydroxide solution to remove the acids as sodium salts. Sulphuric acid is added to the latter and the separated oily acids are again converted into sodium salts, which are dried and fluxed with 2 vols. of gas oil, b.p. 250–350°. The mixture is then heated rapidly to 510° and flashed into a vessel maintained under vacuum (1.5 in. of mercury). The oil-free residue of naphthenates is run into water and naphthenic acids are recovered. C. HOLLINS.

Separation of solids from oil. J. C. DEACON, Assr. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,713,117, 14.5.29. Appl., 10.12.24).—The dirty oil after treatment, *e.g.*, with potassium alum or lime to precipitate negatively-charged (or sodium hydroxide for positively-charged) particles flows through a horizontal pipe which discharges into a vertical pipe placed axially in the settling tank. Gas is deflected upwardly and is removed from the upper part of the tank, while the liquids and solids pass down the pipe and are prevented by deflectors from clinging to the inner surface of the pipe in descending or ascending against the outer surface. The heavier solids deposit and are periodically withdrawn, the lighter particles forming a scum on the water surface through which the oil is filtered in passing upwards. Separation of the oil and water is facilitated by heating coils in the lower part of the settling tank.

R. BRIGHTMAN.

Obtaining solid paraffin [from oils]. J. F. P. SCHÖNFELD, Assr. to N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (U.S.P. 1,724,731, 13.8.29. Appl., 7.8.25. Holl., 4.7.25).—Mineral, animal, or vegetable oils and fats, or mixtures thereof, are atomised in a liquid state into a cooling gaseous atmosphere free from water and of such a temperature that solidification of part of the liquid of the individual droplets takes place, enabling subsequent separation of solid from liquid to be effected.

H. S. GARLICK.

Dewaxing of oil. E. B. PHILLIPS and J. G. STAFFORD, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,714,133, 21.5.29. Appl., 28.11.24).—The oil is treated in an insulated tank, provided with cooling coil and agitator, with fuller's earth or other diatomaceous earth, chilled, and filtered through a press. The press cake is washed with cold naphtha, the naphtha washing added to the dewaxed oil, and the wax melted with hot water and separated from water and earth, the earth being dried and re-cycled.

R. BRIGHTMAN.

Dewaxing lubricants by double centrifuging. G. F. OLSEN (U.S.P. 1,715,670, 4.6.29. Appl., 15.10.27).—Lubricating oil is diluted with naphtha, *e.g.*, 1.5 vols. of b.p. 95–205°, and chilled to about –3° at a rate not exceeding 2° drop per hr. The separated wax is centrifuged and discharged; the oil solution is further cooled to about –30° at 5–8° per hr. and centrifuged, the separated wax and adherent oil being returned to the first cooling tank and mixed with incoming oil and naphtha, whilst the dewaxed solution is discharged and the naphtha recovered. R. BRIGHTMAN.

Treatment of cracked hydrocarbons [containing sulphur]. G. EGLOFF and J. C. MORRELL, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,725,068, 20.8.29. Appl., 3.7.26. Renewed 22.1.29).—Cracked oil containing sulphur is agitated in the presence of copper sulphate in sulphuric acid solution. H. S. GARLICK.

Dehydration of moist fuels and liquid heavy hydrocarbon oils. I. G. FARBERIND. A.-G. (B.P. 317,771, 16.4.28. Addn. to B.P. 274,858; B., 1928, 804).—The process of the main patent is modified by operating at temperatures of 100–200° and under correspondingly lower pressures (3–6 atm.). The elimination of the water may be accelerated by the addition of salts, aniline, or brown-coal tar oils etc. A. B. MANNING.

Refining of hydrocarbon oil. E. H. PAYNE, ASSR. to STANDARD OIL CO. (U.S.P. 1,716,974, 11.6.29. Appl., 17.6.25).—The oil is sprayed on to a bed of fuller's earth or other filtering material in a vertical chamber having a constricted, *e.g.*, inverted conical, lower portion containing a supporting screen. Filtered oil is withdrawn below the screen and when the filtering material becomes exhausted the latter is washed with naphtha. The naphtha is removed by injecting steam both direct and in tubes extending through the chamber, and the residual organic matter is then burnt out by heating the constricted portion of the chamber and admitting air. The combustion zone travels upwards through the filter and is controlled by supplying cold air through the tubes; the filter bed is finally cooled by washing with water and used again. R. BRIGHTMAN.

Refining of hydrocarbon oils. H. K. IHRIG, ASSR. to ASSOCIATED OIL CO. (U.S.P. 1,712,619, 14.5.29. Appl., 15.6.27).—Hydrocarbon oil is vaporised and contacted with a heated mixture of approx. 75% of crude iron oxide and 25% of hydrated lime. R. BRIGHTMAN.

Refining of mineral oils. A. LACHMAN, ASSR. to RICHFIELD OIL CO. OF CALIFORNIA (U.S.P. 1,712,960, 14.5.29. Appl., 18.5.26).—Crude mineral oil is treated with sulphuric acid, *d* 1.84, at, *e.g.*, 45–50°, decanted from sludge, and heated with phosphoric acid (85%) at, *e.g.*, 170–185° in the absence of air. The oil is again decanted, washed with dilute sodium hydroxide or neutralised by agitation with lime at 120°, and filtered. The oil may also be washed with alkali after the sulphuric acid treatment. R. BRIGHTMAN.

Refining of mineral lubricating oils. E. D. GRAY, E. L. SCOFIELD, and E. C. DEFOE, ASSRS. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,716,632, 11.6.29. Appl., 9.11.25).—Mineral oil is treated with sulphuric acid and

after settling from sludge is filtered upwardly through a bed of sawdust from which oil-soluble resins have been removed; the filtered oil is neutralised.

R. BRIGHTMAN.

Removal of acid reaction products from [petroleum] oil [distillates]. J. C. BLACK, W. D. RIAL, and J. R. MCCONNELL, ASSRS. to PAN AMER. PETROLEUM CO. (U.S.P. 1,725,611–2, 20.8.29. Appl., 13.10.26).—The distillate is treated with sulphuric acid, the acid sludge is removed, and to the separated oil is added (A) a pitch, or (B) a coal-tar pitch, to precipitate the sludge remaining in the oil. H. S. GARLICK.

Treatment of crude petroleum oil. D. E. WOODS and E. J. NEEL, ASSRS. to OIL-O-TREAT CO. (U.S.P. 1,715,217, 28.5.29. Appl., 9.3.28).—Crude oil, *e.g.*, Texas crude oil, is treated with 0.025% by vol. of a mixture of 68% of gas oil, 10% of sulphuric acid, 14% of fatty oil, *e.g.*, red oil, and 8% of crude phenol. R. BRIGHTMAN.

Oil-reclaiming process. C. VAN BRUNT, ASSR. to GEN. ELECTRIC CO. (U.S.P. 1,713,200, 14.5.29. Appl., 1.4.25).—Spent lubricating oil is clarified by adding 0.5% of oxidised; sludged mineral oil, *e.g.*, sludge from transformer oil, prior to addition of sodium silicate and sedimentation with water. R. BRIGHTMAN.

Subdividing solid and semi-solid substances [e.g., waxes or resins]. H. GROSSMAN (U.S.P. 1,711,482, 30.4.29. Appl., 26.9.24).—The materials are melted, emulsified hot in an aqueous liquid containing an emulsifying agent (*e.g.*, sodium oleate), and precipitated in fine particles by dilution with cold water, with or without the addition of hydrochloric acid. R. BRIGHTMAN.

Washing apparatus for separating coal and like materials. C. MARCHANT (U.S.P. 1,729,545, 24.9.29. Appl., 7.12.26. Belg., 16.1.26).—See B.P. 264,460; B., 1927, 625.

Carbonising a cellulose-containing substance such as wood, peat, etc. C. G. SCHWALBE (U.S.P. 1,728,807, 17.9.29. Appl., 24.6.24. Ger., 25.6.23).—See B.P. 218,294; B., 1925, 949.

Vertical retort for use in distillation of shale and like materials. R. H. CROZIER (U.S.P. 1,729,418, 24.9.29. Appl., 7.7.25. U.K., 7.10.24).—See B.P. 245,499; B., 1926, 228.

Continuous distillation of hydrocarbons with simultaneous cracking of high-boiling into low-boiling hydrocarbons. H. WOLF, ASSR. to CARBUROL A.-G. (U.S.P. 1,711,869, 7.5.29. Appl., 24.9.25. Ger., 3.11.24).—See B.P. 252,308; B., 1926, 972.

Centrifugal apparatus for purifying [lubricating] oil [from internal-combustion engines]. G. KELM (B.P. 303,412, 29.11.28. Ger., 2.1.28).

Fine-grinding mills (B.P. 318,392). Separation of dry materials (B.P. 317,802–4).—See I. Purification of alcohols (U.S.P. 1,712,475). Porous charcoal (U.S.P. 1,716,606).—See III. Purification of detergent solutions (U.S.P. 1,711,829).—See VI. Lead tetra-alkyl (U.S.P. 1,717,961).—See VII. Paving composition (U.S.P. 1,727,231).—See IX. Castor oil (U.S.P. 1,715,785).—See XII.

III.—ORGANIC INTERMEDIATES.

Tests for methyl alcohol. H. LEFFMANN and C. C. PINES (Amer. J. Pharm., 1929, 101, 584—586).—In the German Pharmacopœia (D.A.B.) VI. test for formaldehyde produced by oxidation of methyl alcohol in ethyl alcohol, potassium guaiacolsulphonate is more satisfactory than guaiacol. The test will detect 1 pt. of formaldehyde in 6000 pts., and also is a check on the fuchsin-sulphurous acid test, serving especially to distinguish the glycerol from the methyl alcohol reaction, as apparently preliminary distillation does not ensure the absence of glycerol from the distillate. The U.S.P. process for oxidation is accurate, delicate, and convenient, and the tedious addition of powdered permanganate as directed by the D.A.B. VI. is unnecessary.

E. H. SHARPLES.

Electrochemical oxidation of anthracene to anthraquinone with a new type of electrode. C. H. RASCH [with A. LOWY] (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 7 pp.).—Whereas with ordinary lead anodes a suspension of anthracene in 2% sulphuric acid is inappreciably oxidised by electrolysis at 80°, and even with addition of ceric sulphate as catalyst the anodic current efficiency is only 8.7%, with the new type of electrode this efficiency rose to over 26% under otherwise similar conditions. This new type of electrode is prepared by making a paste of a finely-ground mixture (1:3) of anthracene and lead peroxide with dilute sulphuric acid and inserting the paste into the openings of the grid plate of a lead accumulator, after the grid had been superficially oxidised with nitric acid. This grid is used as the anode in the electrolytic cell containing dilute sulphuric acid. By using a suspension of anthracene in dilute sulphuric acid with addition of ceric sulphate and employing the new type of anode, a current efficiency of over 40% was obtained.

H. J. T. ELLINGHAM.

See also A., Oct., 1155, **Preparation of azoimide and its salts** (HOTH and PYL). 1168, **Preparation of diamines and amino-alcohols** (PUTOCHIN). 1171, **Sulphonation of phenanthrene** (FIESER). 1172, **Derivatives of 1:1'-dinaphthyl** (CORBELLINI and DEBENEDETTI). **Derivatives of 2:5-dibromonitrobenzene** (RAIFORD and BREN). **Mixed azo derivatives** (FAVREL).

PATENTS.

Manufacture of unsaturated hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 317,500, 14.4.23).—As catalysts for the dehydration of alcohols and glycols to unsaturated hydrocarbons there are used such salts of the acids of phosphorus as have an acid reaction in aqueous solution, excluding aluminium and trisodium phosphates; 2—4% of phosphoric acid may be added with advantage. [Stat. ref.] C. HOLLINS.

Manufacture of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 317,303 and 317,359, 13.2.23).—The deposition of carbon in the pyrogenic production of butadiene etc. is avoided (A) by constructing such parts of the apparatus of chromium, vanadium, manganese, or of iron alloys containing substantial amounts of these metals or nickel, cobalt, molybdenum, or tungsten, particularly Krupp's V2A steel; or an

iron apparatus is lined with chromium, copper, aluminium, graphite, or a noble metal; or (B) by covering the inner surfaces of those parts of the apparatus which are kept at 500—600° with adherent melts or sinters of inorganic compounds, *e.g.*, silicates, phosphates, or borates of alkali or alkaline-earth metals, magnesium, aluminium, or beryllium, with or without addition of salts of chromium, manganese, or copper.

C. HOLLINS.

Manufacture of divinyl and homologues thereof.

I. G. FARBENIND. A.-G. (B.P. 291,748, 7.6.28. Ger., 7.6.27).—Pure divinyl is obtained in 80% yield by passing γ -butylene glycol vapour, preferably with steam, at 240—250° over alum which has been dehydrated at 150—180°.

C. HOLLINS.

Polymerisation of dienes. T. MIDGLEY, JUN., C. A. HOCHWALT, and C. A. THOMAS, ASSRS. to GEN. MOTORS CORP. (U.S.P. 1,713,236, 14.5.29. Appl., 25.8.26).—Dienes are treated with a metal catalyst, *e.g.*, sodium-potassium alloy, which is liquid under the conditions of polymerisation.

R. BRIGHTMAN.

Continuous production of alkylene oxides from alkylene chlorohydrins. T. GOLDSCHMIDT A.-G. (B.P. 293,763, 10.7.28. Ger., 11.7.27).—Alkali and ethylene chlorohydrin solution flow past baffles in a heated still to an overflow and thence to a heat exchanger to warm the inflowing chlorohydrin. The ethylene oxide passes off through a reflux, which returns condensed water etc. to the still, and thence to coolers which separate residual moisture and finally ethylene oxide. The escaping vapours are washed with the chlorohydrin solution entering the system. The operation, excepting the condensation of ethylene oxide, is preferably conducted under decreased pressure.

C. HOLLINS.

Iodine solution and its manufacture. W. A. STEPHENS (U.S.P. 1,719,523, 2.7.29. Appl., 31.5.18. Renewed 20.5.26).—A solvent is distilled in the presence of excess of iodine and, *e.g.*, 10% of glycerol is added to the distillate. Thus a mixture of 25% of iodine and 75% of alcohol gives an alcoholic solution of ethylene iodide.

R. BRIGHTMAN.

Manufacture of etherified polyhydric alcohols and derivatives thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 317,770, 19.3.28).—A polyhydric alcohol (glycerol, erythritol, pentaerythritol, sorbitol, pentoses, hexoses) is heated in a closed vessel with an alkylene oxide or cycloalkylene oxide, or with a chlorohydrin of one of these in presence of alkali. Interaction is facilitated by addition of boric or sulphuric acid. Sorbitol and ethylene oxide at 140° give sorbitol hexa-(β -hydroxyethyl) ether; pentaerythritol yields a similar viscous ether. These may be used in place of glycerol for making films from gelatin etc. The di- γ -hydroxypropyl ether of γ -butylene glycol is a viscous liquid which can be distilled.

C. HOLLINS.

Purification of alcohols. H. E. BUC, ASSR. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,712,475, 7.5.29. Appl., 31.7.23).—The alcohol is agitated with a highly refined petroleum, initial b.p. 299—304°, stratified, separated, and the process repeated 6—8 times, the alcohol being finally distilled.

R. BRIGHTMAN.

Separation of polymerides from crude alcohol mixtures. S. R. MERLEY, Assr. to DOHERTY RES. CO. (U.S.P. 1,713,346, 14.5.29. Appl., 11.8.26).—Paraffin hydrocarbon oil (initial b.p. above 200°, Saybolt viscosity 35—300/40°, and free from sulphur or nitrogen compounds) is added to the diluted alkyl sulphate or alcohol-acid mixtures, and the mixture of hydrocarbons and polymerides is decanted before distillation. Heavy alcohols may be recovered from this layer by washing.

R. BRIGHTMAN.

[Preparation of] alkali alcoholates [alkoxides]. L. P. KYRIDES, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,712,830, 14.5.29. Appl., 20.10.23).—Anhydrous alkali alkoxides containing at least two carbon atoms are obtained by distilling the alcoholic solution of the alkali hydroxide in presence of a liquid which forms an azeotropic mixture with water or with water and the alcohol of lower b.p. than that of any of the single components or of their binary mixtures. *E.g.*, a solution of 21.7 kg. of sodium hydroxide in 350 litres of alcohol is fractionally distilled with 100 litres of benzene until 50 litres of the ternary mixture, b.p. 64.5—65°, are collected. Benzene (350 litres) is added gradually and the binary mixture, b.p. 68—68.5°, is distilled off till the temperature reaches 78.3°, leaving 125 litres of an anhydrous alcoholic solution of sodium ethoxide. Potassium butoxide is similarly obtained with toluene, or by distilling the binary mixture of butyl alcohol and water, b.p. 92—93°.

R. BRIGHTMAN.

Xanthate mixture. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,716,273, 4.6.29. Appl., 16.7.26).—Mixed xanthates from alcohols of higher mol. wt. than amyl alcohol, obtained, *e.g.*, by catalytic vapour-phase oxidation at 170—500° of a mixture of kerosene and gas oil, extracting with aqueous methyl alcohol, hydrolysing the extract with dry sodium hydroxide, and, after washing out the soaps, agitating with sodium hydroxide and carbon disulphide, are claimed as flotation agents, insecticides, or fungicides, or as accelerators for vulcanisation of rubber.

R. BRIGHTMAN.

Manufacture of organic compounds from oxides of carbon and hydrogen. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 317,808, 18.5.28).—For gas mixtures at high pressures in which there is more hydrogen or methane than carbon oxides, a catalyst containing copper, silver, or gold with up to an equal proportion of iron, cobalt, or nickel, and up to 5% of zinc, magnesium, aluminium, chromium, molybdenum, tungsten, uranium, or manganese, is used; the addition of 1% of an alkali-metal compound is advantageous. When more than 25% of carbon oxides is present, the catalyst must be free from cadmium and thallium. The products are mainly oxygenated compounds, especially alcohols, acids, and esters. Examples are: ferric nitrate (1 pt.), cupric nitrate (2 pts.), and potassium carbonate (0.1 pt.), deposited on pumice and calcined, yield a catalyst suitable for a gas mixture containing carbon monoxide (32%), hydrogen (64%), and nitrogen (4%) at 200 atm. and 350°; mixed nitrates of iron (12 pts.), zinc (2 pts.), and copper (12 pts.), precipitated with potassium carbonate, give a catalyst which at 240° and 300 atm. converts a mixture of carbon monoxide

(20%), methane (8%), hydrogen (70%), and nitrogen (2%) largely into ethyl alcohol.

C. HOLLINS.

Effluent gas treatment. F. A. CANON, Assr. to SELDEN CO. (U.S.P. 1,716,028, 4.6.29. Appl., 15.2.29).—Effluent gases from vapour-phase organic oxidation processes are scrubbed by passing over limestone or other insoluble carbonates in countercurrent with a thin stream of water.

R. BRIGHTMAN.

Manufacture of chloroacetaldehyde. I. G. FARBERIND. A.-G. (B.P. 299,319 and Addn. B.P. 299,722, [A] 22.10.28, [B] 29.10.28. Ger., [A] 22.10.27, [B] 28.10.27).—(A) Vinyl chloride and chlorine are separately introduced into water at 35° in a packed tower in the absence of a catalyst and preferably with exclusion of light. $\alpha\beta$ -Trichloroethane is formed as a by-product. (B) The chloroacetaldehyde may be distilled off from the liquor obtained by the process described in (A), if the concentration of hydrogen chloride therein is first raised to 20%.

C. HOLLINS.

Manufacture of chloroacetic anhydride. C. J. STROSACKER and C. C. SCHWEGLER, Assrs. to DOW CHEM. CO. (U.S.P. 1,713,104, 14.5.29. Appl., 15.3.27).—Acid chlorides, *e.g.*, chloroacetyl chloride, are heated with carboxylic acids, *e.g.*, chloroacetic acids, in presence of a metallic chloride (about 1% of aluminium chloride) to give chloroacetic anhydride, m.p. 46°.

R. BRIGHTMAN.

Hydrogenation of crotonaldehyde. H. C. HOLDEN, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,724,761, 13.8.29. Appl., 8.1.23).—Crotonaldehyde is hydrogenated and water added to the reaction product before distillation.

H. S. GARLICK.

Furfuraldehyde reaction product [porous charcoal]. W. O. SNELLING (U.S.P. 1,716,606, 11.6.29. Appl., 25.5.26).—Porous, non-resinous products, *e.g.*, for porous vessels, pigments, absorbents, are obtained by treating 35—90 pts. of furfuraldehyde with 3—30 pts. of hydrochloric acid and 5—50 pts. of water at ordinary temperatures for 3—30 days, and washing out the soluble products.

R. BRIGHTMAN.

Production of oxygenated organic compounds [acetic acid from methyl alcohol and carbon monoxide]. BRIT. CELANESE, LTD., and H. F. OXLEY (B.P. 317,867, 22.2.28).—In the process of B.P. 283,989 (B., 1928, 223) copper or a copper compound (copper phosphate) is added as a promoter to the phosphoric acid catalyst.

C. HOLLINS.

Effecting gaseous dehydrating reactions. T. EWAN, H. M. ROBERTS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 316,898, 5. and 21.3.28).—Carbon monoxide is added to the gas to be dehydrated and under suitable conditions water is removed by reaction with the monoxide. In some cases the removal of water by carbon monoxide may advantageously be achieved after the main reaction by means of a second catalyst at a different temperature. Acetic acid vapour is passed with carbon monoxide over sodium metaphosphate at 500—600° and then over a manganese oxide catalyst at 350°; acetic anhydride and acetic acid result, with only traces of water. The process is applicable to other reversible dehydrations.

C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. H. DREYFUS (B.P. 317,907, 26.5.28).—Acetic acid vapour is passed with 2–7% of ammonia through a copper or silica tube at 550–600°, and the vapours are fractionally condensed. Organic bases or the hydrochlorides, phosphates, or sulphates of these or of ammonia may be used. C. HOLLINS.

Manufacture of alkoxy-aliphatic [acid] anhydrides. II. DREYFUS (B.P. 317,342, 14.4.28).—Alkoxy-aliphatic acids, especially methoxyacetic acid, are converted into anhydrides by passing the vapours with or without diluent gases, preferably with carbon oxides, through a copper, silica, or fire-clay tube at 500–800°, which may contain a dehydrating catalyst on a carrier. Water is absorbed from the resulting mixture by passing over calcium chloride at 250–800°. C. HOLLINS.

Manufacture of ethylene glycol. E. C. BRITTON, Assr. to DOW CHEM. CO. (U.S.P. 1,709,605, 16.4.29. Appl., 21.5.23).—Ethylene chlorobromide is heated below 160°, *e.g.*, at 145° for 4 hrs., with aqueous sodium carbonate. R. BRIGHTMAN.

Recovery of disubstituted guanidines. A. E. PARMELEE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,716,081, 4.6.29. Appl., 9.1.24).—The reaction mixture obtained by desulphurising a thiocarbamide, after removal of any excess of ammonia and alcohol, is acidified, *e.g.*, with hydrochloric acid at 60–85°, and then treated with sodium carbonate or other alkali until faintly alkaline to brilliant-yellow test paper, to precipitate the impurities; the diarylguanidine remains in solution. R. BRIGHTMAN.

Production of sulphuric anhydride compounds of tertiary bases and the application of the products. R. S. BARNES, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 317,736, 16.2.28).—A tertiary base (pyridine, quinoline, or homologues) is boiled with a pyrosulphate of an alkali or alkaline-earth metal (potassium pyrosulphate), and filtered hot. The sulphuric anhydride compound crystallises out on cooling, and may be used for the preparation of leuco-esters of vat dyes. C. HOLLINS.

Manufacture of 1-aminocarbazole and derivatives thereof. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 316,962, 7.5.28).—A 1-hydroxycarbazole is aminated, *e.g.*, with ammonium hydrogen sulphite, aqueous zinc chloride and ammonia, etc. C. HOLLINS.

Manufacture of arylthiazole compounds. H. P. ROBERTS and H. A. MERKLE, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,712,968, 14.5.29. Appl., 13.8.26).—Equimolecular proportions of a thiocarbamide, sulphur, and carbon disulphide are heated at 225–275° until maximum pressure has been developed. R. BRIGHTMAN.

Manufacture of aqueous diazonium salt solutions. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 317,355, 14.5.28).—Solutions of diazonium fluoroborates are made more stable by addition (to the solid salt or the solution) of an acid or an acid salt, *e.g.*, hydrochloric acid, fluoboric acid, sodium hydrogen sulphate, potassium tetroxalate, dibutyl-naphthalene- β -sulphonic acid. C. HOLLINS.

Hydrogenation of homologues of aniline and monoamino-compounds of aromatic hydrocarbons containing condensed benzene nuclei. I. G. FARBERIND. A.-G. (B.P. 290,175, 7.5.28. Ger., 7.5.27).—Free amino-bases are hydrogenated in the liquid phase at 270–300° under about 100 atm. pressure in presence of reduced nickel or cobalt. Toluidines and xylydines give cyclohexylamines, and β -naphthylamine yields 50% of *ar*- and 30% of *ac*-tetrahydro- β -naphthylamine. C. HOLLINS.

Manufacture of condensation products from aldehydes and phenols [moth-proofing materials]. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 316,900, 30.3.28).—An aldehyde (1 mol.), other than an aromatic hydroxyaldehyde, is condensed with 2 mols. of a *p*-halogenated phenol having a free *o*-position, preferably in presence of an acid (sulphuric acid, hydrochloric-acetic acid) or zinc chloride. Products from formaldehyde with 2 : 4-dichlorophenol or 5-chloro-*p*-cresol, from *p*-chlorobenzaldehyde and *p*-chlorophenol, and from benzaldehyde-*o*-sulphonic acid with *p*-chlorophenol or 2 : 4-dichlorophenol are described. They are used for proofing wool, furs, etc. against moths. C. HOLLINS.

Production of aromatic [hydr]oxyaldehydes. GRAESSER-MONSANTO CHEM. WORKS, LTD., and D. P. HUDSON (B.P. 317,381, 14.4., 30.5., and 19.6.28).—Safrole or isosafrole is converted by alcoholic potassium hydroxide into a mixture of the two isomeric ethoxymethyl ethers of 3 : 4-dihydroxypropenylbenzene, which may be selectively oxidised by alkali and nitrobenzene or nitrotoluene with production, after acidification, of 3 : 4-dihydroxybenzaldehyde (protocatechualdehyde), which is isolated as bisulphite compound. The unchanged 3-hydroxy-4-ethoxymethoxypropenylbenzene is alkylated and, after removal of the ethoxymethyl group with dilute alcoholic hydrochloric acid, oxidised to vanillin (or homologue) with alkali and nitrobenzene or nitrotoluene. C. HOLLINS.

Production of 4-hydroxy-3-alkoxybenzaldehydes and derivatives. GRAESSER-MONSANTO CHEM. WORKS, LTD., and D. P. HUDSON (B.P. 317,347, 12.5.28).—In mixtures of monoalkylated 3 : 4-dihydroxypropenylbenzenes oxidation with alkali and nitrobenzene attacks only the isomeride having a free 4-hydroxyl group. *E.g.*, huon pine oil (eugenol methyl ether) may be partially demethylated and isomerised, then oxidised with alkali and nitrobenzene to vanillin: the unattacked *iso*-chavibetol may be remethylated and returned for partial demethylation. C. HOLLINS.

Manufacture of hydroxybenzaldehydes. F. H. KRANZ, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,715,417, 4.6.29. Appl., 7.5.26).—Crude nitrobenzaldehyde (80–85% of *m*- and 20–15% of *o*-compound) is reduced and the aminobenzaldehydes are diazotised and steam-distilled with 10–15% sulphuric acid. The *o*-hydroxybenzaldehyde is collected in the distillate, and the *m*-compound recovered from the residual liquor. R. BRIGHTMAN.

Manufacture of benzoic acid. H. W. DAUDT, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,712,753, 14.5.29. Appl., 23.1.24).—Solutions of hydrogen phthal-

ates are heated at 150—300° in presence of a heavy-metal compound, *e.g.*, copper or mercury sulphate or chloride, as catalyst. R. BRIGHTMAN.

Manufacture of *o*-acylbenzoic acid compounds. J. M. TINKER and I. GUBELMANN, ASSTS. to NEWPORT Co. (U.S.P. 1,713,569, 21.5.29. Appl., 2.4.28).—The condensation product from phthalic anhydride and benzene (or a substituted benzene) in presence of aluminium chloride and a large excess of solvent is charged into ice and water below 40°, preferably below 25°, and the aqueous aluminium salt solution removed by siphoning or decantation. The residual (two) layers are washed free from aluminium salts with water and are then made alkaline; the solvent is removed by steam-distillation. R. BRIGHTMAN.

Manufacture of polysulphides of aromatic carboxylic acids and esters thereof. R. EDER (B.P. 291,100, 22.5.28. Switz., 27.5.27).—A thiolbenzoic acid, SH·Ar·CO₂H, or ester is treated in the cold with sulphur dichloride (for a trisulphide) or sulphur monochloride (for a tetrasulphide), preferably in a solvent (light petroleum, chloroform). The following *tri*- and *tetra*-sulphides are described: *benzoic acid* (2:2', m.p. 302—304°, and 296—298°, respectively; 3:3', m.p. 203°, and 188°; 4:4'-*tetrasulphide*, m.p. 293—295°), *ethyl benzoate* (2:2'-*trisulphide*, m.p. 108—110°; 3:3', m.p. 34—36°, and oily; 4:4'-*trisulphide*, m.p. 50—52°), *salicylic acid* [5:5', m.p. 224—225° (*diacetate*, m.p. 146°), and 228—229°], *ethyl salicylate* (5:5', m.p. 77—78°, and 73—74°), *allyl salicylate* (5:5', both oily). C. HOLLINS.

Purification of α -naphthol. A. B. DAVIS, ASSR. to PENNSYLVANIA COAL PRODUCTS Co. (U.S.P. 1,717,009, 11.6.29. Appl., 15.3.26).—Crude α -naphthol, containing, *e.g.*, 15—20% of β -naphthol, is boiled with water and lime, or other alkaline-earth hydroxide, and the insoluble calcium β -naphthoxide is collected at 20—25°. R. BRIGHTMAN.

Manufacture of β -naphthol-1-sulphonic acid. A. E. PARMELEE, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,716,082, 4.6.29. Appl., 20.2.24).— β -Naphthol is suspended in *o*-nitrotoluene and treated with chlorosulphonic acid below 10°. R. BRIGHTMAN.

Manufacture of nitro-compounds of dinaphthylene dioxide and derivatives thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,304, 9.5.28).—Sulphuric acid is added to a mixture of dinaphthylene dioxide (pasted with acetic acid) and concentrated aqueous potassium nitrate at 15—80°. According to the proportion of nitrate a mononitro- (m.p. 257°) or a dinitro- (m.p. above 300°) compound is obtained. C. HOLLINS.

Manufacture of alkoxyacridinium compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,869, 28.3.28).—3:7-Dihydroxyacridine is dissolved in water with the calculated quantity (2 mols.) of caustic alkali (or other acid-binding agent) and alkylated to give the dialkyl ether of the corresponding quaternary salt, the amount of water being at most equal to the weight of dihydroxyacridine used. The preparation of methyl and β -hydroxyethyl derivatives is described, the alkylating agents being methyl *p*-toluenesulphonate and ethylene chlorohydrin, respectively. C. HOLLINS.

Manufacture of basic derivatives of anthraquinone. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 316,989, 30.4.28).—The leuco-products from the amination or alkylamination of leucohydroxyanthraquinones are oxidised by an aromatic nitro-compound, which may be added before amination. C. HOLLINS.

Manufacture of 4-chloro-1-aminoanthraquinone-2-sulphonic acid. I. G. FARBENIND. A.-G. (B.P. 302,171, 10.12.28. Ger., 8.12.27).—Sodium 1-aminoanthraquinone-2-sulphonate in the form of a fine aqueous suspension is treated with chlorine below 0°. Completion of the reaction is seen by change in the crystalline form. C. HOLLINS.

Manufacture of condensation products of the benzanthrone series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 317,955, 10.7.28. Addn. to B.P. 298,775; B., 1928, 922).—The pyrazolanthrone salt of the prior process is formed *in situ*; *e.g.*, pyrazolanthrone intimately mixed with potassium carbonate is stirred in nitrobenzene and the whole is boiled for several hours before adding 3-nitrobenzanthrone. C. HOLLINS.

Continuous purification of crude carbon disulphide. E. LEGELER and P. ESSELMANN, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,728,686, 17.9.29. Appl., 7.12.25. Ger., 6.7.25).—See B.P. 254,676; B., 1927, 522.

Manufacture of [*o*- and *p*]-xylenes. E. TSCHUNKUR and F. EICHLER, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,727,682, 10.9.29. Appl., 15.9.27. Ger., 10.8.26).—See B.P. 300,940; B., 1929, 123.

Purification of crude aromatic hydrocarbons. A. O. JAEGER, ASSR. to SELDEN Co. (U.S.P. 1,729,543, 24.9.29. Appl., 11.10.27).—See B.P. 290,840; B., 1928, 516.

Carboxylated *N*- ω -aminoalkylaminonaphthalenes. W. HENTRICH, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,727,506, 10.9.29. Appl., 10.9.27. Ger., 23.10.26).—See B.P. 302,212; B., 1929, 200.

Preparation of a diazo compound from an amine of the aromatic series. K. SCHIRMACHER and K. RENN, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,728,217, 17.9.29. Appl., 24.3.27. Ger., 30.3.26).—See B.P. 268,789; B., 1928, 515.

Manufacture of anthraquinone nitriles. K. SCHIRMACHER and L. VAN ZÜTPHEN, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,728,216, 17.9.29. Appl., 6.11.26. Ger., 13.11.25).—See B.P. 261,422; B., 1927, 870.

Recovery of volatile substances from coal (B.P. 317,506). **Synthetic hydrocarbons and alcohols** (U.S.P. 1,711,913). **Phenols from coal tar** (U.S.P. 1,726,638). **Low-boiling hydrocarbons** (B.P. 318,270). **Decomposition of hydrocarbons** (B.P. 298,556). **Organic acids from oils** (B.P. 294,892).—See II. **Condensation products of arylamines and acetylene** (B.P. 292,168).—See XIII. **Tanning agent** (B.P. 297,830).—See XV. **Pyridine derivatives** (B.P. 288,629). **Resorcinol derivatives** (U.S.P. 1,717,105 and 1,717,098). **Ketones** (B.P. 361,750 and 317,194).—See XX.

IV.—DYESTUFFS.

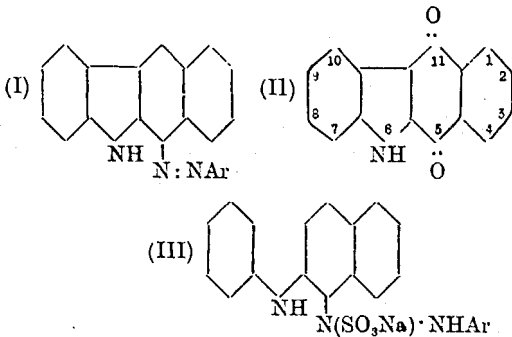
See A., Oct., 1172, **Derivatives of 2:5-dibromonitrobenzene** (RAIFORD and BREN). 1181, **Halo-genated 3:9-dibenzoyl derivatives of perylene** (SCHARVIN and SOBOROVSKI). 1186, **Dyes from cinchomeronic acid** (TEWARI).

PATENTS.

Manufacture of sulphur dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,139, 15.5.28).—2:2'-Dihydroxy-1:1'-dinaphthyl (di- β -naphthol) is heated with sulphur monochloride with or without addition of sulphur and/or a diluent (nitrobenzene) to give reddish-brown sulphide dyes. C. HOLLINS.

Dyes [soluble esters of leuco-vat dyes] and dyeing. R. S. BARNES, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 317,428, 16.5.28).—Malonyl chloride is added to a suspension of the dry leuco-compound of a vat dye in a tertiary base (pyridine). C. HOLLINS.

Manufacture of vat dyes of the α -naphthaquinone series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 317,928, 1.6.28).—An azo dye derived from a *lin*-naphthacarbazole (I) is boiled with dilute acid, by which it is converted into a naphthacarbazolequinone (II). The azo dye is preferably used in the form of its bisulphite compound; or the product (III),



obtained by condensing an arylazo- β -naphthol or - β -naphthylamine with an arylhydrazine bisulphite, is employed, the azo dye bisulphite compound being formed intermediately. The bisulphite compound of the dye made by coupling diazotised sulphanilic acid with *lin*-naphthacarbazole gives, when boiled with acid, *lin*-naphthacarbazole-5:11-quinone (II). From the condensation product of *m*-xylylhydrazine bisulphite and sulphanilic acid \rightarrow β -naphthol there is obtained the 7:9-dimethyl derivative, m.p. above 300°; β -naphthylhydrazine bisulphite and the same azo dye lead to *lin*-dinaphthacarbazole-5:13-quinone, m.p. above 300°. The products are vat dyes. C. HOLLINS.

Manufacture of dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 290,636, 16.5.28. Ger., 18.5.27. Addn. to B.P. 276,408; B., 1927, 809. Cf. B.P. 282,452; B., 1929, 591).—The halogen in 2-halogeno-1-amino-4-cycloalkylaminoanthraquinones or the 4:1:2-isomerides is replaced by a sulphonic group by means of sodium sulphite. C. HOLLINS.

Manufacture of anthraquinone derivatives and their application in dyeing. IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, W. W. TATUM, and G. E. WATTS (B.P. 317,555, 26.5.28).—Morpholine is condensed with halogenated anthraquinones, e.g., with 1-chloroanthraquinone (red on acetate silk), 1-chloro-4-aminoanthraquinone (violet), 1-bromo-4-methylaminoanthraquinone (blue-violet), 1:5-dibromo-4:8-dimethylaminoanthraquinone (lilac), 4:4'-dibromo-1:1'-dianthraquinonylamine (grey), 4-bromo-1-aminoanthraquinone-2-sulphonic acid (reddish-purple on wool). C. HOLLINS.

Manufacture of [thioindigoid vat] dye preparations. SOC. CHEM. IND. IN BASLE (B.P. 292,622, 23.6.28. Switz., 24.6.27).—Purer and fuller browns are obtained by using a mixture of α - and β -naphththioindigos than from either alone. The conversion into mixed leucoesters is described. C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. SOC. CHEM. IND. IN BASLE (B.P. 294,883, 27.7.28. Switz., 30.7.27. Addn. to B.P. 202,984; B., 1924, 1010).—Aryl or aralkyl ethers of *o*-aminophenols are coupled, especially on the fibre, with 2:3-hydroxynaphthoic *o*-aryloxy- or *o*-aralkoxy-anilides; e.g., 4-chloro-2-aminodiphenyl ether with 2:3-hydroxynaphthoic *o*-phenoxylanilide (bluish-red). C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,419, 15.5.28).—2:3-Hydroxynaphthoic arylamides are coupled in substance or on the fibre with diazotised 4:5- or 4:6-dichloro-*o*-toluidine. C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. K. and K. S. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,530, 19.5.28. Addn. to B.P. 294,291; B., 1928, 704).—2:3-Hydroxynaphthoic arylamides are coupled on the fibre or in substance with diazotised aminonaphthol ethers carrying substituents other than sulphonic, carboxyl, and hydroxyl groups. Examples of diazo components are 4-nitro-1-amino-7-naphthyl methyl ether, m.p. 210° (acetyl derivative, m.p. 227°); 4-nitro-1-amino-2-naphthyl ethyl ether, m.p. 152° (formyl derivative, m.p. 211°); 4-chloro-1-amino-2-naphthyl methyl ether, m.p. 225°; and 2:7-dimethoxy- α -naphthylamine. C. HOLLINS.

Manufacture of [acid azo] dyes. SOC. CHEM. IND. IN BASLE (B.P. 297,478, 22.9.28. Switz., 23.9.27).—Diazotised 1:2:4-aminonaphtholsulphonic acid is coupled with α -naphthol and the dye is prechromed, nitrosated, and reduced with sodium sulphide to give a steel-blue acid dye for wool or silk. C. HOLLINS.

Manufacture of [direct, developing] azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,431, 16.5.28).—A sulphonated and/or carboxylated arylamine or aminophenol is diazotised and coupled with a middle component; the product is then condensed with a nitrobenzoyl chloride, reduced, diazotised, and coupled with an aminoarylpyrazolone. The resulting dye may be diazotised and developed on the fibre. A second nitroaroyl residue may be introduced prior to diazotisation. Examples are: β -naphthylamine-4:8-disulphonic acid \rightarrow *p*-xylydine, *p*-nitrobenzoylated, reduced, \rightarrow 1-*m*-aminophenyl-3-methyl-5-pyrazolone

(clear yellow; developed to orange with β -naphthol or yellow with 1-phenyl-3-methyl-5-pyrazolone); β -naphthylamine-4:8-disulphonic acid \rightarrow *o*-anisidine, *p*-nitrobenzoylated, reduced, *p*-nitrobenzoylated, reduced, \rightarrow 1-*m*-aminophenyl-3-methyl-5-pyrazolone (yellow, as preceding).
C. HOLLINS.

Manufacture of [mordant acid] azo dyes and the application thereof. SOC. CHEM. IND. IN BASLE (B.P. 313,095, 26.7.28. Switz., 26.7.27).—Pyrazolonecarboxylic acid or esters made from hydrazine and an oxalacetic ester are coupled with diazotised *o*-aminophenols, *e.g.*, 4-chloro-*o*-aminophenol-6-sulphonic acid (orange on wool, blue-red after chroming), 1:2:4-aminonaphthol-sulphonic acid (after-chromed violet; chrome-printing violet), or its nitro-compound (after-chromed bordeaux).
C. HOLLINS.

Manufacture of azo dyes containing chromium. SOC. CHEM. IND. IN BASLE (B.P. 296,680, 3.9.28. Switz., 3.9.27).—Chromable azo dyes are dissolved or suspended in a solution of chromium acetate (etc.), the whole is evaporated, and the residue is baked at 145–160° for 24 hrs. or more to complete the prechroming.
C. HOLLINS.

Manufacture of azo dyes forming metallic salts. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,338, 13.4.28).—Carbamides or thiocarbamides, derived from 2:5-diaminobenzoic acid and containing two or four aminobenzamido- or aminobenzenesulphonamido-residues, are used as first components in azo dyes suitable for after-coppering. They are best prepared by phosgenation or thiophosgenation of suitable couplings. Examples are: 5-nitroanthranilic acid \rightarrow acetoacetic sulpho-*o*-anisidide, reduced, *m*-nitrobenzoylated, reduced, and phosgenated or thiophosgenated (direct yellow; after-coppered greenish-yellow); 5-nitroanthranilic acid \rightarrow 1-*p*-sulphophenyl-3-methyl-5-pyrazolone, reduced, *m*-nitrobenzoylated, reduced, phosgenated (direct orange; after-coppered, brownish-yellow); 5-nitroanthranilic acid \rightarrow 1-(6-sulpho- β -naphthyl)-3-methyl-5-pyrazolone, reduced, 2-nitroanisoylated, reduced, and phosgenated (direct orange; after-coppered, yellowish-brown); 5-nitroanthranilic acid \rightarrow N.W.-acid, reduced, *m*-nitrobenzoylated, reduced, and either phosgenated (direct bluish-red; after-coppered, bordeaux-red) or 2-nitroanisoylated, reduced, and phosgenated (similar shades); 5-nitroanthranilic acid \rightarrow 1-hydroxy-8-ethoxynaphthalene-3:6-disulphonic acid, reduced, *m*-nitrobenzoylated, reduced, and phosgenated (direct bluish-red; after-coppered, reddish-violet); 5-nitroanthranilic acid \rightarrow acetyl-H-acid, *m*-nitrobenzoylated, reduced, and phosgenated (direct red-violet; after-coppered, clear violet).
C. HOLLINS.

Manufacture of [mixed azo] dyes containing metal. SOC. CHEM. IND. IN BASLE (B.P. 297,331, 17.9.28. Switz., 17.9.27).—Mixtures of chromable azo dyes are prechromed together, or a prechromed azo dye is treated with an azo dye (which may be the same or different), or two or more azo dyes are made together in presence of a chroming agent. Examples are: prechromed [1:2:4-aminonaphtholsulphonic acid \rightarrow α -naphthol], boiled in water with [6-nitro-*o*-aminophenol-4-sulphonic acid \rightarrow α -naphthol] for a blackish-navy-blue

on wool or silk; [nitro-1:2:4-aminonaphtholsulphonic acid \rightarrow β -naphthol] and its reduction product, boiled with chromium formate, for a blue-black on wool or silk.

C. HOLLINS.

Manufacture of [dibenzanthrone] vat dyes. B. MAYER and H. STEBENBÜRGER, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,728,068, 10.9.29. Appl., 12.1.28. Switz., 2.2.27).—See B.P. 284,656; B., 1929, 674.

Dyes of the anthraquinone series [for acetate silk]. R. E. SCHMIDT and R. BERLINER, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,706,498, 26.3.29. Appl., 21.1.28. Ger., 25.1.27).—See B.P. 284,242; B., 1929, 637.

Vat dyes of the anthracene series. G. KRÄNZLEIN and R. EBERT, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,709,985, 23.4.29. Appl., 11.6.25. Ger., 13.8.24).—See B.P. 238,523; B., 1925, 876.

Manufacture of vat dyes. G. KALISCHER and W. ZERWECK, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,705,817, 19.3.29. Appl., 8.7.26. Ger., 20.7.25).—See B.P. 288,666; B., 1928, 441.

Manufacture of vat dyes of the isodibenzanthrone series. A. ZINKE, ASSR. to F. BENSA (U.S.P. 1,705,953, 19.3.29. Appl., 30.9.25. Austr., 11.11.24).—See B.P. 242,306; B., 1927, 325.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of fat in wool. P. KRAIS and K. BILTZ (Leipziger Monatssehr. Textil-Ind., 1929, 44, 69; Chem. Zentr., 1929, i, 1767–1768).—Washed wool is extracted twice with ether and alcohol, then boiled with dilute sulphuric acid, and again extracted. Fine and raw wool contained, respectively, 1.74 and 2.05% of fat.
A. A. ELDRIDGE.

Tensile strength and elongation of continuous cellulose fibres. J. KALFF (Rec. trav. chim., 1929, 48, 997–1004).—An examination of the behaviour of different kinds of fibres of regenerated cellulose shows that artificial silk has no elasticity limit, even very small loads causing extensions of which part is irreversible. A high breaking elongation does not necessarily correspond with a strong flow of the fibre under small tension. The behaviour of these fibres can be interpreted in terms of Shorter's theory of the composition of wool fibres. The tensile strength as found by means of a Schopper tester depends on the duration of the test. Fibres with a small breaking elongation have a higher tensile strength with increasing rate of extension, but beyond a certain speed the tensile strength falls again. For fibres of high breaking elongation only the first ascending part of the curve was observed, the speed which in this case would correspond with the maximum tensile strength being unattainable. The breaking elongation of viscose silk is independent of the duration of the determination. Cuprammonium silk has a greater elongation the greater is the speed of the Schopper machine. Reasons are given for the adoption of the percentage extension per minute as a measure of the speed of the testing machine in place of the usual measure of the speed of the lower jaw. To obtain comparable and constant values for the tensile strength of plastic fibres determinations should

be carried out at that speed which gives the maximum value for the tensile strength. F. G. TRYHORN.

Sulphur balance in the manufacture of sulphate-cellulose. J. DIENISOV (Zellstoff u. Papier, 1929, 9, 607—609).—The distribution of sulphur during the cooking and sulphate-recovery processes has been investigated. During the cooking about one half of the available sulphur is combined with lignin and other organic substances, whilst of the remainder some forms methyl sulphide and methyl mercaptan and some remains as excess unchanged sodium sulphide. Values recorded are: combined with lignin 36.7%, with other organic substances 15.1%, removed as methyl sulphide and mercaptan in the waste gases 15.0%, as unchanged sodium sulphide 16.8%. Dry distillation of the residue obtained by evaporation of black liquor liberates about 50% of the sulphur present in the form of hydrogen sulphide and mercaptan. The sulphate treated in the smelting oven is finally converted into carbonate and the sulphur takes part in only one cycle of operations with the liquor. In the evaluation of the sulphate process as compared with the caustic soda process the sulphate should not be judged according to the amount of sodium sulphide which it can yield, but by its sodium oxide yield; thus, according to its Na_2O content, soda is 1.7 times as costly as sulphate. The consumption of sulphate and losses occurring in technical experiments are discussed. B. P. RIDGE.

Cogon and rice straw as raw material for paper manufacture. F. D. REYES and A. O. CRUZ (Philippine J. Sci., 1929, 38, 367—376).—Cogon grass and rice straw are available in commercial quantities for the manufacture of paper. The yield of cogon is about 12—16 tons of air-dry grass per hectare, but as the more easily accessible land will probably in the future be devoted to agriculture the difficulties of transportation will increase. Rice straw, however, is a by-product and offers fewer transportation difficulties; further, rice straw requires only about 8% of soda as against 20% for cogon to give a bleachable pulp, and shows a higher yield. For cardboard cogon requires digestion with at least 4% of soda and gives a yield of 51.5% of pulp (oven-dry pulp on oven-dry grass), whilst rice straw requires only 2% of caustic soda and gives a yield of 55.4%. Cogon paper is somewhat stronger than that made from rice straw. D. J. NORMAN.

Bisulphite liquors. BERNDT.—See VII.

See also A., Oct., 1124, Action of α -rays on thin films (PAYLOVSKI). 1140, Adsorption of water by cotton (URQUHART). 1141, Adsorption of water vapour by cotton cellulose (PEIRCE).

PATENTS.

Degumming of silk. TAKAMINE LAB., INC., Assees. of J. TAKAMINE, JUN. (B.P. 301,419, 26.10.28. U.S., 29.11.27).—Silk, either woven or in skeins, is agitated for about 15 min. with water at high temperatures and pressures, *e.g.*, 50 lb./in.². This treatment may, if desired, be repeated with the addition of $\frac{1}{2}$ —1% of neutral soap. D. J. NORMAN.

Manufacture of [waterproof] vulcanised fibre. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P.

316,700, 11.5.28).—Vulcanised fibre is impregnated with aqueous solutions of substances capable of giving resinous condensation products, *e.g.*, phenol or its homologues and formaldehyde, and formation of the resin is effected within the mass by means of heat. D. J. NORMAN.

Softening of cellulose fibres to facilitate their acetylation. RUTH-ALDO Co., INC., Assees. of H. L. BARTHELEMY (B.P. 303,134, 3.8.28. Fr., 29.12.27).—Cellulose is treated, prior to esterification, with a halogen or a halogen halide, *e.g.*, iodine trichloride, and glacial acetic acid in either order or in admixture in a vaporised or atomised state, the temperature being kept at below 65° (preferably at 20—40°).

D. J. NORMAN.

Production of cellulose esters. C. RUZICKA (B.P. 316,593, 27.2.28).—Cellulose (*e.g.*, cotton linters) is treated with an esterifying mixture containing the whole of the fatty acid anhydride, with or without a catalyst (sulphuric acid), and some or all of the diluent, and the subsequent esterification is controlled by adding formic, butyric, or propionic acid, or a mixture thereof (preferably anhydrous), in sufficient quantity, *i.e.*, $\frac{1}{3}$ — $\frac{1}{2}$ of that of the acid anhydride used, to reduce local heating and to prevent discoloration and irregular esterification. Esterification is carried out at 20°.

D. J. NORMAN.

Manufacture of cellulose esters of high uniformity. H. LE B. GRAY, Assr. to EASTMAN KODAK Co. (U.S.P. 1,711,940—1, 7.5.29. Appl., 25.8.27).—Cellulose is esterified, *e.g.*, acetylated, (A) in a continuous or discontinuous vacuum (14—40 mm.) until entrapped air is removed, or (B) in presence of an inert volatile organic solvent, the latter being evaporated under a vacuum after mixing. *E.g.*, cotton fibre under vacuum is mixed with a solution of acetic anhydride in chloroform (or ether) containing sulphuric and phosphoric acids; the chloroform is allowed to evaporate and the acetylation completed as usual.

R. BRIGHTMAN.

Treatment of hydrated cellulose. I. G. FARBENIND. A.-G. (B.P. 294,551, 25.7.28. Ger., 25.7.27. Addn. to B.P. 278,684; B., 1929, 242).—In the process of the prior patent, the aqueous solution of the condensation product may contain an organic solvent, *e.g.*, an alcohol, a water-soluble ester or ether, a glycol, and, if desired, a wetting agent such as soap or a sulphionate; condensation is then effected in a feebly acid solution with the addition of a buffer mixture (mono- and di-sodium phosphate). F. R. ENNOS.

Reducing the viscosity of cellulose ethers. B. N. LOUGOVY, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,726,357, 27.8.29. Appl., 9.6.26).—A solution of the ester in a solvent of high dispersing power is diluted with a mixture of the latter with a miscible non-solvent, whereby an ester of lower viscosity is precipitated.

F. G. CLARKE.

Apparatus for manufacture of artificial silk by the dry-spinning method. C. RUZICKA (B.P. 317,108, 17.12.28).—The spinning cell is jacketed and the hot air used for spinning is passed downwards through the jacket and then upwards through the spinning cell. D. J. NORMAN.

Apparatus for spinning solutions of cellulose ethers or esters. RUTH-ALDO Co., INC., Assees. of (Miss) M. KLEIN (B.P. 300,166, 22.6.28. Fr., 7.11.27. Addn. to B.P. 292,561 and 292,608; B., 1929, 595, 774).—The air-circulation arrangements described in the earlier patents are modified. Each zone of the cell has its own inlet and outlet so that the circulation of the evaporative medium in one zone is independent of that in the other. The evaporative medium issuing from one zone may, optionally after passing through heating, cooling, or other devices, be circulated in the other zone.
D. J. NORMAN.

Manufacture [spinning] of artificial silk [of fine titer]. I. G. FARBENIND. A.-G. (B.P. 287,862 and 288,153, [A] 26.3.28, [B] 29.3.28. Ger., [A] 24.3.27, [B] 1.4.27).—In (A) the box-spinning method is used, the draught and speed of revolution of the spinning box being reduced and the diameter of the box correspondingly increased to maintain a sufficiently high centrifugal force. In (B) an ordinary spool-spinning machine without diminished output is employed, each spinning nozzle being replaced by a pair arranged one behind the other. To effect this the supply ducts and spinning pumps are duplicated or each pump is replaced by a unit consisting of two pumps carried on a common mounting and fed from the existing supply duct; also the thread guides are replaced by double thread guides each of which is provided with a pair of spools. F. R. ENNOS.

Manufacture of artificial filaments. C. J. HEALEY (B.P. 317,126, 11.5.28).—Cuprammonium cellulose solutions are extruded into triangular troughs placed just below the surface of the coagulating liquid in a large bath; this is divided by a partition into forward and rear compartments, which are in communication with each other near the bottom. The troughs are situated in the rear compartment, but their lips protrude over the partition, and a filament in passing through them causes circulation of the liquid by carrying the overflow from the lip to the forward chamber; the return flow is made below the partition at the bottom of the bath. Ammonia escapes from the liquid surface at the lips of the troughs, and precipitated copper is collected in the forward compartment of the bath. The temperature of the liquid is regulated by means of, *e.g.*, a heating coil.
B. P. RIDGE.

Production of highly valuable viscose products. O. VON KOHORN (O. KOHORN & Co.), and A. JÄGER (B.P. 301,305, 18.10.28. Ger., 26.11.27).—Viscose silk of improved quality is obtained by adding to the spinning bath substances containing keratin or decomposition products thereof, *e.g.*, the soluble products of wool, leather, skin, horn, etc. Dissolution of such substances is facilitated if they are first treated with metal salts, *e.g.*, chromium salts. A suitable spinning bath contains 100—120 g. of chrome leather (previously dissolved in warm 10% sulphuric acid), 120—130 g. of sodium sulphate, and 130—140 g. of sulphuric acid per litre. The presence of keratin substances in the spinning bath minimises the effects on the quality of the thread of variations in the maturity of the viscose solution.
D. J. NORMAN.

[Improving the physical properties of] artificial

silk. E. C. R. MARKS. From CHEM. FABR. STOCKHAUSEN & Co. (B.P. 317,951, 3.7.28).—The tensile strength, flexibility, and touch of artificial silk threads are improved by treatment with a 0.2% solution of the sulphonated fats and oils described in B.P. 293,480 and 293,717 (B., 1928, 678, 718). In the case of viscose and cuprammonium silks, an addition of 2 g. of the sulphonated products may also be made to each litre of the spinning bath.
A. J. HALL.

Treatment of threads, films, etc. of cellulose esters. COURTAULDS, LTD., and C. DIAMOND (B.P. 317,936, 11.6.28).—In order to reduce the tendency of cellulose acetate products to melt when heated, a solution of an organic ester of silicic acid or "silicon ester," *e.g.*, ethyl silicate, is added before or after spinning.
F. R. ENNOS.

Production of artificial threads and the like from viscose. COURTAULDS, LTD., H. J. HEGAN, and E. HAZELEY (B.P. 317,912, 29.5.28).—After leaving the spinning bath viscose threads are passed over a rotating wheel on the same level as the bath, thence vertically downwards on to a second rotating wheel on which they are treated with water or other liquid, then on to a third rotating wheel with or without such treatment, and finally to a collecting device.
F. R. ENNOS.

Production of pressed masses from cellulose derivatives. A. EICHENGRÜN (B.P. 290,989, 21.5.28. Ger., 20.5.27).—Highly concentrated, viscous solutions of cellulose esters or ethers, with or without the addition of softening agents, filling materials, resins, artificial resins, condensation products, rubber, etc., are extruded in the form of a heated paste through narrow apertures. The extruded material, on reduction of the pressure, swells up and forms a porous mass which is pressed in heated moulds or is extruded in a hot fluid condition through suitable apertures to form finished articles.
F. R. ENNOS.

Fibre liberation and recovery of valuable constituents of the waste liquor. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,719,129, 2.7.29. Appl., 28.10.22).—The waste liquor from the digestion of cellulose with sulphurous acid and sodium-sulphur compounds is mixed with the waste liquor from an alkaline-digestion process, and the whole is concentrated, smelted in a reducing atmosphere, and the residue extracted with water to give a liquor which may be used for the alkaline digestion of further quantities of cellulosic material.
D. J. NORMAN.

Recovery of heat and chemicals [from cellulose waste liquor]. G. A. RICHTER and W. B. VAN ARSDEL, Assrs. to BROWN Co. (U.S.P. 1,719,130, 2.7.29. Appl., 16.12.25).—The gases arising from the combustion and smelting of waste liquor from the digestion of cellulosic material are passed through a mass of inert interstitial material to effect condensation of water vapour and dissolution of the soluble constituents of the waste gases therein, the resulting liquor being subsequently used for preparing fresh digestion liquor. A second cooling tower is provided for use while the heat is recovered from the first tower.
D. J. NORMAN.

Utilisation of sulphite-cellulose waste liquor. W. MICHAEL and A. PALM, Assrs. to I. G. FARBENIND.

A.-G. (U.S.P. 1,723,800, 8.6.29. Appl., 16.3.28. Ger., 21.3.27).—The free sulphurous acid is expelled, the liquor heated in a closed vessel to above 150°, the carbon collected on a filter, and the ammonia liberated by means of a base stronger than ammonia. B. P. RIDGE.

Extracting cellulose-containing materials from the sudd or papyrus plant. B. DORNER (B.P. 316,413, 7.7.28).—The material is cut into lengths and boiled for 1–2 hrs. at 1–2 atm. pressure with sulphurous acid. The boiled material is then washed on a screen to remove pithy matter. Alternatively, the material may be digested with a very dilute sulphurous acid solution, and then left to ferment, after which the pithy matter is readily removed by washing. The resulting half-stuff is preferably treated with salt to prevent mould growth. D. J. NORMAN.

Production of [wood] pulp. L. BRADLEY and E. P. McKEEFE (U.S.P. 1,722,993, 6.8.29. Appl., 3.11.21. Renewed 30.10.28).—Wood is submitted to a preliminary partial digestion with an acid cooking liquor containing sulphurous acid, and is then cooked with a liquor containing normal alkali sulphite. B. P. RIDGE.

Manufacture of artificial board. (A) C. C. VOGT, (B) C. C. VOGT and E. J. PIEPER, Assrs. to ARMSTRONG CORK Co. (U.S.P. 1,718,011–2, 18.6.29. Appl., 29.9.27).—(A) Plant material, *e.g.*, maize stalks or cobs, is autoclaved with water, *e.g.*, at 140–180° for $\frac{1}{2}$ –1 hr., the fibres are mechanically separated, the product is saturated with an aqueous solution of the natural binding material, treated with a reagent, *e.g.*, with a phenolic compound and a condensing agent, to render the soluble binder insoluble, before or after forming into board, and the board is baked. (B) A water-insoluble toughening agent, *e.g.*, pitch, asphalt, coal tar, or wood tar, is added. R. BRIGHTMAN.

Treatment of cellulose with lactic acid. H. HEIMANN and A. BAYERL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,728,206, 17.9.29. Appl., 11.12.26. Ger., 24.3.26).—See B.P. 263,239; B., 1928, 444.

Celluloid compounds. R. ROLAND (B.P. 318,818, 14.1.29).—See U.S.P. 1,713,482; B., 1929, 750.

Settling tanks particularly adapted for treatment of wool-washing liquors. H. W. FAWCETT (B.P. 318,558, 6.3.28).

Washing artificial silk skeins and other similar material. D. F. BATE (B.P. 318,715, 26.7.28).

[Machine for] manufacturing [sheet] paper. J. R. HAPPER (B.P. 318,771, 12.10.28).

Impregnating paper for insulation of electric cables and apparatus therefor. C. J. BEAVER, and W. T. GLOVER & Co., LTD. (B.P. 318,647, 7.3.28).

Moth-proofing agents (B.P. 316,900).—See III. **Converting cellulose into sugar** (B.P. 315,462).—See XVII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching clays. ECKART.—See VIII.

PATENTS.

Dyeing [with oxidation colours]. E. SCHUELLER (B.P. 307,480, 23.8.28. Fr., 10.3.28).—In the dyeing of

textiles, hairs, skins, etc. with oxidation colours (*e.g.*, phenols, *p*-phenylenediamine or its homologues) by the aid of hydrogen peroxide, rapid and complete oxidation is obtained by oxidising in the presence of colloidal catalysts which operate similarly to peroxidases. Suitable catalysts include colloidal metals, but more particularly colloidal vegetable substances, such as extracts of potatoes, onions, cereals, etc., or animal substances, such as milk and blood; during oxidation gaseous oxygen is not liberated, and the resulting pigments are usually faster and less toxic. A. J. HALL.

Dyeing of fabric. T. E. ROBERTSON (B.P. 316,641, 1.5.28).—Dyed fabric is stripped to any desired degree by means of a stripping solution, levelness being ensured by addition of crude naphtha which has been treated with 10% sulphuric acid; the action is stopped by addition of acid. Dye may be added to the stripping solution, and has greater colouring effect than when applied to completely stripped fabric. Material dyed with aniline-black may, *e.g.*, be changed to yellow, red, or blue. C. HOLLINS.

Dyeing, printing, or stencilling of materials made of or containing cellulose derivatives [esters or ethers]. BRIT. CELANESE, LTD., H. DREYFUS, G. H. ELLIS, T. OCKMAN, and H. C. OLPIN (B.P. 316,526, 26.4.28. Addn. to B.P. 291,118; B., 1928, 601).—Acetate silk etc. is dyed with unsulphonated nitroarylated carbamides or thiocarbamides, or with unsulphonated aminoarylated carbamides or thiocarbamides diazotised and coupled in substance or on the fibre with coupling components. Examples are: 2:4-dinitrophenylcarbamide (yellow); 4:4'-diaminodiphenylcarbamide \rightarrow 2 mols. of phenol (yellow) or *m*-phenylenediamine (golden-orange). C. HOLLINS.

Loading of silk or silk-containing textile material in the form of hanks or runs. R. CLAVEL (B.P. 296,017–8, 31.5.28. Switz., 23.8.27).—(A) The material is placed in a centrifuge which is connected by means of a multiway cock with a series of supply tanks and a corresponding number of reservoirs. The former, which contain the liquids to be used, *e.g.*, formic acid, stannic chloride, sodium dihydrogen phosphate solution, are connected in turn with the centrifuge in which the yarn is treated for the required time, and afterwards the liquid is removed by operation of the centrifuge, and transferred to the corresponding reservoir, from which it is subsequently circulated again to the supply tank. The yarn is thus left in the centrifuge without intermediate washing until all stages of the loading process are completed. (B) Fabric is similarly loaded without intermediate washing by treatment in a tank which is connected in turn to reservoirs containing the required liquids as in (A). The material is suspended in the tank from perforated pipes through which the liquid from the bottom of the tank is pumped until the desired treatment is completed. The liquid so used is then transferred again to the reservoir, the tank is re-charged with the next solution, which is circulated over the fabric, and the sequence of operations is continued until the loading treatment is finished. B. P. RIDGE.

Dressing of artificial silk. I. G. FARBENIND. A.-G. (B.P. 317,117, 9.5.28).—Artificial silk yarns are treated

with 1–2% solutions of the sodium salts of cellulose-di- or -poly-fatty acids, and the free acids are liberated, if desired, by subsequent treatment of the material with acetic acid. In an example, alkali cellulose is formed by treatment of cellulose with concentrated sodium hydroxide solution, and the product treated with a sufficient excess of the sodium salt of monochloroacetic acid to produce at least the cellulose diacetate. After keeping for 2–3 days at room temperature, the viscous mass obtained is stirred with water, diluted to the required concentration, and applied to the yarn in the usual manner. Since both the cellulose-fatty acid and the sodium salt are soluble in water, they may be removed from the yarn after fabric has been woven from it and the latter may be dyed without further treatment.

B. P. RIDGE.

Method of preparing buckram. S. HARARY (U.S.P. 1,712,991, 14.5.29. Appl., 20.1.28).—Fabric material is sized with starch or glue, saturated with hydrochloric acid, and dried.

R. BRIGHTMAN.

Manufacture of leather substitute. MECHANICAL RUBBER CO., Assees. of R. P. ROSE and A. F. OWEN (B.P. 295,387, 2.8.28. U.S., 11.8.27).—In the manufacture of leather substitutes by incorporating rubber latex with beaten fibres, a more uniform deposition of the rubber on the fibre is obtained by adding the coagulant, *e.g.*, alum, very slowly or preferably in two stages, the mixture being first brought to a faintly alkaline or neutral state, and later, after mixing for about $\frac{1}{2}$ hr., made distinctly acid by further additions of alum.

D. J. NORMAN.

Purification of detergent [dry-cleaning] solutions. G. B. BOON, Assr. to R. R. STOLLEY, INC. (U.S.P. 1,711,829, 7.5.29. Appl., 16.1.28).—Used gasoline or other dry-cleaning solvent is passed upwardly through sodium hydroxide solution and "darco" or other cleaning agent in the usual manner, and is then washed by passing upwards through water and finally through calcium chloride to remove traces of caustic and water.

R. BRIGHTMAN.

Production of azo dyes on silk. K. WOETZEL and H. LINT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,727,920, 10.9.29. Appl., 9.11.25. Ger., 20.6.25).—See B.P. 253,865; B., 1927, 553.

Cleaning process. A. E. HATFIELD and E. A. ALLIOTT (U.S.P. 1,728,343, 17.9.29. Appl., 24.6.27. U.K., 15.2.27).—See B.P. 289,581; B., 1928, 637.

[Feeding air-steam mixture into drums for] oxidation of articles to be dyed with aniline oxidation black. P. BAUMERT and I. G. HERMSDORF (B.P. 318,745, 4.9.28).

Apparatus for washing and after-treatment of artificial threads spun on spools, bobbins, or other carriers. I. G. FARBENIND. A.-G. (B.P. 294,547, 24.7.28. Ger., 25.7.27).

Colouring, sizing, impregnating, and similarly treating paper. I. G. FARBENIND. A.-G. (B.P. 295,288, 8.8.28. Ger., 8.8.27. Addn. to B.P. 293,000).

Production of imitation [antique] leather [from vegetable fabrics]. E. KATZ (B.P. 296,661, 22.6.28. Austr., 3.9.27).

Dyeing with leuco-vat dyes (B.P. 317,428). Anthraquinone derivatives for dyeing (B.P. 317,555). Mordant acid azo dyes (B.P. 313,095).—See IV. Resinous condensation products (B.P. 292,168).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Artificial crystallised Karlsbad salts. G. WALLRABE (Pharm. Zentr., 1929, 70, 581–583).—The material marketed under this name by manufacturers is technically pure Glauber salt, containing only traces of carbonate and bicarbonate, which are essential ingredients of the material prepared according to the German Pharmacopœia.

S. I. LEVY.

Bisulphite liquors containing magnesia. K. BERNDT (Papier-Fabr., 1929, 27, 565–569).—Methods at present in use for the production of these liquors from dolomite, magnesite, magnesium chloride, and sulphate are discussed, and a new method is described by which magnesium sulphite is obtained as a by-product in the manufacture of ammonium sulphate from magnesium sulphate, ammonia, and sulphurous acid. The specific advantages or disadvantages of these methods depend on the availability of the raw materials, cost of transport, magnesium oxide content of the raw material, etc. Results obtained by prominent workers showing the conditions under which the use of magnesium bisulphite liquors is of importance, and the properties of the cellulose pulps obtained, are discussed, and the conclusion is drawn that such liquors offer considerable advantages in pulp manufacture.

B. P. RIDGE.

Determination of sulphate in chromic acid and in chromium-plating baths. H. H. WILLARD and R. SCHNEIDEWIND (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 14 pp.).—The chromic acid must first be reduced to chromic salt, since otherwise the barium sulphate precipitate is liable to be contaminated with chromate. Reduction may be effected by hydroxylamine hydrochloride, hydrogen peroxide, ethyl alcohol, or methyl alcohol. The reduced solution contains some of the chromium in the form of complex ions containing sulphate, and this sulphate is precipitated very slowly and incompletely by barium chloride. This difficulty is avoided by heating the reduced solution with acetic acid, whereby the sulphate in the complex ions is replaced by acetate and all the sulphate can then be rapidly precipitated. As a rapid control method for plating baths, turbidimetric determination of barium sulphate is satisfactory. A modified turbidimeter is described in which, instead of measuring the depth of liquid required to extinguish the light, a fixed depth of liquid is maintained and the intensity of illumination varied by varying the resistance in the lamp circuit. A calibration chart may be prepared by plotting concentration of sulphate against the resistance in the lamp circuit or the voltage across the lamp terminals.

H. J. T. ELLINGHAM.

Determination of hydroxide and carbonate in solutions. J. LINDNER (Z. anal. Chem., 1929, 78, 188–198).—The determination of the hydroxide content of alkali solutions containing carbonate by addition of

barium chloride followed by titration with acid, using phenolphthalein as indicator, is subject to several sources of error. When silicate is present, part is titrated with the hydroxide, and when the precipitation is made in a hot solution, part of the hydroxide appears to be adsorbed by the barium carbonate. In the absence of silicate, precipitation from an ice-cold solution is satisfactory, but almost as good results are obtained by adding the greater part of the acid required to neutralise the hydroxide previous to addition of barium chloride. Alternatively, the carbonate may be determined by passing a current of air through the hot solution after acidification and collecting the carbon dioxide in barium hydroxide solution, which is then titrated with standard acid.

A. R. POWELL.

Sulphate in chromium-plating baths. STOUT and PETCHART.—See XI. **Determination of mercuric iodide.** BROCKMAN.—See XX.

See also A., Oct., 1151, **Activation of sulphur** (SCHWARTZ and SCHENK). **Electrolytic preparation of metal permanganates** (RAPIN). **Perchlorate from electrolytic preparation of chlorate** (PAMFILOV and FEDOROVA). 1153, **Preparation of luminophores** (VANINO and PREM). 1157, **Recommended specifications for analytical reagents** (COLLINS and others). 1158, **Determination of halogen in perchlorates** (HELLER and others). **Potentiometric titration of iodides** (TOMÍČEK). **Volumetric determination of alkali fluorides and silica** (SIEGEL). **Colorimetric determination of phosphorus and arsenic** (TRUOG and MEYER). **Iodometric determination of phosphorous acid** (SCHWICKER). **Colorimetric determination of phosphoric acid** (ROZANOV). 1159, **Volumetric determination of phosphoric acid** (SMITH). **Determination of neon in natural gases** (PÉNTCHEV). **Determination of thallium** (JILEK and LUKAS). **Determination of copper with 5:7-dibromo-8-hydroxyquinoline** (HAASE). **Potentiometric determination of iron and molybdenum** (BRINTZINGER and SCHIEFERDECKER). 1160, **Preparation of antimony-free arsenious oxide and its analysis** (FOULK and HORTON).

PATENTS.

Concentration of sulphuric acid. W. H. SIMONSON and O. MANTIUS (U.S.P. 1,726,321, 27.8.29. Appl., 8.6.23).—The acid is indirectly heated by a stream of a liquid heating medium, the b.p. of which is above that of the acid at all stages, and which is always sufficiently hot to produce evaporation of the acid.

F. G. CLARKE.

Manufacture of phosphoric acid. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 318,274, 31.5.28).—Concentrated sulphuric acid is treated with successive small quantities of material containing calcium phosphate until the acid is converted completely into phosphoric acid, the calcium sulphate formed being removed after each addition.

L. A. COLES.

Production of ammonia from its elements. C. MÜLLER and F. KRÄGELOH, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,727,174, 3.9.29. Appl., 14.3.28. Ger., 16.3.27).—Before reaching the catalyst, the nitrogen-hydrogen mixture is freed from sulphur compounds,

carbon monoxide, oxygen, and water vapour by passing it over anhydrous potassium aluminium ferrocyanide at a lower temperature than that of the catalyst.

F. G. CLARKE.

Manufacture of diammonium phosphate. G. H. BUCHANAN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,716,415, 11.6.29. Appl., 17.6.24).—Phosphoric acid solution containing 28–43% P_2O_5 is treated with gaseous ammonia at, e.g., 110°, and cooled to below 90°. The diammonium phosphate is separated by centrifuging and the mother-liquor is treated with phosphoric acid to convert the remaining diammonium salt into ammonium hydrogen phosphate, concentrated, and again treated with ammonia.

R. BRIGHTMAN.

Manufacture of potassium carbonate. R. D. PIKE, Assec. of L. V. STECK and R. CUMMINGS (U.S.P. 1,716,663, 11.6.29. Appl., 6.3.28).—Crude carbonate brine (e.g., total solids 5%) containing sodium and potassium carbonate is circulated slowly upwards through potassium zeolite, giving an effluent containing, e.g., 96.2% of potassium carbonate. The potassium zeolite is regenerated with, e.g., potassium chloride solution, the potassium chloride from the waste regenerating solution being recovered by evaporation and salting out.

R. BRIGHTMAN.

Purification of water-soluble metal sulphides. E. A. TAYLOR, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,713,409, 14.5.29. Appl., 14.5.28).—The technical aqueous solutions of the soluble sulphide, e.g., sodium or barium sulphide, are decolorised by boiling with 0.1–2.0% of sodium cyanide for 5–15 min.

R. BRIGHTMAN.

Manufacture of black ash [barium sulphide]. Z. DE HORVATH, Assr. to EAGLE PITCHER LEAD Co. (U.S.P. 1,723,138, 6.8.29. Appl., 12.10.26).—Sufficient calcium carbonate is added to the barytes and carbonaceous material to combine with most of the silica present, before the charge is heated in a rotary kiln.

F. G. CLARKE.

Manufacture of aluminium chloride. H. I. LEA and C. W. HUMPHREY (U.S.P. 1,713,968, 21.5.29. Appl., 31.5.22).—Aluminium oxide is heated in a retort and impregnated by spraying with hot hydrocarbon vapours. Excess of hydrocarbon vapour is removed with a current of neutral gas, and the impregnated oxide is then chlorinated. The carbon monoxide produced may be used to heat the retort. Alternatively, the alumina may be impregnated with liquid or solid carbonaceous material before charging into the retort.

R. BRIGHTMAN.

Filtering material. H. BLUMENBERG, JUN. (U.S.P. 1,713,250—1, 14.5.29. Appl., 10.8.27).—The materials, which also serve as purifying, deodorising, and oxidising agents, consist of (A) aluminium sulphate, or (B) a soluble sulphate of an alkali-producing metal, mixed with calcium hypochlorite and incorporated with diatomaceous earth.

R. BRIGHTMAN.

Manufacture of filtration and decolorising absorbents. H. T. MAITLAND, Assr. to SUN OIL Co. (U.S.P. 1,711,504, 7.5.29. Appl., 6.2.24).—A solution of ammonium aluminium sulphate is gradually added to a water-glass or other silicate solution with agitation and boiling.

The product is washed from soluble salts and dehydrated at 260—535°. R. BRIGHTMAN.

Direct production of arsenic oxide. F. G. HILLS, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,713,127, 14.5.29. Appl., 10.11.20).—The arsenic-bearing material is mixed with carbonaceous matter and placed in a shallow layer on supports pervious to air. The carbonaceous matter is ignited in a current of low-pressure air and the arsenic is reduced and then oxidised to arsenious oxide, which is volatilised and collected substantially free from dust and zinc or lead oxides.

R. BRIGHTMAN.

Recovery of zinc [sulphate] from slags. H. H. MAYER and R. G. LA MOTTE (U.S.P. 1,727,073, 3.9.29. Appl., 10.8.26).—Slag containing substantial proportions of zinc and ferrous oxides is heated with sulphuric acid sufficient to form the corresponding sulphates, and the product is then further heated to decompose the ferrous sulphate. After cooling, the mass is leached to extract zinc sulphate. A. R. POWELL.

Manufacture of lead tetra-alkyl. H. W. DAUDT, A. E. PARMELEE, and K. P. MONROE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,717,961, 18.6.29. Appl., 17.10.25).—Sodium-lead alloy is treated with, *e.g.*, ethyl chloride in presence of less than 10% of ether and of 0.005—0.25% of ethyl alcohol.

R. BRIGHTMAN.

Production of hydrogen. F. C. BLAKE, Assr. to LAZOTE, INC. (U.S.P. 1,713,325, 14.5.29. Appl., 23.6.27).—Methane and steam in suitable preparations are caused to react in presence of a catalyst, *e.g.*, nickel-ceria-alumina, at 400—700°. Thus 10 vols. of steam at 508° give a 90% methane conversion and the dry gas contains 2% CO. R. BRIGHTMAN.

Sulphur-burning process. J. C. BOERTLEIN, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,713,416, 14.5.29. Appl., 4.10.24).—Molten sulphur is supplied as fuel to an internal-combustion engine, the air supply being previously dried; the exhaust gases, containing sulphur dioxide, after passing in heat exchange with a sulphur melting pot and through a device for removing unburned sulphur, are supplied, *e.g.*, to sulphuric acid plant after admixture with, *e.g.*, oxides of nitrogen.

R. BRIGHTMAN.

Base-exchange body. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,728,732, 17.9.29. Appl., 19.10.26).—See B.P. 279,466; B., 1928, 603.

Manufacture of colloidal compounds. A. CHWALA (U.S.P. 1,728,662, 17.9.29. Appl., 5.10.27. Austr., 30.3.27).—See B.P. 315,254; B., 1929, 814.

Purification of aluminium oxide. S. E. SIEURIN and A. S. EDLUNG (U.S.P. 1,726,001, 27.8.29. Appl., 27.4.26. Swed., 27.5.25).—See B.P. 252,693; B., 1927, 107.

Manufacture of titanium oxide. J. BLUMENFELD, Assr. to COMMERCIAL PIGMENTS CORP. (U.S.P. 1,707,248, 2.4.29. Appl., 8.6.26. Fr., 11.6.25).—See B.P. 253,550; B., 1927, 749.

Preparation of titanium hydroxide. J. BLUMENFELD, Assr. to COMMERCIAL PIGMENTS CORP. (Reissues 17,429—30, 10.9.29, of U.S.P. 1,504,672, 4.2.24).—See B., 1925, 991.

Utilisation of [solid] carbonic acid [carbon dioxide]. H. DEHOTTAY (U.S.P. 1,727,865, 10.9.29. Appl., 28.11.27. Belg., 9.4.27).—See B.P. 301,764; B., 1929, 95.

Preparation of fluorine. J. H. DE BOER, Assr. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,728,230, 17.9.29. Appl., 18.8.26. Holl., 28.10.25).—See B.P. 262,918; B., 1927, 105.

Apparatus for elevating acids and other [corrosive] liquids. W. H. EXLEY (B.P. 318,941, 12.6.28).

Fire-extinguishing media (B.P. 318,302).—See I. Organic compounds from oxides of carbon and hydrogen (B.P. 317,808).—See III. Colloidal silver chloride (B.P. 317,121).—See XX. Cleansing agent (B.P. 318,344).—See XXIII.

VIII.—GLASS; CERAMICS.

Glasses transparent to ultra-violet radiation. A. R. WOOD and M. N. LEATHWOOD (Nature, 1929, 124, 441—442).—When kept in the dark, glass solarised by the quartz-mercury lamp does not regain any of the lost transparency to ultra-violet radiation, but does so (a) when exposed to sunlight, (b) when covered with window glass and exposed to sunlight, or (c) when covered with a thick layer of window glass and then further exposed to the light of the mercury-vapour lamp. Hence the use of the mercury-vapour lamp to determine the loss of transparency of glass when exposed to sunlight gives misleading results.

A. A. ELDRIDGE.

Characterisation of bleaching clays. O. ECKART (Z. angew. Chem., 1929, 42, 939—941).—Clays suitable for decolorising and general adsorption are distinguished by the ease with which they lose water at temperatures up to 400°; under these conditions kaolin and ordinary clays lose very little water. The loss of 16—18% of the total alumina and ferric oxide content by boiling for 3 hrs. with 0.5*N*-hydrochloric acid is also characteristic.

S. I. LEVY.

See also A., Oct., 1138, Systems kaolin-alumina-felspar and kaolin-zirconia-felspar (SCHWARZ and others).

PATENTS.

Decolorising and fining glass. W. C. TAYLOR, Assr. to CORNING GLASS WORKS (U.S.P. 1,726,635, 3.9.29. Appl., 11.5.26. Renewed 4.8.28).—Glass containing iron is decolorised by the addition of cerium oxide, in amount equivalent to not more than 1.5% of the finished glass, and a small quantity of manganese dioxide. A. B. MANNING.

Manufacture of ceramic products. J. W. MELLOR, and CERAMIC PATENT HOLDINGS, LTD. (B.P. 318,188, 30.5.28).—About 2—4% of alumina or a mixture or compound of it with an alkaline-earth compound or magnesia is added as bleaching agent to the constituents of white pottery, tiles, etc., especially when made by the processes described in B.P. 253,184 or 302,519 (B., 1926, 667; 1929, 130). L. A. COLES.

Skimmer blocks for fore-hearths of glass-melting furnaces or other apparatus containing molten glass. BRIT. HARTFORD-FAIRMONT SYND., LTD., and T. WARDLEY (B.P. 318,852, 8.5.28).

Means for circulating glass in fore-hearths of glass-melting tanks. C. H. RANKIN, and RANKIN AUTOMATIC GLASS FEEDER Co. (B.P. 319,178, 6.2.29).

Process and apparatus for rolling plate glass. A. E. WHITE. From PITTSBURGH PLATE GLASS Co. (B.P. 319,182, 19.2.29).

Manufacture of refractory substances [from high-grade zirconia]. J. D'ANS, Assr. to DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (U.S.P. 1,728,748, 17.9.29. Appl., 24.12.25. Ger., 24.12.26).—See B.P. 245,101; B., 1926, 947.

IX.—BUILDING MATERIALS.

Effect of organic impurities on the mechanical properties of Portland cement. F. FEW (Chem. Eng. Min. Rev., 1929, 21, 441—443).—The deleterious effect of loams containing organic matter when introduced into a concrete mix has been studied, using tannic acid as an impurity in mixtures of cement and standard sand. All specimens containing 0.1—0.5% of tannic acid showed a weakening effect due to the impurity, especially as the quantity increased, and though results were not obtained for longer than a 28-day setting the effect is thought to be progressive with duration of time. Although compression tests also indicated a similar tendency, tests for longer intervals are required to establish definite results. C. A. KING.

PATENTS.

Manufacture of firebrick. E. P. STEVENS (U.S.P. 1,727,138, 3.9.29. Appl., 23.5.22).—A raw refractory clay is used, which, on artificially weathering, by applying moisture and heat and calcining, becomes granulated.

F. G. CLARKE.

Asphalt paving composition. J. S. DOWNARD (U.S.P. 1,727,231, 3.9.29. Appl., 30.7.26).—A fine mineral dust is agitated with melted asphalt and sufficient hot water to form a paste. On drying and crushing, a product results which remains granular under ordinary atmospheric conditions. F. G. CLARKE.

Apparatus for manufacture of cement by fusion. G. DUMAS, Assr. to SOC. ANON. DES CHAUX ET CEMENTS DE LAFARGE ET DU TEIL (U.S.P. 1,728,597, 17.9.29. Appl., 15.1.24. Fr., 29.9.23).—See B.P. 222,427; B., 1924, 1014.

Industrial manufacture of artificial Portland cement. J. A. HENDRICKX, Assr. to SOC. ANON. ETABL. POLIET & CHAUSSON (U.S.P. 1,728,828, 17.9.29. Appl., 18.5.25. Fr., 6.2.25).—See B.P. 237,182; B., 1925, 762.

Damp-proof paints (B.P. 318,358).—See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

On the road to pure iron and some of its indicated properties. T. D. YENSEN (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 9 pp.).—Progress in the elimination of impurities from iron is reviewed and the effect on its properties discussed. In unpublished work with Ziegler the oxygen content of electrolytic iron was reduced by melting in a zircon crucible in a vacuum furnace and carefully adding small amounts of carbon. Carbon does not combine appreciably with

the iron until oxygen has been eliminated, provided the carbon monoxide formed is removed by pumping off. The metal was kept molten until the pressure was reduced to 0.001 mm. After forging, machining, and annealing, permeabilities varying from 20,000 to over 60,000 were obtained, the carbon content being between 0.001 and 0.005%. It is concluded that progressive removal of oxygen and carbon will lead to still higher permeabilities and lower hysteresis values. The purer is the iron the less complex become its actions and behaviour due to external conditions.

H. J. T. ELLINGHAM.

Corrosion rate of ferro-nickel alloys. C. G. FINK and C. M. DECROLY (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 35 pp.).—The rate of corrosion of iron-nickel alloys in solutions of various composition and concentration under various conditions has been investigated, using an intermittent immersion test carried out in accordance with the recommendations of the American Society for Testing Materials. The details of the method are discussed. With all of the alloys examined the rate of corrosion in sulphuric acid solutions at first increased rapidly with increasing acid concentration, but after reaching a maximum decreased more or less rapidly again. Finally, for most of the alloys there was a second increase in rate of corrosion in very concentrated solutions. In a given solution of sulphuric acid content below 25% the rate of corrosion of various alloys increased continuously with increasing iron content of the alloy, but in a more concentrated acid solution the alloy containing 47.9% Ni always showed the minimum rate of corrosion. At the highest acid concentrations all the alloys were less readily attacked than was either pure nickel or pure iron. Addition of chlorate or chromate to the sulphuric acid did not produce any passivation, but actually accelerated the corrosion. The addition of less than 4% of chromium to the alloy did not improve the resistance to corrosion, but with nichrome containing 12.5% Cr there is a much lower rate of corrosion, especially in solutions of low acid concentration. Experiments were also made in sodium chloride solutions and the effect of varying the temperature was studied. The single potentials of iron-nickel alloys against sulphuric acid solutions of various concentrations were measured. In a given solution increasing iron content of the alloy makes it continuously baser, and for a given alloy the potential assumes increasingly base values with decrease in acid concentration. With pure nickel, however, an anomalous result was obtained. The theoretical interpretations of the observed rates of corrosion and potentials are discussed.

H. J. T. ELLINGHAM.

Accelerated corrosion tests for coatings of the iron phosphate type. E. M. BAKER, A. J. HERZIG, and R. M. PARKE (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 10 pp.).—Coils of soft steel wire were kept for 5 hrs. in solutions containing various concentrations of phosphoric acid and ferrous hydrogen phosphate maintained at 100°. The protective value of the coatings produced by this "coslettising" process was determined by the standard salt-spray test and by intermittent immersion tests using 20% salt solution,

5% salt solution, distilled water, or 0.01*N*-sulphuric acid. The last two tests proved to be the most sensitive, but the salt-spray test is far more rapid, and seems to be sufficiently reliable to warrant its being recommended for testing coatings of this type. It appears that a "coslettising" solution should be about 0.16*N* in phosphoric acid (reckoned as $\text{H}_3\text{PO}_4/3$), and that the concentration of the ferrous hydrogen phosphate is of less importance, but should lie between 0.00125 and 0.005*M*. These conditions might be modified under commercial conditions of coslettising, but independent observations indicate that they represent good practice.

H. J. T. ELLINGHAM.

Dissolution of various oxidised copper minerals.

J. D. SULLIVAN (U.S. Bur. Mines Rep. Invest., 1929, No. 2934, 9 pp.).—Practically all of the copper in azurite, malachite, chrysocolla, and tenorite is soluble in sulphuric acid or acidified ferric sulphate solutions; it is more slowly dissolved by neutral 1–2% ferric sulphate solution.

CHEMICAL ABSTRACTS.

Factors governing the entry of solutions into ores during leaching. J. D. SULLIVAN, W. E. KECK, and G. L. OLDRIGHT (U.S. Bur. Mines Tech. Paper, 1929, No. 441, 88 pp.).—The rate of ingress of solutions is determined by the solubility of the rock gases in the penetration liquid; also it diminishes with increase in the size of the particle. Fine crushing and displacement of the air in dry heaps by waste gases are recommended.

CHEMICAL ABSTRACTS.

Beneficiation of oxidised manganese ores by magnetic separation of roasted jig concentrates.

F. D. DE VANEY and W. H. COGHILL (U.S. Bur. Mines Rep. Invest., 1929, No. 2936, 4 pp.).—Some manganese-iron ores are amenable to magnetic separation after brief roasting. The phosphorus and insoluble matter are preferentially associated with the iron.

CHEMICAL ABSTRACTS.

High-temperature modification of manganese.

E. PERSSON and E. ÖHMAN (Nature, 1929, 124, 333–334).—Bradley's view (A., 1925, ii, 1124) that α - and β -manganese exist together in a state of isodynamic isomerism in the interval 650–850° is not supported. Although pure manganese, on quenching, gives only the β -modification, by alloying with a small quantity of other metals the existence of another high-temperature modification (γ -manganese) has been established. γ -Manganese is readily transformed into α -manganese by heating at 150°. By using copper alloys the γ -phase alone (with copper) or in equilibrium with the β -phase could be obtained. The transition point ($\gamma \rightarrow \beta$) is $1191 \pm 3^\circ$ (cf. Gayler, B., 1927, 444).

A. A. ELDRIDGE.

Thermo-electric tests for aluminium-manganese and other alloys. C. S. TAYLOR and J. D. EDWARDS (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 4 pp.).—The considerable thermo-*E.M.F.* between aluminium and the aluminium alloy containing 1% Mn provides a ready means of distinguishing the pure metal from the alloy. A simple test apparatus for this purpose is described. Its use for other metals and alloys is suggested.

H. J. T. ELLINGHAM.

Determination of calcium and magnesium in aluminium containing other alloying elements.

K. STEINHÄUSER (Z. anal. Chem., 1929, 78, 181–188).—The alloy (10 g.) is dissolved in 90 c.c. of 25% sodium hydroxide solution, the residue collected on a glass filtering crucible and dissolved in bromine and hydrochloric acid, and the solution boiled with an excess of sodium hydroxide solution, treated with 30 mg. of copper as sulphate, and subjected for 5 min. to a rapid current of air containing bromine vapour to oxidise the manganese to peroxide. Any permanganate formed is reduced by boiling with 1–2 c.c. of alcohol and the precipitate is collected in a glass filtering crucible and washed free from alumina with sodium hydroxide solution. Lime and magnesia are extracted from the precipitate by digesting it for 2 hrs. with 100 c.c. of water containing 4 c.c. of nitric acid, the filtered solution being evaporated with 20 c.c. of 1:1 sulphuric acid to remove silica and treated with ammonium acetate and oxalate to precipitate the lime. Magnesia is determined as usual in the filtrate.

A. R. POWELL.

Determination of small quantities of selenium in ores. E. T. ERICKSON (J. Wash. Acad. Sci., 1929, 19, 319–321).—The ore (25 g.) is ground with 60 g. of zinc oxide and 20 g. of sodium carbonate and the mixture added to a similar quantity of the same reagents. The whole is roasted with occasional rabbling for 1 hr. at 700–750° and, when glowing has ceased, for a further 1 hr. at 800°. The cold mass is repeatedly extracted with water until about 900 c.c. of clear solution are obtained. The solution is acidified and evaporated to a pasty mass of crystals, which is extracted twice with 25 c.c. of hydrochloric acid, *d* 1.19. The filtered acid liquor is saturated with sulphur dioxide to precipitate selenium. If only traces are present a little silica gel is added to assist in its collection; the colour of the mass may be used as a guide to the quantity of selenium present, using standards prepared in the same way.

A. R. POWELL.

Combustion temperatures of sulphur, pyrites, and zinc blende. J. S. DOTING (Chem.-Ztg., 1929, 53, 737–739).—The approximate combustion temperatures under industrial conditions, calculated from the heats of combustion and the heat capacities of the reaction products, are for sulphur, pyrites, and zinc blende, respectively: 1025°, 965°, and 1050°.

F. G. TRYHORN.

Atmospheric corrosion of metals. Third (experimental) report to the Atmospheric Corrosion Research Committee (British Non-Ferrous Metals Research Association). J. C. HUDSON (Trans. Faraday Soc., 1929, 25, 475–496).—Additional data and a further discussion of the report are given (cf. B., 1929, 684).

F. J. WILKINS.

Determination of sulphate in chromium-plating baths. WILLARD and SCHNEIDEWIND.—See VII. Aluminium conductors and corrosion. WILSON.—See XI.

See also A., Oct., 1130, **X-Ray investigation of iron and zinc alloys** (OSAWA and OGAWA). 1145, **System iron-oxygen** (SCHENCK and others). 1151, **Deposition of zinc from cyanide solutions** (PINES). 1157,

Recovery of platinum (HOUGH). 1159, **Permanganate titration of antimony in white metal (VASILIEV and STUTZER).** 1160, **Volumetric determination of tin (WOLF and HEILINGÖTTER).**

PATENTS.

Flotation concentration [of ores]. S. P. LOWE, Assr. to R. H. CHANNING (U.S.P. 1,728,352—3, 17.9.29. Appl., 26.3.28).—(A) Sulphide ores containing copper, zinc, and iron sulphides are subjected to a flotation treatment in a pulp the alkalinity of which corresponds with 0.01—0.15 lb. of lime per ton of water, whereby the copper mineral alone is floated. The alkalinity is then adjusted to 0.4—0.7 lb. of lime per ton of water and the zinc sulphide floated, leaving a tailing containing pyrites. (B) Zinc oxide is added to the pulp in addition to the usual reagents.

A. R. POWELL.

[Cast-iron alloys.] A. F. HILTON, Assr. to FARREL-BIRMINGHAM Co., Inc. (U.S.P. 1,729,386—7, 24.9.29. Appl., [A] 31.1.28, [B] 1.8.29).—Iron alloys containing above 2.25% C, 0.25—2% Cu, and 0.10—2% Mo as essential elements are claimed, with the addition, in (A) of up to 3.5% Cr.

F. G. CROSSE.

[Silicon-iron alloy.] J. A. PARSONS, JUN., Assr. to DURIRON Co., Inc. (U.S.P. 1,728,360, 17.9.29. Appl., 10.1.29).—The alloy comprises iron with 9—20% Si, 1—3% Ni, and 0.1—10% W, V, or Mo in the form of carbide.

A. R. POWELL.

Manufacture of ferrosilicon. T. F. BAILY (U.S.P. 1,727,193, 3.9.29. Appl., 19.7.27).—Silica is reduced with carbon to form molten silicon, which is then mixed in the desired proportion with molten iron containing carbon.

A. R. POWELL.

Manufacture of protected metal. F. M. CRAPO, Assr. to INDIANA STEEL & WIRE Co. (U.S.P. 1,726,652, 3.9.29. Appl., 25.3.25).—Iron or low-carbon steel material, e.g., wire, after being heated in order to anneal it, is immersed in a carbonising bath of molten salts and then coated with zinc.

F. G. CLARKE.

Production of alloy-surface castings. R. WILLIAMS, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,725,039, 20.8.29. Appl., 13.2.26).—The casting mould is coated with a fabric impregnated with powdered chromium and a reducing agent which promotes alloying of the chromium with the surface of the casting.

A. R. POWELL.

Copper-cobalt alloy. M. G. CORSON, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,723,922, 6.8.29. Appl., 13.4.26).—In order to strengthen it, copper is alloyed with 2—5% of cobalt, heated to above 850°, and quenched.

F. G. CLARKE.

Utilisation of light-metal scrap. H. CHAMBERLAIN (U.S.P. 1,725,780, 27.8.29. Appl., 4.4.27).—Scrap sheet metal mixed with finely-divided carbon is pressed into bales which are heated at 1200° and subjected to high pressure to form solid bodies of metal.

A. R. POWELL.

Separating metallic [zinc-lead sulphide] ores [by flotation]. R. W. LOYD and B. W. MOYE (U.S.P. 1,727,472, 10.9.29. Appl., 12.12.27).—The ores are

ground with sodium chloride and zinc sulphate, water is added, and the mixture subjected to flotation.

A. R. POWELL.

Recovery of cadmium [from lead-bearing material]. R. TEATS, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,727,492, 10.9.29. Appl., 20.3.26).—The material is ground with limestone and a carbonaceous reducing agent and heated at 825—850° to volatilise the cadmium without appreciable volatilisation of lead. The fume is allowed to oxidise and the cadmium oxide collected.

A. R. POWELL.

Lead refining. B. W. GONSER and C. W. HAFLEY, Assrs. to AMER. SMELTING & REFINING Co. (U.S.P. 1,726,346, 27.8.29. Appl., 15.2.28).—Lead rich in antimony is heated at a bright red heat under alternately oxidising and reducing conditions to effect complete volatilisation of the antimony as trioxide.

A. R. POWELL.

Inhibitor [for use in pickling metals]. J. G. DAVIDSON, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,723,923, 6.8.29. Appl., 14.3.28).—A hydroxy-alkylamine is added to the bath.

F. G. CLARKE.

[Electro]plating with chromium. R. GRAH (B.P. 317,110, 22.6.28).—Turning tools and metal articles to be surface-hardened without heat are electroplated in a bath at 35° containing 50 pts. of chromic acid, 50 pts. of water, and 0.05 pt. of chromium sulphate. A current density of 0.03—0.04 amp./cm.² is used.

H. ROYAL-DAWSON.

Metallurgical furnace. B. TALBOT (U.S.P. 1,729,230, 24.9.29. Appl., 19.12.27. Belg., 30.6.27).—See B.P. 293,010; B., 1929, 522.

Purification of gases from blast furnaces. W. and H. MATHESIUS (U.S.P. 1,728,130, 10.9.29. Appl., 30.12.25. Ger., 3.1.25).—See B.P. 255,344; B., 1926, 862.

Magnetic [iron] alloy. W. S. SMITH, II, J. GARNETT, and J. A. HOLDEN (U.S.P. 1,728,451, 17.9.29. Appl., 3.5.28. U.K., 13.5.27).—See B.P. 295,443; B., 1928, 756.

Making tools from hard-metal alloys produced by sintering. K. SCHRÖTER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,728,909, 17.9.29. Appl., 23.7.26. Ger., 12.12.25).—See B.P. 262,723; B., 1927, 658.

Alloy-steel building material. W. HÜLSBRUCH, Assr. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,727,775, 10.9.29. Appl., 13.5.27. Ger., 22.5.26).—See B.P. 271,470; B., 1928, 644.

Manufacture of articles [iron and steel alloys] for which a resistability against the noxious effect of aged state is required. A. FRY, Assr. to F. KRUPP A.-G. (U.S.P. 1,729,378, 24.9.29. Appl., 8.9.26. Ger., 3.4.26).—See B.P. 268,716; B., 1928, 197.

Material for use in protecting iron and steel. W. H. COLE (U.S.P. 1,729,065, 24.9.29. Appl., 21.4.27. U.K., 19.3.27).—See B.P. 292,666; B., 1928, 608.

Treatment of sulphide ores or sulphidic metallurgical products. F. JOHANNSEN, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,728,681, 17.9.29. Appl., 28.5.25. Ger., 30.5.24).—See B.P. 234,826; B., 1925, 676.

Treatment of copper slag. S. YOSHIMOTO, Assr. to T. OKUMURA and S. NAKAMURA (U.S.P. 1,728,095,

10.9.29. Appl., 6.7.26. Jap., 2.6.26).—See F.P. 620,456; B., 1929, 131.

[Copper-silicon] alloy and method of working and heat-treating the same. M. G. CORSON, Asst. to ELECTRO METALLURGICAL Co. (U.S.P. 1,729,208, 24.9.29. Appl., 4.10.26).—See B.P. 278,355; B., 1928, 527.

Reduction of zinc ores. H. WITTEK (U.S.P. 1,728,094, 10.9.29. Appl., 1.12.26. Ger., 23.10.26).—See B.P. 279,370; B., 1928, 128.

Electromagnetic separation or concentration of minerals. W. M. MORDEY (U.S.P. 1,729,589, 24.9.29. Appl., 8.5.24. U.K., 17.5.23).—See B.P. 224,924; B., 1925, 76.

Apparatus for electrolytic production of metallic powders. M. A. ADAM (U.S.P. 1,728,989, 24.9.29. Appl., 31.12.25. U.K., 1.1.25).—See B.P. 253,958; B., 1926, 757.

Dust from reduction furnaces (B.P. 292,931).—See II. Xanthate mixture (U.S.P. 1,716,273).—See III. Mercury from rubber waste (U.S.P. 1,728,359).—See XIV.

XI.—ELECTROTECHNICS.

Corona on aluminium conductors as affected by corrosion due to atmospheric exposure. E. WILSON (Trans. Faraday Soc., 1929, 25, 496—502).—A study has been made of the influence of atmospheric corrosion on corona production. The results indicate that the aluminium conductors used became severely pitted by corrosion. A conductor of an aluminium-nickel alloy exposed to the atmosphere for 24 years possessed a surface practically free from corrosion. The discharge from the surface of such a conductor is of the same character as that given by a new wire, so that in so far as corona losses are concerned they will behave similarly. F. J. WILKINS.

Turbidimetric method for determination of the sulphate content of chromium-plating baths. L. E. STOUT and A. W. PETCHAFT (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 11 pp.).—A sample of the chromium-plating solution is diluted to a tenth of its concentration and treated with excess of strontium chloride. The precipitate formed is less dense than that given by barium chloride, and by working under controlled conditions the sulphate content of the original solution can be determined from the turbidity as measured in a Parr sulphur-photometer. A calibration chart is drawn up to convert photometer readings into sulphate concentrations for solutions containing varying amounts of chromic acid. This chromic acid concentration can be determined with sufficient accuracy from the density of the original solution as found by means of a hydrometer. The method has been tested on solutions made up to known sulphate concentration, and on actual plating baths, and is shown to give results of an accuracy comparable with that given by the gravimetric method of the Bureau of Standards. H. J. T. ELLINGHAM.

Oxidation of anthracene to anthraquinone. RASCH and LOWY.—See III. Thermo-electric tests for alloys. TAYLOR and EDWARDS.—See X.

See also A., Oct., 1135, Superconductors (DE HAAS and others). 1147, Polarisation and hysteresis in

standard cells (NIEDERHAUSER and HULETT). 1151, Activation of sulphur (SCHWARTZ and SCHENK). Deposition of zinc from cyanide solutions (PINES). Preparation of permanganates (RAPIN). Electrolytic preparation of chlorate (PAMFILOV and FEDOROVA).

PATENTS.

Electric furnace. E. BORNAND and H. A. SCHLÄPFER (B.P. 301,912, 3.12.28. Switz., 8.12.27).—One or more vertically movable electrodes are enclosed in electrically insulating refractory material, so as to form a block leaving only the end faces of the electrodes uncovered. The block is inserted into the molten metal bath in the furnace chamber, whereby slag or other material floating on the bath is caused to react within the bath. J. S. G. THOMAS.

Electric furnace. C. I. HAYES (U.S.P. 1,724,583, 13.8.29. Appl., 12.5.28).—Metal to be treated in a closed chamber is introduced and, after treatment, withdrawn through an opening across which a stream of burnt gases is projected under pressure in order to exclude air from the chamber. J. S. G. THOMAS.

Charging of electrical furnaces. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 317,416, 14.5.28).—Segregation of the charge is avoided by delivering it continuously to the electrodes, without its coming into contact with the walls of the furnace, over adjustable guide surfaces arranged above the melting or reaction zone and sloping towards the electrodes. J. S. G. THOMAS.

Electrodes for electric furnaces. SOC. ELECTRO-MÉTALL. DE MONTRICHER (B.P. 308,750, 14.6.28. Fr., 31.3.28).—Hollow metal sections of the electrodes are filled with coal mixed, if desired, with carbon, and the filling is carbonised at 500—600°. J. S. G. THOMAS.

Electric thermometer. D. O. GHADIALI (U.S.P. 1,724,469, 13.8.29. Appl., 20.7.26).—A variable resistance is arranged on a dielectric filling piece near the open end of a dielectric case enclosing a metallic tube, the whole forming a variable resistance unit contained in a heating chamber provided with a thermostat, and inserted in a Wheatstone bridge. J. S. G. THOMAS.

Impregnating high-tension electric cables. FELTEN & GUILLEAUME CARLSWERK A.-G. (B.P. 291,722, 5.6.28. Ger., 3.6.27).—Cables are impregnated with oils, resins, &c., which during the process of impregnation or previous storage are kept in closed vessels, the upper part of which contains a chemically inert gas, more especially nitrogen, in order to prevent the incorporation of oxidised impregnating substances. [Stat. ref.] J. S. G. THOMAS.

Manufacture of insulated articles for electrical engineering purposes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,412, 15.10.28).—Vegetable, animal, mineral, or other materials are treated with metal salts of naphthenic acids which are difficultly soluble in water, together with, if desired, oils, resins, gums, rubber, or bituminous materials. J. S. G. THOMAS.

Luminous electric discharge tube. R. FRANCOTTE (U.S.P. 1,723,929, 6.8.29. Appl., 2.10.26. Belg., 9.3.26).—Electrodes of spirally-rolled sheets of metal of

high m.p. are exposed to electric discharges in a separate tube filled with nitrogen, in order to free them from occluded gases, and are then mounted in parallel in small branch tubes. The latter are fused into one end of a discharge tube, and again purified by means of electric discharges in a nitrogen atmosphere. Residual gases are absorbed by an auxiliary palladium electrode, and the tube is finally filled with a purified rare gas.

F. G. CLARKE.

Manufacture of [cathodes for] electric discharge devices. GEN. ELECTRIC CO., LTD. From A. JUST (B.P. 317,504, 18.4.28).—The surface of a core of iron, preferably pure iron, or of a refractory metal, e.g., molybdenum coated with iron, is coated or alloyed with one or more alkali, alkaline-earth, or rare-earth metals by heating *in vacuo* or in a non-oxidising atmosphere containing the vapours of these metals.

J. S. G. THOMAS.

Electron emitter. H. T. REEVE, ASST. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,719,128, 2.7.29. Appl., 26.8.24).—"Nickelites" of barium and strontium are intimately mixed with platinum powder to form a core, which latter is then heated to effect reduction of the nickelites. The resulting product is converted into filaments.

D. J. NORMAN.

Rectifier electrode. C. W. BALKE, ASST. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,724,065, 13.8.29. Appl., 14.1.28).—An electrode composed of tantalum-tungsten alloy (90% Ta) and a non-film-forming electrode are immersed in an electrolyte (sulphuric acid).

J. S. G. THOMAS.

Manufacture of lead or lead peroxide electrodes for accumulators. I. G. FARBERIND, A.-G. (B.P. 291,413, 1.6.28. Ger., 1.6.27).—A core of lead or hard lead is embedded in a homogeneous lead alloy, and the admixed metal is removed from the composite bar, preferably by electrolysis.

J. S. G. THOMAS.

Photo-electric plate. J. E. POLLACK. From ASSOCIATED TELEPHONE & TELEGRAPH CO. (B.P. 318,299, 1.6.28).—One face of an insulating slab is coated with material, e.g., selenium or potassium hydride, which becomes conducting under the action of cathode rays; the other has a coating of material, e.g., selenium, the conductivity of which varies with the intensity of the incident light.

J. S. G. THOMAS.

Electroplating apparatus. W. J. O'NEILL, ASST. to E. J. MERCIL (U.S.P. 1,726,308, 27.8.29. Appl., 7.3.27).—A horizontal barrel comprises perforated panels which are assembled upon bars of insulating material mounted between two end walls. Terminals project laterally into the drum from conductors embedded in each bar. Screws, which hold the end walls and bars together, also connect the conductors with additional conductors embedded in one of the end walls.

F. G. CLARKE.

Electrode for electric furnaces. P. L. J. MIGUET, ASST. to Soc. ELECTROMÉTALLURG. DE MONTRICHER (U.S.P. 1,728,071, 10.9.29. Appl., 23.12.27. Fr., 13.10.27).—See B.P. 294,837; B., 1928, 716.

Battery depolariser. B. K. BROWN, ASST. to C. F. BURGESS LABS., INC. (U.S.P. 1,729,416, 24.9.29. Appl., 7.7.25).—See B.P. 255,067; B., 1927, 786.

Selenium cell. J. NEALE (U.S.P. 1,728,073, 10.9.29. Appl., 6.6.28. U.K., 6.8.27).—See B.P. 284,942; B., 1928, 272.

Manufacture of X-ray anodes. BRIT. THOMSON-HOUSTON CO., LTD., and E. G. GILSON (B.P. 293,864, 7.7.28. U.S., 15.7.27).

Chemical reactions in gases (B.P. 317,558 and 317,920). **Oxidation of hydrocarbons** (U.S.P. 1,710,155). **Distillation of tar** (B.P. 289,832). **Low-boiling hydrocarbons** (B.P. 318,270).—See II. **Chromium plating** (B.P. 317,110).—See X. **Anti-rachitic products** (B.P. 318,269).—See XX.

XII.—FATS; OILS; WAXES.

Chia seed oil. W. F. BAUGHMAN and G. S. JAMIESON (Oil & Fat Ind., 1929, 6, [9], 15—17).—An expressed oil from chia seed (*Salvia hispanica*, L.) had: d_{20}^{25} 0.9358, n_D^{20} 1.4838, acid value 1.4, saponif. value 194.8, unsaponif. matter 0.7%, iodine value (Hanus) 190.0, thiocyanogen-iodine value 113.2, hexabromide value of fatty acids 51.2, saturated acids 8.1% (corr.), unsaturated acids (iodine value 214.6) 85.2% (corr.), loss in weight when heated for 3 hrs. at 110° in carbon dioxide 0.3%. The content of unsaturated acids was deduced from the iodine and thiocyanogen values; from the hexabromide value the total linolenic acid (39.3%) is considered to consist of 17.6% of α -linolenic acid and 21.7% of the β -acid. The saturated acids were separated from the oil by the lead salt-ether method and fractionated as their methyl esters. The oil was found to consist of the glycerides of the following acids: linolenic 41.1, linoleic 47.2, oleic 0.7, myristic 0.1, palmitic 5.1, stearic 2.9, arachidic 0.3%, and unsaponif. matter 0.7%. The drying power, therefore, should be somewhat superior to that of linseed oil. (Cf. Gardner and Holdt, B., 1920, 789 A.) E. LEWKOWITSCH.

Determining [fatty oil] bleaching-loss coefficients. A. S. RICHARDSON, J. T. R. ANDREWS, and R. G. FOLZENLOGEN (Oil & Fat Ind., 1929, 6, [9], 19—20).—A laboratory method for determining oil retention by bleaching earths and carbons is described. The increase in weight is measured of a sample of the material which has been treated with oil, the excess being removed by suction in a stream of hot inert gas under standardised conditions. The test requires less than 10 g. of material for duplicate determinations and gives reproducible results, the oil-retention values obtained falling within the range of those observed in practical plant operation.

E. LEWKOWITSCH.

Test for neutral oil in soap or fatty acids. J. E. RUTZLER, JUN. (Oil & Fat Ind., 1929, 6, [9], 23—24, 29).—The ammonia-emulsion test has been standardised for quantitative use. Since it is found that fatty acids containing very small quantities only of unsaponifiable matter fail to show turbidity when the neutral oil content is below 1%, definite amounts of white mineral oil are added to such fatty acids to increase the sensitivity of the test. Thus 6 c.c. of the fatty acids (at 25°) are taken, 4 drops of white oil are added, the volume is made up to 36 c.c. with 96% alcohol, 30 c.c. of concentrated ammonia solution are stirred in, and the turbidity produced is measured in a simple form of

emulsometer. It is important that emulsometer readings should be taken within a standard time ($4\frac{1}{2}$ min.) of making the emulsion, as the opacity of the mixtures varies with age. The proportion of neutral oil is estimated by a comparison with readings obtained from known mixtures of fatty acids and oil under the same conditions. E. LEWKOWITSCH.

Determination of fat in wool. KRAIS and BILTZ.—See V.

See also A., Oct., 1202, **Assay of vitamin-A in fish oils** (DRUMMOND and MORTON; NORRIS and DANIELSON). 1204, **Transformation of reserve fat of seeds during germination** (LEMARCHANDS).

PATENTS.

Manufacture of emulsive preparations. A. N. MACNICOL (U.S.P. 1,711,503, 7.5.29. Appl., 21.6.23. Austral., 7.7.22).—Wool fat is heated with caustic alkali and emulsified with an alkali arsenite, 1–3% of gelatin, casein, or starch, etc. being added as protective colloid.

R. BRIGHTMAN.

Processing of [castor] oils. L. J. REIZENSTEIN (U.S.P. 1,715,785, 4.6.29. Appl., 27.12.24).—Castor oil is rendered soluble in mineral oil by heating at 150–200° with about 5% of aluminium sulphate, chloride, or oxide, stannous sulphate, thorium oxide or chloride.

R. BRIGHTMAN.

Treatment of [vegetable] oils. A. S. MORRIS, Assr. to CRESSON-MORRIS Co. (U.S.P. 1,714,231, 21.5.29. Appl., 23.12.22).—Vegetable oil is heated with sodium hydroxide solution in a kettle, a part of the charge being continuously circulated from the bottom of the kettle through a centrifugal pump and returned against a deflector plate covering the opening at the bottom of the kettle to agitate the charge. The treated oil is finally centrifuged. A continuous process is described.

R. BRIGHTMAN.

Manufacture of soap. A. E. HATFIELD and E. A. ALLIOTT (U.S.P. 1,728,342, 17.9.29. Appl., 11.6.27. U.K., 15.2.27).—See B.P. 289,582; B., 1928, 492.

Solid paraffin from oils (U.S.P. 1,724,731).—See II. **Antirachitic products** (B.P. 318,268–9).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

See A., Oct., 1204, “Minjak pelandjau” exudation from wood of *Pentaspadon Motleyi* (VAN ROMBURGH and VAN VEEN).

PATENTS.

Phenol resin moulding mixture. L. V. REDMAN and V. H. TURKINGTON, Assr. to BAKELITE CORP. (U.S.P. 1,716,665, 11.6.29. Appl., 1.5.23).—A mixture of 25–40% of infusible phenolic resin, 50% or more of fibrous filler, and the balance furfuraldehyde (or other high-boiling aldehyde) or nitrogen derivatives thereof as solvent for the resin is claimed. R. BRIGHTMAN.

Production of phenolic condensation products. O. A. CHERRY and F. KURATH, Assrs. to ECONOMY FUSE & MANUF. Co. (U.S.P. 1,726,650, 3.9.29. Appl., 21.4.26).—A mixture containing phenol, furfuraldehyde, a substance which liberates ammonia, and sufficient reactive substance containing methylene to give an infusible product, is heated. F. G. CLARKE.

Manufacture of [resinous basic] condensation products [from arylamines and acetylene]. Soc. CHEM. IND. IN BASLE (B.P. 292,168, 15.6.28. Switz., 16.6.27).—An aromatic amine or its salt is stirred with mercuric chloride with or without an organic diluent (benzene) in an atmosphere of acetylene. Acid is added and, after removal of diluent and mercury sludge, excess arylamine is distilled off from alkali in steam, leaving an acid-soluble resin useful for alkali-stable varnishes, and as wetting-agents, reserve substances, assistants to vulcanisation, etc. C. HOLLINS.

Manufacture of transparent, hard, insoluble, and infusible condensation products from phenols and aldehydes. L. DEUTSCH and I. THORN, Assrs. to SELDEN Co. (U.S.P. 1,710,019 and 1,710,045, 23.4.29. Appl., [A] 15.9.23, Yugoslav., 28.9.22; [B] 2.3.28, Austr., 30.11.22).—See B.P. 207,791–2; B., 1925, 216.

Manufacture of phthalic glyceride resin. H. C. P. WEBER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,715,688, 4.6.29. Appl., 10.6.25).—See B.P. 253,519; B., 1927, 85.

(A) Resin from cashew nut-shell oil. (B, D) Cashew nut-shell oil reaction product. (C) Substitute for shellac etc. M. T. HARVEY, Assr. to HARVEL CORP. (U.S.P. 1,725,791–4, 27.8.29. Appl., [A] 17.6.25, [B] 16.10.25, [C] 8.6.26, and [D] 6.8.26).—See B.P. 283,803, 259,959, 272,510, and 275,574; B., 1928, 237, 376, 902; 1929, 179.

Damp-proof paints for walls. F. S. PLANT (B.P. 318,358, 31.7.28).

Subdividing resins (U.S.P. 1,711,482).—See II. **Porous charcoal** (U.S.P. 1,716,606).—See III. **Vulcanised fibre** (B.P. 316,700).—See V. **Projectile fuses** (B.P. 298,948).—See XXII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Preservation of manufactured rubber and softening of hardened goods. BRUÈRE (J. Pharm. Chim., 1929, [viii], 10, 196–199).—Treatment with cold or hot (115°) liquid vaseline is recommended for restoring softness and pliability to rubber goods which have become hard in storage. D. F. TWISS.

Surface vulcanisation in ultra-violet light. R. DITMAR and O. GRÜNFELD (Gummi-Ztg., 1929, 44, 2801–2803, 2859–2861).—Ultra-violet light accelerates vulcanisation of rubber films by activating the sulphur which is present; the use of organic accelerators and inorganic activators for vulcanisation under such conditions is superfluous. The nature of the supporting material influences the rate of the vulcanisation, largely by its thermal properties. As vulcanisation is effected in such short periods as 4–8 min. between 20° and 90°, brightly coloured products can be obtained.

D. F. TWISS.

PATENTS.

Manufacture of artificial rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,115, 26.4.28).—The polymerisation of diolefines emulsified with suitable liquids containing, e.g., albumin or other colloids and buffer mixtures is expedited, and the products are

improved, by the presence of hydrogen peroxide in loose combination with an organic substance, *e.g.*, urea, or with potassium fluoride. D. F. TWISS.

Reclamation of vulcanised rubber. T. YOKOYAMA (U.S.P. 1,714,835, 28.5.29. Appl., 15.7.27. Jap., 17.7.26).—Pulverised vulcanised rubber is dissolved in decalin, tetralin, or other hydrogenated naphthalene solvent for rubber and sulphur, *e.g.*, at 135–140°, in presence of a vulcanisation accelerator, *e.g.*, sodium ethoxide, which readily reacts with sulphur, and the solution is poured into water. R. BRIGHTMAN.

Recovering volatile metals [mercury from rubber waste]. B. ORMONT, Assr. to KEYSTONE CHEM. & MANUF. CO. (U.S.P. 1,728,359, 17.9.29. Appl., 1.8.27).—The material is heated to melt the rubber and to volatilise the mercury which is contaminated with rubber. The first condensate is again distilled and the resulting product washed with a solvent for rubber.

A. R. POWELL.

Xanthate mixture (U.S.P. 1,716,273).—See III. **Leather substitute** (B.P. 295,387).—See VI. **Resinous condensation products** (B.P. 292,168).—See XIII.

XV.—LEATHER; GLUE.

See A., Oct., 1143, **Swelling of gelatin in aqueous solutions** (KÜNTZEL; LLOYD). 1157, **Colorimetric determination of p_H in coloured solutions** (THOMPSON and ATKIN).

PATENTS.

Manufacture of synthetic tanning agents. I. G. FARBENIND. A.-G. (B.P. 297,830, 19.9.28. Ger., 30.9.27).—An alkali salt of a sulphurised phenol solubilised by treatment with formaldehyde and a sulphite or bisulphite is mixed in solid form with the requisite amount of a solid acid or acid-generating substance, *e.g.*, oxalic acid, potassium hydrogen sulphate, or naphthalene- β -sulphonic acid. The products on dissolution in water give a tanning liquor ready for use. C. HOLLINS.

Mineral tanning process. O. RÖHM (B.P. 292,501, 6.6.28. Ger., 20.6.27).—One or more of the salts of chromium, aluminium, or iron together with orthophosphoric acid, or its salts, esters, or salts of its esters, *e.g.*, glycerophosphoric acid, are used for tanning.

D. WOODROFFE.

Tanning. F. S. Low (U.S.P. 1,723,568, 6.8.29. Appl., 13.8.25).—A mixture of anhydrous chromic chloride with a metal which effects reduction is used.

F. G. CLARKE.

Manufacture of water-resistant adhesives. C. N. CONE, G. DAVIDSON, and I. F. LAUCKS, Assrs. to I. F. LAUCKS, INC. (U.S.P. 1,726,510, 27.8.29. Appl., 3.1.28).—A halogenated paraffin hydrocarbon is added to a dispersed vegetable protein, *e.g.*, soya-bean protein.

F. G. CLARKE.

Water-soluble, mineral tanning agent. G. KRÄNZLEIN, A. VOSS, and F. BRUNNTRÄGER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,727,719, 10.9.29. Appl., 13.4.26. Ger., 24.4.25).—See B.P. 251,627; B., 1927, 440.

Manufacture of leather substitutes. R. CHAVEL (U.S.P. 1,727,862, 10.9.29. Appl., 25.7.25. Fr., 30.5.25).—See F.P. 611,401; B., 1927, 362.

Viscose products (B.P. 301,305).—See V. **Leather substitute** (B.P. 295,387).—See VI.

XVI.—AGRICULTURE.

Laws of soil colloidal behaviour. I. S. MATTSON (Soil Sci., 1929, 28, 179–220).—The relationships between the mineral soil colloids, the solution permeating these colloids, and that surrounding the gel structure are theoretically discussed and experimentally examined. Ions which neither combine with nor are positively adsorbed by the colloids are negatively adsorbed to an extent dependent on the nature of the adsorbed bases. The swelling, viscosity, and potential of the gels in the absence of free electrolytes is governed by the degree of dissociation of the exchangeable cations, and is suppressed by electrolytes according to the valency of their ions. The dissociation of adsorbed bases depends on certain specific properties of the ions, notably hydration and potential differences, as well as on their valency. The essential difference is emphasised between the free soil solution and that existing as an integral part of the gel structure. The latter cannot be removed by pressure or by other means now adopted for the isolation of soil solutions. A. G. POLLARD.

Colloidal properties of Willamette valley soils. R. E. STEPHENSON (Soil Sci., 1929, 28, 235–247).—Soils were examined in horizons for iron, aluminium, lime, organic matter, exchangeable calcium, and p_H value. The separated colloids showed higher retentive capacities for phosphates than the whole soils. The exchangeable calcium of the colloids was insufficient to correspond with the total phosphate retained. A. G. POLLARD.

Behaviour of Kosseir's phosphate in neutral soils. F. SCURTI and G. PIANO (Annali Chim. Appl., 1929, 19, 283–296).—When in granules of less diameter than 0.165 mm., Kosseir's phosphate serves to replace superphosphates not merely for acid soils, but also for neutral soils. T. H. POPE.

Behaviour of Kosseir's phosphate in arable soil. G. PIANO (Annali Chim. Appl., 1929, 19, 343–370).—When used as a fertiliser for wheat, rye, maize, gladioli, and meadowland, Kosseir's phosphate in granules not exceeding 0.165 mm. in size is as effective as superphosphate on acid or neutral soils. T. H. POPE.

Composition of natural organic materials and their decomposition in the soil. IV. **Nature and rapidity of decomposition of the various organic complexes in different plant materials under aerobic conditions.** F. G. TENNEY and S. A. WAKSMAN (Soil Sci., 1929, 28, 55–84; cf. B., 1927, 951; 1928, 62).—The chemical composition of various plant materials largely determines the manner and rate of their decomposition in soils. Sugars, cellulose, hemicellulose, fats, and proteins are the chief constituents concerned. Lignins are more resistant, but suffer slow decomposition under aerobic conditions. During the decomposition of materials of low nitrogen content (0.2–1.7%) a definite increase in crude protein occurs due to the

synthesising activities of the micro-organisms. Available inorganic nitrogen accelerates the decomposition of cellulose and hemicellulose. After decomposition for 12–14 months under optimum conditions in the laboratory the residues of plant materials possess all the properties of soil humus. The latter consists of lignin and modified lignins of plant origin, proteins and complex nitrogen compounds of micro-biological origin, together with other matter in the process of decomposition. Humus is not a stable material, but undergoes continuous but slow changes. A. G. POLLARD.

Relation of water-soluble, replaceable, and acid-soluble potash to the potash removed by crops in pot experiments. G. S. FRAPS (Texas Agric. Exp. Sta. Bull., 1929, No. 391, 18 pp.).—The amount of potassium taken up by two crops is usually 5–6 times that of the water-soluble, and half the replaceable, potassium in the soil. The amount of potassium removed by the crops from the soil is usually 8–16 times that of the water-soluble, and about twice that of the replaceable, potassium lost in cropping. The difference in the amounts of potassium removed by a first and a second extraction of soil with 0.2*N*-nitric acid increases as the potassium taken up by the crops becomes greater. The amount of acid used is also of significance.

CHEMICAL ABSTRACTS.

Percolation experiments. I. Nitrification and effect of cover plants. W. N. C. BELGRAVE (Malayan Agric. J., 1929, 17, 192–205).—Comparison is made of the nitrogen losses in fallowed, fertilised, and cropped soils. Fallowing considerably increased the nitrate content of the soil percolates, whilst cover crops, *e.g.*, grass, reduced the value almost to zero. The acidity of the percolates increased with their nitrate content, and *vice versa*. A. G. POLLARD.

Rapid approximate method of determining the exchangeable bases in non-calcareous soils. W. N. C. BELGRAVE (Malayan Agric. J., 1929, 17, 206–208).—Kappen's method (B., 1929, 487) for determining exchangeable bases in soils by extracting with hydrochloric acid and titrating the extract with alkali is unsatisfactory, in that some chloride ion is adsorbed, and in certain soils the extract becomes coloured with iron and organic matter, yielding falsely high values. In the method recommended the soil (5–20 g.) is warmed at 50° with 100 c.c. of *M*/60-aluminium chloride solution and, after filtering, 50 c.c. are titrated with 0.1*N*-sodium hydroxide, using phenol-red as indicator. If the base content is less than 3 milliequivalents-%, the chloride content of the aliquot is titrated with 0.1*N*-silver nitrate, using potassium chromate as indicator, and the absorption found is deducted from the apparent base content. A. G. POLLARD.

Application of the antimony electrode to the determination of the p_H value and lime requirement of soils. W. H. HARRISON and P. N. VRIDHACHALAM (Mem. Dept. Agric. India, 1929, 10, 157–167).—The antimony electrode can be used satisfactorily in the measurement of the p_H value of soil suspensions. Values found were slightly higher for unstirred than for stirred suspensions. Values obtained by dipping the suitably connected electrode into moist

soil were almost identical with those obtained with soil emulsions. The antimony electrode is eminently suited for field measurements. A. G. POLLARD.

Determination of total carbon and also of the carbon dioxide evolved from soils. A. F. HECK (Soil Sci., 1929, 28, 225–233).—Apparatus is described for determining the organic carbon in soils by wet combustion with chromic acid and for determining carbonate-carbon and free carbon dioxide. A special carbon dioxide trap is described. The total carbon in soil is determined in 45 min., and free carbon dioxide in 3–6 min. A. G. POLLARD.

Principal Schweinfurth greens used for agricultural purposes. L. A. and J. DESHUSSES (Ann. Falsif., 1929, 22, 392–396).—Methods are described for the determination of moisture, arsenious oxide (free and combined), "water-soluble" arsenic, copper, and degree of fineness. Attention is drawn to the empirical nature of the usual method for determining water-soluble arsenic, and the following more exact process is recommended. A sample (1 g.) of material is maintained in contact with 950 c.c. of water at 32° for 24 hrs., with shaking every hour for the first eight. The solution is diluted to 1 litre, sodium bicarbonate is added, and an aliquot portion titrated with *N*/10- or *N*/15-iodine solution. For the determination of copper, 2 g. of material are boiled with 100 c.c. of caustic soda solution and filtered. The precipitated oxide is dissolved in dilute nitric acid, diluted to 250 c.c., and 100 c.c. of the solution are electrolysed. The importance of the degree of fineness is emphasised, and Chancel's method of determination is recommended; 5 g. of the green are suspended in 25 c.c. of dry ether at 17.5°, and the reading is taken when the suspension no longer settles (40–60 min.). From a study of the analyses of more than 100 samples of French, German, and Swiss origins the following limits are proposed for a material of good quality: total arsenious oxide not less than 55%, free arsenious oxide not more than 0.5%, "soluble" arsenic not more than 3.5%, moisture not more than 2%, and degree of fineness at least 30° Chancel.

H. J. DOWDEN.

Diastase activity in plants: effect of phosphates in the soil media. D. T. ENGLIS and L. GERBER (Soil Sci., 1929, 28, 221–223).—Changes in the diastase activity of soya-bean plants grown in pot cultures with phosphatic fertilisers were slight, although the dry weights of the crops were considerably greater than those of unfertilised controls. When very large amounts of fertiliser were used diastatic activity declined, although the crop yields still increased. With rock phosphates no differences in any respect were observed between the fertilised and untreated cultures. A. G. POLLARD.

Influence of soil reaction on the development of meadow plants. F. WEISKE (Landw. Jahrb., 1929, 68, 873–900; Bied. Zentr., 1929, 58, 289–290).—An acid soil reaction, particularly p_H 4.6–5.6, favoured the growth of all meadow grasses examined. Yields were reduced slightly by a neutral reaction and considerably by an acid one. The influence of soil reaction was most pronounced in the earlier stages of plant growth. The harmful effect of neutral and alkaline reactions on

the feeding grasses varied somewhat with the species. The relative sensitivities of a number of the common meadow grasses and legumes are recorded.

A. G. POLLARD.

Relation between the failure of plants and the lime and acid condition of the soil. S. GOY [with P. MÜLLER and O. ROOS] (*Z. Pflanz. Düng.*, 1929, **8B**, 321—348).—Numerous soils are classified according to the authors' scheme (B., 1929, 335). Plant injury is generally associated with the degree of soil acidity. The range of injurious acidity is well defined, but there is an intermediate zone in which the response to liming is dependent on the nature of the crop, cultural methods, and to a large extent on climatic conditions. For practical purposes liming to produce a reaction of p_H 7.0 is sufficient for most crops and to p_H 7.7 for the more sensitive plants. The electrometric methods previously described are normally sufficient to characterise a soil in respect of its lime condition, but may be supplemented in certain cases by determinations of the chalk content and the easily exchangeable calcium. The authors' grouping of soils is discussed in the light of the experimental values recorded and its bearing on the general question of lime requirement shown.

A. G. POLLARD.

Effect of sodium silicate in increasing the yield of barley. A. D. HALL (*J. Agric. Sci.*, 1929, **19**, 586—588).—Mainly polemical against Fisher (B., 1929, 185). Results of the Rothamsted trials for the 41 years preceding 1904, and the deductions made therefrom, are recapitulated.

E. HOLMES.

Effect of fresh straw on the growth of certain legumes. H. G. THORNTON (*J. Agric. Sci.*, 1929, **19**, 563—572).—Beneficial effects of fresh chaff on soya and broad beans are recorded.

E. HOLMES.

Variations in the calcium and magnesium contents of pea plants on different soil types. J. F. FONDER (*Soil Sci.*, 1929, **28**, 15—26).—During the growing of peas the concentration of magnesium and calcium in the soil solution was consistently reduced. The growing plants tended to reduce the acidity of very acid soils and increase that of neutral and alkaline soils. The magnesium and calcium contents of pea plants growing in different soils varied considerably. In soils of similar nature the intake of calcium by the plants varied with the concentration of that element in the soil solution. Variations in the magnesium content of the plants were influenced mainly by the soil texture. The concentration of calcium and magnesium in the tissues and juices of pea leaves was greater than that in the juices and tissues of the stems; that in the leaves increased with the age of the plant, whereas that in the stems was variable. In all cases the amounts of calcium exceeded those of magnesium. More rapid growth coincided with smaller amounts of these bases in the plants. (Cf. B., 1929, 408, 694.)

A. G. POLLARD.

Dredger-mud as fertiliser. HEINE (*Landw. Jahrb.*, 1928, **68**, Suppl. I, 356—357; *Chem. Zentr.*, 1929, i, 130).

Determination of the degree of fineness of Schweinfurt green. L. DESHUSSES (*Mitt. Lebensm. Hyg.*, 1928, **19**, 360—366; *Chem. Zentr.*, 1929, i, 433).

Effect of quantity of precipitation and large applications of nitrogen on the yield and behaviour of hay and pasture meadows. FRECKMANN, BROUWER, STAERK, and SIEGERT (*Landw. Jahrb.*, 1928, **68**, Suppl. I, 39—44; *Chem. Zentr.*, 1929, i, 131).

Effect of heavy mineral nitrogenous fertilisation on clover. FRECKMANN, BROUWER, STAERK, and SIEGERT (*Landw. Jahrb.*, 1928, **68**, Suppl. I, 47—48; *Chem. Zentr.*, 1929, i, 131).

Comparison of the effect of ammonium sulphate with that of Chile saltpetre on the value of lupins as preparation for oats and the effect of superphosphate fertilisation of sugar beet. DENSCH and VON STROTHA (*Landw. Jahrb.*, 1928, **68**, Suppl. I, 28—29; *Chem. Zentr.*, 1929, i, 130—131).

Effect of large applications of ammonium sulphate on the yield and starch content of potatoes. DENSCH and VON STROTHA (*Landw. Jahrb.*, 1928, **68**, Suppl. I, 24—25; *Chem. Zentr.*, 1929, i, 130).

Effect of various potassium salts on potatoes. DENSCH and VON STROTHA (*Landw. Jahrb.*, 1928, **68**, Suppl. I, 22—24, 24, 26; *Chem. Zentr.*, 1929, i, 131).

Vegetation experiments with potassium salts on acid soils. H. KAPPEN (*Ernähr. Pflanze*, 1929, **25**, 6—10; *Chem. Zentr.*, 1929, i, 1391).

• **Potassium fertilisation of fruit trees.** G. A. COWIE (*Ernähr. Pflanze*, 1929, **24**, 12—17; *Chem. Zentr.*, 1929, i, 1391—1392).

Tolerance of different species and varieties of plants to naphthalene vapour. A. HARTZELL (*J. Econ. Entomol.*, 1929, **22**, 354—360).

Relative energy value of lucerne, clover, and timothy hay for the maintenance of sheep. H. H. MITCHELL, W. G. KAMMLADE, and T. S. HAMILTON (*Univ. Ill. Agric. Exp. Sta. Bull.*, 1928, No. 317, 127—167).

See also A., Oct., 1203, **Vitamins-B and -C in young plants** (SCHEPILEVSKAJA). 1204, **Behaviour of nitrogenous compounds during germination** (BONNET).

PATENT.

Xanthate mixture (U.S.P. 1,716,273).—See III.

XVII.—SUGARS; STARCHES; GUMS.

Electrodialytic demineralisation of sucrose solutions. S. ZIEMINSKI (*Przemysl Chem.*, 1929, **13**, 429—445).—Various solutions containing 15% of sucrose and 1% of sodium or potassium chloride and potassium sulphate or carbonate were subjected to electrodialysis in a modified Pauli apparatus, in which the entrance and exit tubes were made movable, as were also the electrodes, made of graphite instead of metal, as being easier to clean and more acid-resistant. One half of the salt content of the above solutions was removed in 8 min., using a current of 1 amp. at 200 volts, and in 90 min. only 0—5% of the original salt remained, whilst at the same time the concentration of sucrose was practically unaffected. Parallel results were obtained with beet juice, except that in this case the use of other than

platinum electrodes is impossible, as a result of dissolution of iron and of adsorption of sugar on carbon electrodes; using initially stronger currents than 0.18 amp. at 100 volts, the liquid foamed to an extent which arrested the electrodialytic process. The juice is of a lighter colour, and a precipitate forms which is readily collected and yields a clean filtrate. Agitation of the solution during the process adversely affected the results.

R. TRUSZKOWSKI.

See also A., Oct., 1168, Dissolution and acetylation of starch (TSUZUKI). 1189, Rapid determination of alcohol (WHALEY). Methods used in sugar analysis (VOICU and DUMITRESCU). 1201, "Soluble specific substance" from gum arabic (HEIDELBERGER and others).

PATENTS.

Converting cellulose and the like into sugar. H. SCHOLLER (B.P. 315,462, 13.4.28).—The process of B.P. 273,317 (B., 1929, 186) is modified to work on the countercurrent principle, the acid being passed first through partially exhausted cellulosic material and finally through fresh material.

D. J. NORMAN.

Purification of liquids containing sugar. G. E. VAN NES (U.S.P. 1,727,738, 10.9.29. Appl., 30.4.27. Holl., 13.9.26).—See B.P. 295,831; B., 1928, 832.

XVIII.—FERMENTATION INDUSTRIES.

Preservation of yeast by pressure, freezing, or storage under water. F. WINDISCH (Woch. Brau., 1929, 46, 349—355).—Samples of five races of bottom-fermentation yeast after storage under water at 0—1° for 3—4 weeks did not differ significantly from the corresponding fresh yeasts in fermentative and reproductive powers. Under similar conditions, two top-fermentation yeasts deteriorated slightly in the first 2 days, after which their vitality remained constant till the end of the experiment (10 days). Storage under water is better than storage as pressed cake either at 8°, at 0°, or in the frozen condition. Yeast is not materially harmed by freezing or by even, rapid thawing.

F. E. DAY.

Influence of temperature on the p_H optimum of diastase during mashing. W. WINDISCH, P. KOLBACH, and L. VON BENEDEK (Woch. Brau., 1929, 46, 345—349).—The existing evidence shows that the optimum p_H for diastase rises with rising temperature, but is dependent on other conditions. Windisch, Kolbach, and Banholzer (B., 1929, 69) have shown that diastatic power is materially lessened below p_H 5.3 if hydrochloric acid is the acidifying agent, and below p_H 5.2 when lactic acid is employed. A comparison with the latter now suggests that with acetic acid this weakening occurs at a still lower p_H value, supporting the previous conclusion that this effect is due to damage by the acid to the diastase before dissolution of the buffer substances from the malt. During decoction mashing the acidity decreased, in a case quoted, from p_H 4.50 at the start (cold) to p_H 4.72 after 30 min. at 45°, finally reaching p_H 4.76. A series of mashes at 50°, 60°, and 70° with reactions adjusted by the addition of acetate buffer solutions were made under decoction and infusion

conditions. The latter method avoids disturbances due to diastatic action at temperatures below the maximum desired. Fermentable extract and p_H were determined on the worts, the latter value having already been shown to agree with that prevailing during the saccharifying period. The results of the two mashing methods were in good agreement, and the optimal zones were found to be p_H 4.5—5.0 at 50°, p_H 4.8—5.4 at 60°, and above p_H 5.6 at 70°. It is remarkable that from a malt mashed for 3 hrs. at 60° and p_H 4.93 a wort with an apparent fermentability of 95.8%, actually 77.7%, was obtained.

F. E. DAY.

Influence of treatment of yeast with varying amounts of sulphuric acid on the fermentative power. STAIGER and GLAUBITZ (Z. Spiritusind., 1929, 52, 270—271).—Yeast treated for $\frac{1}{2}$, 1, and 2 hrs. with sulphuric acid of concentrations increasing from 0.025*N* to 0.4*N* showed a corresponding increase from 15 to 88% in the proportion of cells stained by 0.0001% solution of methylene-blue. In all cases 3 g. of treated yeast yielded about 15.5 g. of carbon dioxide and 19.5 g. of alcohol in 3 days when placed in 12% malt extract. It is concluded that in the purification of yeast by treatment with sulphuric acid a sufficient number of cells survive even drastic treatment to give a normal fermentation if a high pitching rate is used.

F. E. DAY.

Importance of hydrogen-ion concentration for the distillery. III. W. DIEMAIR and K. SICHERT (Biochem. Z., 1929, 210, 286—295).—Experiments with dari (white Kaffir corn) were carried out under similar conditions to those with maize (B., 1929, 299) and gave similar results. The time of steaming and the period at 4 atm. pressure require to be longer (2.25 hrs.). During the whole period of fermentation dari mashes resemble closely as regards acidity and state of combination of nitrogen the maize mashes, and are distinct from potato mashes (cf. B., 1928, 797), but dari attains a slightly different p_H maximum (4.52, maize 4.58).

P. W. CLUTTERBUCK.

Comparative determination of diastatic power by the methods of Lintner and of Windisch and Kolbach. B. LAMPE (Z. Spiritusind., 1929, 52, 276).—Lintner's method is more subject to errors than is that of Windisch and Kolbach. Lintner values of 53, 53, 54, 57, 61, 63, 63, 73, 81, and 101 were found to correspond with Windisch-Kolbach values of 215, 247, 269, 266, 314, 312, 330, 338, 425, and 487, respectively. As a standard for green malt for distillery purposes, formerly taken as 100 on Lintner's scale, a Windisch-Kolbach value of 500 is suitable.

F. E. DAY.

Iron content of wines from the Hérault district. E. HUGUES (Ann. Falsif., 1929, 22, 407—410).—Samples of wines in commerce showed an iron content of 15—39 mg./litre for red wines, and 14—24 mg. for white wines. The high iron content of some wines is due to the corrosion of iron vessels by sulphites in the wine (cf. Fabre and Brémond, B., 1927, 500). The prohibition of the use of potassium ferrocyanide to remove iron compounds is considered justifiable, as with suitable precautions the iron content of wines need not be excessively high.

H. J. DOWDEN.

Determination of copper in grape must and wines. G. DEBORDES (Ann. Falsif., 1929, 22, 410—414).—The customary methods involve the use of large quantities of the material which have to be freed from organic matter by calcination or by treatment with concentrated acids, and these may be a source of error. The method described is more rapid and exact as it comprises separation of the copper as sulphide followed by electrolysis and determination of the copper by Fontès and Thivolle's method (Chim. et Ind., 1925, 97). The reagents required are (i) a standard solution of pure copper sulphate containing 0.0005 g. of copper per c.c. strongly acidified with 10% sulphuric acid; (ii) Fontès and Thivolle's phosphomolybdic solution (cf. A., 1926, 1232) consisting of 10 g. of molybdic acid in 500 c.c. of water to which are added gradually with repeated shaking 200 c.c. of phosphoric acid, *d* 1.38. After keeping for 12 hrs. with occasionally shaking, the mixture is filtered and boiled for $\frac{1}{2}$ hr.; (iii) an 8% solution of potassium permanganate, diluted 100 times at the moment of use. The electrolytic cell consists of a cathode of platinum foil, 1 cm. \times 3 cm., supported by a platinum wire close to the bottom of a narrow test-tube, and an anode of platinum wire passing through the bottom of the tube and extending along its axis. The tube has a side limb inserted immediately above the electrodes as an outlet for washing purposes. A volume of wine containing 0.25—2.0 mg. of copper is acidified with 5—10% of its volume of concentrated sulphuric acid, and after adding 0.1—0.2 g. of mercuric chloride the solution is brought to the boil and saturated with hydrogen sulphide until cold. The precipitate of sulphur with copper and mercury sulphides is collected, washed with 4% acetic acid saturated with hydrogen sulphide, and calcined. The ash, taken up in a few drops of a mixture of equal volumes of nitric and sulphuric acids, is transferred with washings into the electrolytic cell, bringing the total volume to about 5 c.c. The solution is electrolysed for $\frac{1}{2}$ hr. with a current of 0.2 amp. at 2.0—2.2 volts. After washing with water until the current ceases to flow, 5 c.c. of reagent (ii) are introduced and the resultant intense blue liquid is transferred to a wide test-tube and titrated with permanganate solution (iii) until the blue colour vanishes. The process is repeated with 2 c.c. of the standard copper solution (i), the amount of permanganate in both cases being proportional to the copper content. Tests made with materials of known copper content showed that the precipitation of copper is complete and the results are most consistent.

H. J. DOWDEN.

See also A., Oct., 1165, **Formation of *l*-malic acid from fumaric acid by *Aspergillus niger*** (CHALLENGER and KLEIN). 1189, **Rapid determination of alcohol** (WHALEY). 1197, **Adsorption of amylase by blood-charcoal and kaolin** (SABALITSCHKA and WEIDLICH). 1199, **Inactivation of urease by heavy metals** (KITAGAWA). **Dried yeast** (MYRBACK and VON EULER). **Fermentation and growth in dry yeast cells** (BARTEL and others). **Fermentation of yeast at high pressures** (LIESKE and HOFMANN). **Influence of alcohols on alcoholic fermentation** (ABDERHALDEN). 1200, **Determination of micro-organisms in suspension** (WILLIAMS and others). **Butyric acid fermenta-**

tation of calcium lactate (SHAPOSHNIKOV and ZAKHAROV). 1203, **Effect of p_H on vitamin-B of yeast** (WILLIAMS and others).

XIX.—FOODS.

Analysis of coffee adulterated by addition of chick peas. J. FRÉZOULS (Ann. Falsif., 1929, 22, 415—420).—The presence of roasted peas in coffee is readily detected under the microscope by the blue coloration of the starch granules in the presence of iodine, and by the characteristic forms of the pea cells. Although coffee contains no starch, a determination of the proportion of added peas in a mixture, by counting the number of starch granules, is by no means easy or exact. Determination of reducing sugars after autoclaving and boiling with hydrochloric acid is also unsuitable, since roasted coffee contains a proportion (20—33%) of saccharifiable matter. Samples of roasted peas were found to contain 50.6—52.95% of starch, the proportion being reduced to 44.9% by over-roasting. Polarimetric methods were found to be applicable to the determination of the amount of added peas. The material (3 g.), ground to pass 80-mesh, is de-fatted with 50—60 c.c. of ether, dried, and decolorised with 10 c.c. of sodium hypochlorite solution (12°) and 20 c.c. of concentrated hydrochloric acid. To the mixture are added gradually with continual shaking 2 c.c. of 4% phosphotungstic acid solution, and after dilution to 100 c.c. the solution is examined polarimetrically through a 2-dm. tube. A solution of pure pea starch (1 g. in 100 c.c. of hydrochloric acid) gave $[\alpha]_D +193.9^\circ$. The starch content of peas, calculated on this figure, averaged 49.70% of the dry material. Pentosans and saccharifiable celluloses are not determined by this method, hence the slight difference between the polarimetric and saccharimetric results. Application of the polarimetric method to coffee containing known amounts of added peas gave results accurate to within 5%.

H. J. DOWDEN.

Micro-determination of caffeine in coffee. A. C. RÖTTINGER (Mikrochem., 1929, Pregl Fest., 303—312).—A modification of the author's method (B., 1927, 731) is described, in which 5 g. of coffee are moistened with 5 c.c. of 10% ammonia solution and extracted by shaking with 100 c.c. of chloroform. Then 20 c.c. of the extract are evaporated, a small fragment of paraffin wax is added, followed by a few c.c. of ether, and the whole is well shaken with a few c.c. of 0.5% hydrochloric acid. The solution is filtered through cotton wool, the residue extracted four times more with hydrochloric acid, and the united solutions are extracted four times with chloroform. After evaporation of the chloroform the nitrogen in the residue is determined by Kjeldahl's method, and the amount of caffeine calculated from this. In examining "caffeine-free" coffees a larger quantity of material must be taken, and the crude caffeine specially treated to purify it before the Kjeldahl determination is carried out.

H. F. HARWOOD.

Iodine content of Norwegian fish and fish products. G. LUNDE, K. CLOSS, H. HAALAND, and S. O. MADSEN (Årsber. ved. Norges Fisk., 1928, 39 pp.; Chem. Zentr., 1929, i, 1834).

Preservatives. SABALITSCHKA. Determination of theobromine. JALADE.—See XX.

See also A., Oct., 1188, Denaturing of proteins (TADOKORO and others).

PATENTS.

Manufacture of flour. E. A. FISHER and C. R. JONES (U.S.P. 1,727,429, 10.9.29. Appl., 25.5.28. U.K., 27.5.28).—See B.P. 300,537; B., 1929, 71.

[Autoclave treatment for] preservation of eggs and/or other perishables. E. NIERINCK (B.P. 318,534, 6.6.28).

Preservation of [sliced] meat [under fat]. (SIR) T. CLEMENT, and A. CLEMENT & SONS, LTD. (B.P. 319,057, 16.6.28).

Antirachitic foodstuffs (B.P. 318,268—9).—See XX.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of mercuric iodide by iodate reactions. F. G. BROCKMAN (Amer. J. Pharm., 1929, 101, 596—601).—Five g. of dry mercuric iodide are mixed in a 100-c.c. flask with 50 c.c. of a 5% potassium cyanide solution and 40 c.c. of distilled water. When the iodide has dissolved, the solution is diluted to 100 c.c. and 10 c.c. are titrated with 0.2*N*-potassium iodate solution, adding about half the necessary volume at once (about 13 c.c.); 20 c.c. of hydrochloric acid and 5 c.c. of chloroform are added and the titration is continued, stoppering and shaking the bottle after each addition until the colour imparted to the chloroform by the liberated iodine is just discharged. One c.c. of potassium iodate solution corresponds to 0.02272 g. of mercuric iodide. Results from a series of 12 titrations of one sample showed greatest deviations of +0.3% and -0.2% from the average. E. H. SHARPLES.

Silver-ion concentration of colloidal silver germicides. III. Titration of soluble iodides in colloidal silver iodide. R. B. SMITH and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1929, 18, 686—687; cf. B., 1928, 547).—The p_{Ag} of colloidal silver iodide preparations is usually between 12.5 and 14.0, whereas the silver-ion concentration for pure silver iodide is equivalent to p_{Ag} 7.95; showing that these preparations contain a small excess of sodium iodide. The latter can be determined potentiometrically, using 0.01*N*-silver nitrate and silver electrodes prepared by treatment with hot 1.0% potassium cyanide solution (cf. *loc. cit.*). In the absence of other conducting materials in the colloidal silver iodide the conductivity follows closely the soluble iodide content. S. COFFEY.

Preservatives. Detection and pharmacology of alkyl *p*-hydroxybenzoates. T. SABALITSCHKA (Z. angew. Chem., 1929, 42, 936—939).—The superiority of the methyl, ethyl, and propyl esters, as against benzoic acid and phenols, is illustrated by a large number of examples. The propyl ester is generally the most effective, but the methyl ester is specific in some cases. The esters are practically free from odour, and are less poisonous than other common preservatives. The reactions with ferric chloride, Millon's reagent, and Nickel's reagent are employed for detection of the esters; details of the tests are given. S. I. LEVY.

Assay of ground linseed for non-volatile, ether-soluble extractive. J. L. MAYER (J. Amer. Pharm. Assoc., 1929, 18, 683—684).—In order to obtain accurate results in the determination of non-volatile, ether-soluble extract the extraction must be carried out for 20 hrs. in a continuous apparatus. S. COFFEY.

[Novel form of extractor and its application in the] determination of theobromine and assay of kola preparations. JALADE (Ann. Falsif., 1929, 22, 396—405).—The "perforator" described is a combination of Soxhlet extractor and separator, for use with solvents heavier than water (e.g., chloroform). A wide cylindrical vessel (A) has its top closed by a reflux condenser, whilst its lower end is sealed to one limb of a narrow U-tube (B). The other limb of B is longer and connects laterally with a wider tube (C) arranged vertically and in turn sealed laterally close to the top of A. In the bottom of A are placed glass wool and beads covered by a layer of chloroform, on which is floated the liquid to be extracted. The solvent is boiled in a flask fitted to the bottom end of tube C and the vapours condensing in the reflux fall gently through the liquid to be extracted, the excess overflowing slowly into the solvent flask by way of tubes B and C. The extraction is very rapid as it occurs at a high temperature, no emulsions are formed, and only a small quantity of solvent is needed, this being completely recoverable. In the determination of caffeine in kola products the apparatus gives excellent results. For kola extracts and granules, the material is dissolved in warm water, rendered weakly alkaline with ammonia, and extracted for 4 hrs. with chloroform. In the case of kola nuts, the finely-ground material is macerated on a brine-bath for 1 hr. with dilute sulphuric acid. When the paste due to starch has cleared, the solution is neutralised with caustic soda, rendered feebly alkaline with ammonia, and extracted with chloroform for 4—5 hrs. The anhydrous caffeine crystallises out after removal of the chloroform and is 98% pure. In the determination of theobromine in cacao products, the necessity for making numerous extractions with boiling chloroform is obviated by using the new apparatus. The very finely-ground material (10 g.) is freed from fat by extraction with carbon tetrachloride in a Soxhlet apparatus and after removal of the solvent the powder is suspended in 30—40 c.c. of water, to which are added 25 c.c. of *N*-caustic soda, and the suspension is diluted to 250 c.c. with vigorous shaking. After keeping overnight, 125 c.c. of the supernatant liquid are slightly acidified with hydrochloric acid and extracted with chloroform for 5 hrs. The white product after evaporation of the chloroform is freed from traces of caffeine by means of benzene and, after drying, the purity of the theobromine is checked by dissolving in 10 c.c. of *N*-sodium hydroxide, to which are added 10 c.c. of *N*-acetic acid and 20 c.c. of 0.1*N*-silver nitrate. After shaking and keeping overnight, the excess silver is titrated with 0.1*N*-thiocyanate (1 c.c. of 0.1*N*-silver nitrate = 0.0180 g. of theobromine). The apparatus is also very efficient in the determination of alkaloids in materials such as tea and *nux vomica*.

H. J. DOWDEN.

Preparation of digitalis suitable for injection or oral administration. R. A. HATCHER and H. B.

HAAG (J. Amer. Pharm. Assoc., 1929, 18, 551—563, 670—677).—A detailed study has been made of the preparation of digitalis extract (digisol) by the method advocated in the Netherlands Pharmacopœia V, in which an aqueous extract is extracted with chloroform, and as a result a method is recommended for the preparation of stable digisol in which digitalis (No. 30 powder) is extracted with eight times its weight of water for 24 hrs. below 25°. The filtrate is extracted for 2 hrs. with an equal volume of chloroform, and the chloroformic extract evaporated to dryness. The residue is dissolved in chloroform (20 pts.) and precipitated with light petroleum (375 pts.). The precipitate from 200 mg. of residue is finally dissolved in 20 c.c. of alcohol and made up to 100 c.c. with sterile water. A second chloroformic extract contains no active principle. The purified chloroformic residue obtained by repeated precipitation of a chloroform solution of digisol with ether and light petroleum consists mainly of gitalin, with small amounts of digitalin, digitoxin, free genins of the gitalin group, and saponin. The unpurified residue is quite as pure physiologically as any extract from digitalin, whilst it still retains the necessary solubility in water or normal saline to permit of its being used for intramuscular injection. The less soluble, purified residue is suitable for oral administration. Sterile solutions of digisol in normal saline are stable for over six months *in vitro*. The gitalin fraction of digisol or the purified extract is unstable in 0.2% hydrochloric acid. The purified residue obtained from different specimens of digitalis is of nearly constant activity. Digisol is a more active emetic than tincture of digitalis U.S.P.; the rates of absorption are similar. The action following the intravenous injection is immediate, but the full effects are not induced so rapidly as in the case of digitoxin. The persistence of action is greater than that of strophanthin or ouabain, but less than that of digitoxin or digitalis. S. COFFEY.

Comparative chemical examination of different brands of acriflavine hydrochloride (acriflavine) and acriflavine base ("neutral" acriflavine). G. W. COLLINS and A. STASIAK (J. Amer. Pharm. Assoc., 1929, 18, 659—669).—Comparative chemical analyses have been made on representative samples of acriflavine and "neutral" acriflavine from American, English, and French sources. Acriflavine, the hydrated hydrochloride, loses only its water of crystallisation, which varies from 2.45% to 9.34%, on drying over sulphuric acid in a vacuum, but heating to 100° causes the loss of both water and hydrochloric acid and gives anhydrous diamino-*N*-methylacridinium chloride. The hydrochloride can be titrated electrometrically with 0.1*N*-sodium hydroxide; the p_H curves obtained in this titration appear to afford an excellent criterion of the identity and purity of the compound. The samples of "neutral" acriflavine differed considerably from one another in appearance, due to the fineness of the powder and the presence or absence of a very small amount of impurity, which may be removed by treatment with animal charcoal. The water content varies from 5.6% to 10.17% and the sulphated ash from 2.7% to 11.28%. The ash is due mainly to the presence of sodium chloride.

The p_H value varies from 2.8 to 4.8, and electrometric titration gives a good indication of the purity of the sample. This variation in p_H might account for some of the unfavourable results reported clinically. The results obtained indicate that the products of the American manufacturers are quite equal to those obtained from Europe. The term "acriflavine base" is proposed instead of "neutral acriflavine," and extensive standard specifications both for the base and acriflavine hydrochloride are given. S. COFFEY.

Phenol ether in fennel oil and star anise oil. E. TAKENS (Riechstoffind., 1929, 4, 8—9; Chem. Zentr., 1929, i, 1755).—Fennel oil contains a compound, $C_{14}H_{18}O$, b.p. 147°/5 mm., f.p. 21.5°, d^{15}_4 0.967, $\alpha_D^{20} \pm 0^\circ$, similar to anethole, which when distilled at ordinary pressure yields at about 260° anol, $C_9H_{10}O$. Star anise oil gives similar results. A. A. ELDRIDGE.

Australian sandalwood oil compared with the official [oil]. E. SWALLOW (J. Amer. Pharm. Assoc., 1929, 18, 684—686).—Australian sandalwood oil from *Santalum spicatum* contains 90—96% of santalol and is in every way equivalent to the East Indian official oil from *Santalum album*. S. COFFEY.

Medicinal use of the essential and pyroligneous oils of *Cedrus atlantica*. R. MASSY (Bull. Inst. Pin, 1929, Special No., 30—31; Chem. Zentr., 1929, i, 1756).—The essential oil has d^{15}_4 0.939—0.945, $\alpha_D^{15} +15^\circ$ to $+55^\circ$, acid value 1 (max.), acid value after acetylation about 20. The tarry oil has d^{20}_4 less than 1.05, acid content (as acetic) 1%, α_D^{15} of steam-distilled oil above $+20^\circ$. A. A. ELDRIDGE.

Essential oils of Caucasian and Crimean *Thymus*. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst., Moscow, 1927, No. 17, 98—108).—The oil of *Thymus vulgaris*, L., contained thymol and carvacrol (2:1), *p*-cymene, *l*-linalool, and borneol. The sesquiterpene fraction gave no nitroschloride or nitrosite. CHEMICAL ABSTRACTS.

Caucasian eucalyptus oils. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst. Moscow, 1927, No. 17, 39—68).—A sample contained α -pinene 5, cineole 82, aldehyde 2, alcohols about 5.5%, and globulol. Oil from leaves of *Eucalyptus pulverulenta*, Sims., contained α -pinene 8, cineole 78, aldehyde about 5, alcohols about 13%. Oil from *E. viminalis*, Labill., contained (approx.) d - α -pinene 5, cineole 65, aldehyde 4-eudesmol 10, other alcohols 11%. Oil from *E. maideni* contained (approx.) α -pinene 3, cineole 64, *n*- and *iso*, valeraldehyde 3, alcohols 8, eudesmol 5%. CHEMICAL ABSTRACTS.

Caffeine in coffee. RÖTTINGER.—See XIX.

See also A., Oct., 1169, Additive compounds of calcium chloride and iodide (GREENBAUM). 1178, Anæsthetics. Acylaniline derivatives (HARTUNG and MUNCH). 1181, Constituent of *Lactucarium germanicum* (BAUER and SCHUB). 1183, Atophanurotropin (VANINO and MUSSGUG). 1186, Nicotine and its derivatives (LOWRY and LLOYD). Sparteine (WINTERFELD and HOLSCHNEIDER). 1187, Synthesis of

aporphine alkaloids (GULLAND and others). 1188, Determination of antimony in organic antimony compounds (GHOSH). 1196, Active principle of camomile flowers (JUNKMANN and WIECHOWSKI). 1203, Colour test for vitamin-A (EVERS). Vitamin-D in ergot of rye (MELLANBY and others). 1204, Spike disease of sandal (*Santalum album*) (RAO and SREENIVASAYA).

PATENTS.

Manufacture of a substance containing silver chloride in the colloidal state. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 317,121, 10.5.28).—Chlorine is passed into an aqueous solution of a silver protein compound, which is maintained neutral by addition of small quantities of alkali; the dialysed solution is evaporated to dryness below 40° in a vacuum. The product, which dissolves in water and is not precipitated by salts, has pharmaceutical application.

C. HOLLINS.

Manufacture of 2-halogeno-derivatives of pyridine. SCHERING-KAHLBAUM A.-G. (B.P. 288,629, 3.4.28. Ger., 14.4.27).—5-Nitro- or 5-halogeno-derivatives of *N*-substituted 2-pyridones are treated with phosphorus, thionyl, or carbonyl halides. 5-Nitro-*N*-ethyl-2-pyridone is thus converted into 2-chloro-5-nitropyridine.

C. HOLLINS.

Preparation of hexylresorcinol. H. HIRZEL, Assr. to SHARP & DOHME, INC. (U.S.P. 1,717,105, 11.6.29. Appl., 4.3.26. Switz., 10.3.25).—Resorcinol is condensed with hexyl alcohol or a hexyl halide in presence of zinc chloride, zinc, or other condensing agent.

R. BRIGHTMAN.

Manufacture of heptylresorcinols. A. R. L. DOHME, Assr. to SHARP & DOHME, INC. (U.S.P. 1,717,098, 11.6.29. Appl., 15.11.27).—Resorcinol (95 pts.) is heated with zinc chloride (125 pts.) in heptonic acid (300 pts.) at 125–135° and the *resorcyloxy hexyl ketone*, b.p. 195–210°/7 mm., is reduced with zinc amalgam and hydrochloric acid to 4-*n*-heptylresorcinol, m.p. 73–74.5°, b.p. 186–188°/7 mm., claimed as an antiseptic and germicide.

R. BRIGHTMAN.

Manufacture of ketones. BOOT'S PURE DRUG CO., LTD., and J. MARSHALL (B.P. 316,750 and 317,194, 6.7.28).—(A) When a pyrocatechol monoalkyl ether (or its acetate etc.) is condensed with an aliphatic acid in presence of phosphoryl chloride the acyl group enters *para* to the alkoxy group. Guaiacol (or guaiacol propionate) with propionic acid and phosphoryl chloride gives 3-propionyloxy-4-methoxyphenyl ethyl ketone, b.p. 170–220°, readily hydrolysed to 3-hydroxy-4-methoxyphenyl ethyl ketone, m.p. 93–94°. The methyl ketone is similarly prepared. (B) Aliphatic esters of pyrocatechol monoalkyl ethers are converted by aluminium chloride (2 mols.) in nitrobenzene at 15–80° into 4-hydroxy-3-alkoxyphenyl alkyl ketones; e.g., guaiacol propionate, butyrate, and hexoate give 4-hydroxy-3-methoxyphenyl ethyl, *n*-propyl, and *n*-amyl ketones, which have, respectively, b.p. 185–195°, m.p. 61–62°; b.p. 185–195°, m.p. 54–55°; and m.p. 60–62°.

C. HOLLINS.

Manufacture of piperidine derivatives. S. M.

McELVAIN (U.S.P. 1,714,180, 21.5.29. Appl., 3.6.26).—Primary alkylamines are condensed in presence of silver oxide with alkyl β -halogenopropionates, the tertiary amine converted into the piperidone by heating, e.g., with sodium, sodamide, or sodium ethoxide in xylene, and the hydroxypiperidine obtained on reduction is benzoylated, to give a 3-carbalkoxy-1-alkyl-4-piperidyl benzoate, the salts of which (already described; cf. A., 1926, 1044) are claimed as local anaesthetics.

R. BRIGHTMAN.

Preparation of salvarsan. K. MATSUMIYA and H. NAKATA (U.S.P. 1,713,475, 14.5.29. Appl., 12.1.27. Jap., 1.5.26).—3-Nitro-4-hydroxyphenylarsinic acid is reduced in alcoholic hydrochloric acid (at least 4.7*N*) as cathode solution, with hydrochloric acid of any concentration as anode solution, and mercury, lead, or amalgamated lead as cathode and a platinum anode. (Cf. A., 1927, 785.)

R. BRIGHTMAN.

Manufacture of [antirachitic] foodstuffs and/or medicines. I. M. HEILBRON (B.P. 318,268, 2.3.28).—Products with antirachitic properties are prepared by heating sterols, e.g., ergosterol, or material containing sterols, in the presence of catalysts, e.g., finely-divided copper, nickel, copper bronze, etc., and under conditions excluding the risk of oxidation, e.g., in an inert gas or *in vacuo*.

L. A. COLES.

Production of antirachitic products. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,269, 2.4.28).—Ergosterol or material containing it is treated in the absence of oxygen, halogens, etc. with corona discharges, electric corpuscular rays, or Röntgen rays.

L. A. COLES.

Manufacture of active extracts from sexual organs. SCHERING-KAHLBAUM A.-G. (B.P. 291,005, 2.5.28. Ger., 23.5.27).—The tissue-free extract acquires greatly increased activity after irradiation, e.g., for 30 min. at 20 cm. from a Heraeus lamp of 550 watts and 4–5 amp.

C. HOLLINS.

Manufacture of 6-alkoxy-8-aminoquinolines. W. SCHULEMANN, F. SCHÖNHÖFER, and A. WINGLER, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,728,189, 17.9.29. Appl., 21.1.28. Ger., 26.1.27).—See B.P. 310,559; B., 1929, 661.

Medicinal ointment and its manufacture (B.P. 318,661, 7.6.28).

Aromatic hydroxyaldehydes (B.P. 317,381). **4-Hydroxy-3-alkoxybenzaldehydes and derivatives** (B.P. 317,347). **Polysulphides of aromatic carboxylic acids** (B.P. 291,100).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Action of Capstaff's "fine-grain" development in relation to the grain pre-existing in photographic emulsions. E. VITERBI and A. BARBATO (Annali Chim. Appl., 1929, 19, 306–309).—With plates of medium or high rapidity, Capstaff's fine-grain development yields granules appreciably smaller than

those obtained with ordinary developers, but with moderately slow plates its reducing action on the depth of the granules is negligible. This method of development comprises the three processes: direct reducing action on altered silver halide granules, diminution of the depth of the granules due to the solvent action of the sodium sulphite, and reduction of the silver thus passing into solution and its preferential deposition on the altered nuclei. T. H. POPE.

See also A., Oct., 1151, **Photochemistry of silver halides** (LÜPPO-CRAMER). 1152, **Photochemical reductions and oxidations** (STEIGMANN). **Photochemistry of silver halides** (SCHMIDT and PRETSCHNER). **Action of ultra-violet light on certain inorganic compounds** (MONTIGNIE).

PATENTS.

Providing photographic raw film with visible reproducible inscriptions. I. G. FARBENIND. A.-G. (B.P. 295,578, 28.7.28. Ger., 13.8.27. Addn. to B.P. 287,124; B., 1928, 914).—The coloured impression produced by the method of the prior process is protected by applying to the surface containing it a colloid coating which is insoluble in any of the solvents used in the subsequent treatment of the film and is applied in a solvent which does not dissolve the dye.

J. W. GLASSETT.

Tropical packing for photographic plates, films, and papers. I. G. FARBENIND. A.-G. (B.P. 317,267, 13.11.28. Ger., 15.9.28).—The inner paper or cardboard wrapping is completely surrounded with aluminium or other suitable metal foil, which is then covered with a waterproof varnish containing a cellulose derivative.

J. W. GLASSETT.

Photographic developers. I. G. FARBENIND. A.-G. (B.P. 300,970, 21.11.28. Ger., 21.11.27. Addn. to B.P. 280,873; B., 1929, 38).—Nuclear substituted *N*-hydroxyethyl-*p*-aminophenols containing no nitro-group are useful photographic developers. Examples are 2-chloro-4- β -hydroxyethylaminophenol, 6- β -hydroxyethylamino-*m*-cresol, and 5- β -hydroxyethylamino-salicylic acid.

C. HOLLINS.

Preparation of photographic gelatin. W. DIETERLE, O. MATTHIES, E. MAUERHOFF, and J. REITSTÖTTER, Assrs. to AGFA ANSCO CORP. (U.S.P. 1,727,866, 10.9.29. Appl., 21.4.27. Ger., 14.5.26).—See B.P. 271,475; B., 1928, 69.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Treatment of black [gun]powder. IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 300,611, 2.11.28. U.S., 16.11.27).—The speed of combustion of black powder is retarded by coating each grain with a film of nitro-cellulose of the desired thickness. Acetylcellulose and cellulose ethers such as ethylcellulose and benzylcellulose may be used instead of nitrocellulose. S. BINNING.

Manufacture of explosive projectile fuses. SOC. ITAL. POLVERI ESPILODENTI (B.P. 298,948, 16.10.28.

Italy, 17.10.27).—The body of the fuse is made by compressing synthetic or natural plastic masses in suitable heated moulds. S. BINNING.

Detonating firework composition. W. F. GEHRIG, Assr. to ESSEX SPECIALTY CO., INC. (U.S.P. 1,712,555, 14.5.29. Appl., 7.12.27).—A phosphorus sulphide is incorporated with at least one oxidant and an inorganic binder consisting of magnesium oxide and an alum. *E.g.*, a solution of 10 pts. of chrome alum and 35 pts. of potassium chlorate is made into a paste with 8 pts. of beach sand or other filler, incorporated with 35 pts. of magnesium oxide and 12 pts. of phosphorus sesquisulphide, and dried at 40–70°. R. BRIGHTMAN.

Blasting explosive. HERCULES POWDER CO., Asses. of B. I. STOOPS (B.P. 299,862, 11.6.28. U.S., 4.11.27).—See U.S.P. 1,671,793; B., 1928, 626.

Manufacture [dipping] of matches. A. OVTSCHINNIKOV (B.P. 316,927, 27.1.28. Addn. to B.P. 316,592).

XXIII.—SANITATION; WATER PURIFICATION.

Reduction of carbonate hardness [in water] by lime softening to the theoretical limit. C. P. HOOVER and J. M. MONTGOMERY (J. Amer. Water Works' Assoc., 1929, 21, 1218–1224).—The reduction of carbonate hardness is usually limited by the slowness of reaction of the lime employed and, when the magnesium content is high, by the formation of soluble basic carbonates, especially in the presence of soda ash. If excess lime be added sufficient to produce a caustic alkalinity of 40 p.p.m., magnesium salts are precipitated as hydroxide and bicarbonates as carbonates. The excess lime is removed by carbon dioxide, and, if a long reaction period or sand filtration follow, the hardness of the final water is little above the solubility limit. At Columbus, Ohio, the average hardness has been reduced in this way from 55 p.p.m. to 30 p.p.m. at an estimated cost of \$0.81 per million gallons, as compared with \$3.41 to achieve a similar result with soda ash or \$1.67 by the zeolite process. No incrustation of water-mains or damage to meters has been attributed to the method, but incrustation of the sand grains of the filters has been observed which is liable to limit their useful life to 10 years (cf. B., 1927, 510). C. JEPSON.

See also A., Oct., 1162, **Ground waters in Balakhani** (KREMS).

Germicides. SMITH and CHRISTIANSEN.—See XX.

PATENTS.

Disinfecting. T. SABALITSCHKA and H. JACOBSON (U.S.P. 1,715,251, 28.5.29. Appl., 22.12.25. Ger., 24.12.24).—Alkyl esters of *m*- and *p*-hydroxybenzoic acids are used as disinfectants, *e.g.*, in soaps, ointments, mouth washes, etc. R. BRIGHTMAN.

Sanitary cleansing agent. H. PICKUP and W. E. CLARINGBOLD (B.P. 318,344, 11.7.28).—A mixture of powdered sodium bisulphate and anhydrous sodium sulphate is claimed. H. ROYAL-DAWSON.

Moth-proofing agents (B.P. 316,900).—See III.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOV. 22, 1929.

L—GENERAL; PLANT; MACHINERY.

Mechanical handling of materials in and about the chemical plant. II, III. A. K. BURDITT and W. F. SCHAPHORST (*Ind. Eng. Chem.*, 1929, 21, 649—654, 844—850).—Conveyers described include belt conveyers, the drag cable in a trough, scraper, apron, and bucket conveyers, gravity systems such as chutes and roller conveyers, and screw conveyers. Pneumatic conveyers operating by either pressure or suction are suitable for longer travels. C. IRWIN.

So-called Kick law applied to fine grinding. A. M. GAUDIN, J. GROSS, and S. R. ZIMMERLEY (*Min. and Met.*, 1929, 10, 447—448).—The Kick law is generally accepted to mean that for each reduction to one half in particle diameter, in unit weight, the same amount of work is required. In an actual crushing test on quartz the product was graded and the energy units supplied by Kick's law were calculated. This gave a relationship between these units and work expended as determined experimentally. By extrapolating with the help of this ratio the energy corresponding to any degree of fineness can be obtained. With finer division the efficiency, *i.e.*, the relation of this figure to the theoretical surface energy of quartz, steadily increases and greatly exceeds 100%. It is therefore inferred that Kick's law is invalid. C. IRWIN.

Separation of particulate matter [dusts] smaller than screen sizes into graded fractions. D. E. CUMMINGS (*J. Ind. Hygiene*, 1929, 11, 245—256).—The dust is thoroughly dried and screened to pass 270-mesh; 25 g. of this material are made into a smooth paste with 125 c.c. of 95% alcohol diluted to 250 c.c. with distilled water, and 1 litre of water is added to the suspension, with stirring. After settling for $\frac{1}{2}$ min. the supernatant suspension is decanted carefully, allowed to settle for 1 min., again decanted, and the suspension settled for 2 min. This process is repeated at intervals each of which is twice as long as the previous one up to 64 min., then at intervals of 1, 2, and 4 days, after which no further deposition takes place. The first sediment is re-treated as before, the suspension being poured into the vessel with the second sediment and so on, treatment being continued until the supernatant liquid is clear in every case. The mean diameters of quartz particles graded in this way are, respectively, 45.3, 34.0, 25.2, 16.7, 12.1, 8.8, 6.3, and 4.2 microns for settling times of $\frac{1}{2}$, 1, 2, 4, 8, 16, 32, and 64 min. Stokes' law may be applied to settling quartz particles having a diameter between 430 microns (*i.e.*, about 40-mesh) and 0.5 micron. A chart is given from which can be read the settling times for quartz particles of different sizes. A. R. POWELL.

Distillation and rectification of complex mixtures. L. GAY (*Chim. et Ind.*, 1929, 22, 3—18; cf. A., 1927, 1133).—It has been shown that for mixtures of more than four constituents continuous distillation to yield pure products is practically impossible. In such processes as petroleum refining the production of chemically pure compounds is not aimed at. The case of a single column, with source of heat at the bottom and dephlegmator at the top from different portions of which mixtures of different composition are withdrawn, is discussed in detail. Compositions are depicted spatially on a tetrahedron if there are four constituents. It is concluded that it is more economical to withdraw intermediate mixtures as vapour than as liquid. In the case of the dephlegmator, however, it is shown that fractions removed here should be in the liquid state. Discontinuous distillation cannot be treated on the theory developed with strict accuracy, but if the quantity of the mixture in the rectifying column is small compared with that in the still, so that composition changes are slow, conditions are approximately the same. The composition changes during the progress of a distillation are traced. C. IRWIN.

Measurement of plasticity. PARMELEE and RUDD.—See VIII. **Gas filter.** COLLES. **Alloy for vacuum chambers.** NICHOLS.—See X.

PATENTS.

Jaw crusher. J. E. KENNEDY (U.S.P. 1,719,333, 2.7.29. Appl., 20.2.28).—Along one side of the rectangular jaw is a tubular portion, which is integral therewith and protrudes at both sides, so as to form trunnions for supporting the jaw in the crusher-frame. F. G. CLARKE.

Crushing machine for hard materials. J. INLEFELDT, Assr. to G. POLYSIUS (U.S.P. 1,719,979, 9.7.29. Appl., 27.6.27. Ger., 4.2.26).—A pair of vertical grinding rings are resiliently pressed against the rotary crushing ring of a ring-roller crushing mill, whereby it both grinds and crushes. F. G. CLARKE.

Two-zone pulverising apparatus. F. N. DANIELS, Assr. to RILEY STOKER CORP. (U.S.P. 1,719,831, 9.7.29. Appl., 12.4.27).—Coarse material enters one end of a drum near its axis, which is horizontal, and is crushed by rotary members against accumulated material at the bottom of the first zone. A second set of rotary members crushes the charge against stationary members in the second zone, the two zones communicating at the upper portion of the periphery only. F. G. CLARKE.

Drying apparatus. J. FARASEY (U.S.P. 1,719,603, 2.7.29. Appl., 28.3.28).—A horizontal cylindrical shell rotates upon two peripheral rings, which bear upon two

sets of wheels, one set being at the front and the other some distance from the rear of the shell. The rear wheels are carried by a transverse support located between the shell and the firebox below. The latter is divided by a bridge-wall beneath the transverse support. The front portion of the firebox communicates directly with the shell by means of apertures, and a passageway leads combustion gases from the rear portion round the end of the shell and into the interior. The shell is jacketed between the rings, and also between the rear ring and the end of the shell.

F. G. CLARKE.

Treatment [drying] of finely-divided solids.

V. S. HENRY, A. WRIGHT, and F. W. YOUNG, ASSRS. to FILTRATION ENGINEERS, INC. (U.S.P. 1,726,511, 27.8.29. Appl., 8.6.23. Renewed 7.12.27).—A pasty substance, *e.g.*, a filter cake, in a hopper, is forced down upon an endless conveyer of open-mesh material, which carries it between ironing rollers, and then through a dryer, *e.g.*, of the festoon type. A scraper regulates the quantity of substance applied to the conveyer, and endless bands, pressed on opposite sides of the conveyer, may replace the ironing rollers. After leaving the dryer the substance is detached by means of a beater.

F. G. CLARKE.

Decohering solid substances. P. G. M. A. PIGACHE (B.P. 292,965, 27.6.28. Fr., 27.6.27).—Solid substances (colours, catalysts, medicaments, graphite, etc.) are subjected after a preliminary crushing to a slow and continuous or intermittent trituration in the presence of one or more hydrosol colloids, *e.g.*, gelatins, hydro-celluloses, oxycelluloses, albumins, gums, etc., dispersed or expanded in an aqueous medium so as to form a paste sufficiently tacky to prevent water from dripping off during the process. Solid, liquid, or gaseous material capable of facilitating the decohering process and/or preventing putrefaction, *e.g.*, mercuric chloride, acids, glycerin, formaldehyde, ammonia, etc., may also be added, and the products may be diluted with an aqueous or non-aqueous medium or may be dried, preferably after admixture with a substance, *e.g.*, glycerin, which prevents complete desiccation. In examples the preparation of graphitic mineral oil, of hydrosol paints, and of coloured alcohol, oil, or cellulosic paints is described.

L. A. COLES.

Machine for mixing dry and liquid materials.

G. V. ROBERTS, JUN. (U.S.P. 1,720,573, 9.7.29. Appl., 2.3.29).—The mixer is provided with a vertical screw-conveyer, mounted upon a hollow shaft, which is provided with horizontal discharge pipes disposed just below each turn of the conveyer blades.

F. G. CLARKE.

Apparatus for mixing. W. A. GILCHRIST (U.S.P. 1,720,549, 9.7.29. Appl., 19.6.25).—A tank encloses a chamber, open at the top and bottom and not in contact with the inside surface of the tank, and a device, *e.g.*, a propeller, is used to circulate the liquid upwards through staggered baffles in the chamber, and downwards through baffles in the space between the chamber and the tank.

F. G. CLARKE.

Separation of liquids and solids. W. B. EDDISON (U.S.P. 1,720,211, 9.7.29. Appl., 14.3.25).—The suspended solids are separated in the form of a filter cake,

and, while still on the filtering surface, are subjected to pressure by a flexible, impervious surface to remove further liquid.

F. G. CLARKE.

Filtering drum. J. B. VERNAY (U.S.P. 1,720,156, 9.7.29. Appl., 13.1.28. Ger., 18.1.27).—A filtering drum is divided into independent sectors, comprising a number of individual blocks covered with filtering cloth. Each block has a set of grooves in its outer surface, and a single channel cross-connecting such channels and disposed inwardly of the plane thereof, the channels of any two successive blocks registering endwise. Radial tubes connect distributing gear at the centre of the drum with a channel of each sector.

F. G. CLARKE.

Liquid-clarifying apparatus. H. MILLER (U.S.P. 1,720,185, 9.7.29. Appl., 25.4.27).—After issuing through a trap, the liquid is passed in succession through charge, mixing, washing, and precipitating tanks to a filter; the outlet of the precipitating tank is above the floor.

F. G. CLARKE.

Compressed gas. M. G. FARRAR, ASST. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,726,018, 27.8.29. Appl., 1.5.28).—To prevent water which is condensed in the outlet of a vessel containing gases under pressure from freezing, about 1% of an organic substance which is soluble in water and is expelled with the water vapour, *e.g.*, alcohol or acetone, is introduced into the vessel.

F. G. CLARKE.

Separation [of gases] by liquefaction. R. C. TOLMAN, W. L. DE BAUFRE, J. W. DAVIS, and M. H. ROBERTS, ASSRS. to S. G. ALLEN (U.S.P. 1,728,947, 24.9.29. Appl., 16.9.22).—During the condensation of a mixture of gases the composition of the mixture of the liquid and vapour is maintained approximately constant until the gas to be separated forms a large proportion of the uncondensed fraction. Final separation is effected by external refrigeration, using part of the condensed gases as a cooling medium.

C. A. KING.

Extraction of volatile substances (U.S.P. 1,715,313).—See III.

II.—FUEL; GAS; TAR; MINERAL OILS.

Comparison of the conditions of occurrence of bituminous coal and petroleum. E. McK. TAYLOR (J. Inst. Petroleum Tech., 1929, 15, 372—384).—Examination of the roofs of bituminous coal seams and the cap-rocks of oil sands shows both types of strata to possess identical chemical, physical, and biological properties, and to consist of material that has undergone base exchange with solutions of sodium chloride followed by hydrolysis in fresh water of the sodium clay thus formed. Evidence points to the formation of bituminous coal and petroleum by the same type of decomposition, but from different types of organic matter. Laboratory tests confirm field observation that plant material containing lignocellulose when decomposed in a sodium clay yields materials similar to those in bituminous coal; hence a source of organic matter free from lignocellulose is indicated as the material from which petroleum has been formed,

a condition satisfied by material of estuarine and marine origin.

H. S. GARLICK.

Determination of best temperature of hydrogenation for bituminous coals. J. P. AREND (Proc. 2nd Int. Conf. Bit. Coal, 1928, 2, 485—490).—The first deviation of the expansion curve of the heated coal indicates the first appreciable extent of hydrogenation. At the maximal rate of expansion hydrogenation gives the greatest yield of oils and the maximal quantity of light gasoline.

CHEMICAL ABSTRACTS.

Presence of chlorine in coal. H. TER MEULEN (Rec. trav. chim., 1929, 48, 938—940).—For 22 samples of coal examined the chlorine content varied from 0.03 to 0.33%. Extraction of the powdered coal with cold 0.2N-nitric acid removes inorganic chlorine (0—0.12%); the remainder is probably organic in nature. A Yorkshire coal contained as much as 0.46% Cl (0.19% extractable). Calcination of the extracted coal gives a residue free or almost free from chlorine.

H. BURTON.

Peat as a source of nitrogen. Z. V. LOGVINOVA (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 56, 1—66).—Moss peat contains more adsorbed ammonia than meadow peat; the upper layers contain more than the lower. The calcium is in the adsorbed state, and is easily replaced by sodium. Moss peats, but not meadow peats, give appreciable amounts of phosphorus when treated with sodium chloride solution.

CHEMICAL ABSTRACTS.

Significance of the hydrogen content of charcoals. H. H. LOWRY (J. Physical Chem., 1929, 33, 1332—1342; cf. A., 1924, ii, 393).—Determinations of the hydrogen contents of coal under various conditions show that at a constant temperature, exceeding some temperature characteristic of a given material, the hydrogen content decreases regularly with an increase in the time of treatment. The range investigated varied from 800° to 1500° with times of heating between 0.5 and 141.5 hrs. Two equations of similar form, one being $H = H_0 e^{-a(T-T_0)^n}$, where H is the hydrogen content after heating at T° for a time t with a material having an original hydrogen content H_0 , a and n being constants, represent the data for three different coals. The effect of two separate heat treatments is additive and, in all cases, the hydrogen content is more sensitive to a change in temperature than to duration of heating at a particular temperature. In the case of 14 different coals, the hydrogen contents (originally ranging from 2.13 to 4.73%) after heating for 30 min. at 900—1200° lie within a narrow range, and, in general, the greater the hydrogen content of the raw material, the less is the hydrogen content of the remaining charcoal for a specific treatment. Analyses of charcoals prepared between 900° and 1300° in hydrogen, air, and carbon dioxide show that the atmosphere in which a sample of coal is heated does not influence the hydrogen content of the resultant charcoal, indicating that this factor has not the importance in activation processes which is commonly supposed. An accurate method for the determination of hydrogen contents less than 0.2% is described.

L. S. THEOBALD.

Decomposition of coal into definite organic compounds by oxidation. O. HORN (Brennstoff-

Chem., 1929, 10, 362—364).—The literature bearing on the oxidation of charcoal and coal with the production of definite organic compounds is summarised.

A. B. MANNING.

Oxidation of Willstätter's lignin with nitric acid. O. HORN (Brennstoff-Chem., 1929, 10, 364—365).—By the treatment of Willstätter's lignin with 5N-nitric acid oxalic acid was obtained together with an insoluble oxidation product. By further oxidation of the latter with more concentrated acid and finally with fuming acid a small quantity of mellitic acid was obtained.

A. B. MANNING.

"Wet" purification of coal gas—a review of past and present methods. L. S. PICKLES (Gas J., 1929, 188, 107—114).—A historical survey of purification processes from 1792 to 1923 is given, and a new method is described in which crude gas passes into an acid liquor and is maintained in an acid atmosphere until cold, when it is treated with alkali. A small setting of two horizontal iron retorts was used, of which the hydraulic main was fitted with special sprays for the acid liquor. The annular condensers were followed by the cyanogen washer, the exhauster, and the iron oxide purifiers. The acid liquor contained 0.75% of sulphuric acid and the alkali cream was made as in the Davis-Neill process, with 5% of excess alkali. The gas and the various liquids were analysed at frequent intervals and full working data are given for an 8-weeks' operation of the plant; the results confirm the predictions made for the process. Noxious constituents of the liquor are considerably reduced, and no important difficulties arise in handling the dilute acid; corrosion due to cyanogen compounds is practically eliminated, and cyanides are more readily recoverable. The recovery of phenol is increased and treatment of effluent in order to render it harmless is far less difficult.

R. H. GRIFFITH.

Determination of the calorific value and analysis of coal gas. G. NEUMANN (Arch. Eisenhüttenw., 1929—30, 3, 123—132, 199—203; Stahl u. Eisen, 1929, 49, 1455—1457).—The precautions necessary for obtaining correct results with the Junker calorimeter and the calorimeter are described, and the magnitude of the errors that may be caused by various deviations from standard conditions have been determined. A deviation of $\pm 5^\circ$ from the standard temperature of the gases passing into and out of the Junker calorimeter causes an error of $\mp 0.77\%$ in the calorific value. The calorimeter usually gives results which are 2—3% too low. In the analysis of the gas, fuming sulphuric acid is preferable to bromine for the determination of the unsaturated hydrocarbons and phosphorus to pyrogallol for oxygen absorption. Combustion in the Drehschmidt-Winkler platinum capillary avoids the formation of nitrous oxide and gives excellent results for hydrogen and methane.

A. R. POWELL.

Determination of tar fog in coke-oven gas. W. KLEMP (Brennstoff-Chem., 1929, 10, 359—361).—Tests have been carried out with the following types of tar-fog filter: (1) tube packed with glass wool, (2) tube packed with cotton wool, (3) tube containing two porous discs of sintered glass, and (4) extraction thimble packed with cotton wool and fitted in a glass container so that

the gas passes successively through the thimble and cotton wool. With (1) the tar fog was not completely retained, whilst to obtain complete retention with (2) it was necessary to pack the tube so tightly that the velocity of the gas current was very greatly decreased. Only tar fogs of low concentration were absorbed completely by (3). The arrangement (4) was the most efficient of those tested. A difficulty arose from the loss of volatile constituents of the tar during drying. A partial correction for this was made by drying in a constant current of air, plotting weight against volume of air passed, and extrapolating back to zero from the constant rate of loss of weight which is eventually reached.

A. B. MANNING.

Exactitude of measurement of gas-investigation methods. A. SCHNEIDER (Gas- u. Wasserfach, 1929, 72, 829—831).—The errors involved in measurements with gases are discussed in connexion with calorific value, sp. gr., hydrogen sulphide, ammonia, oxygen, and naphthalene. By the methods recommended 0.1 g. of ammonia in 100 m.³ of gas, 0.02% of oxygen, and 0.2 g. of naphthalene/100 m.³ can be determined.

R. H. GRIFFITH.

Inflammability of mixed gases. G. W. JONES (U.S. Bur. Mines Tech. Paper No. 450, 1929, 38 pp.).—The limits of inflammability have been determined for mixtures of methane, of hydrogen, and of carbon monoxide with air and nitrogen or air and carbon dioxide. The inflammability of mixtures can be calculated with fair accuracy by means of a simple formula.

O. W. GIBBY.

Inflammability of mixtures of ethyl alcohol, benzene, furfuraldehyde, and acetone. G. W. JONES and J. R. KLICK (Ind. Eng. Chem., 1929, 21, 791—793).—The lower inflammable limits found for the solvents when mixed with air and at 125° were: ethyl alcohol 3.85%; benzene 1.53%; furfuraldehyde 2.10%; acetone (pure) 2.92%, (commercial) 3.18%. The results of tests made on mixtures of these four solvents give limits of inflammability (lower) when mixed with air which agreed closely with calculated values obtained by the application of Le Chatelier's law.

H. S. GARLICK.

Effects of knock-suppressing and knock-inducing substances on the ignition and partial combustion of certain fuels. R. E. SCHAAD and C. E. BOORD (Ind. Eng. Chem., 1929, 21, 756—762).—Hot-wire ignition curves were determined for *n*-propyl, *n*-butyl, and isoamyl alcohols, toluene, isoamyl acetate, kerosene, ethyl maleate, and ethyl fumarate between the lower and upper limits of inflammability. The fuel-air mixtures investigated were produced by a vapour-pressure method described and shown to be applicable to the continuous and reproducible preparation of such a series of mixtures. The current required by an electrically heated platinum wire for ignition of the most easily ignitable mixture of air and toluene, isoamyl acetate, or kerosene was increased by the addition to the fuel of a knock suppressor such as lead tetraethyl or selenium diethyl. On the contrary, the addition of one of the knock inducers decreased the hot-wire ignition current. The addition to each of the above-

mentioned fuels of either a knock suppressor or a knock inducer had no noticeable effect on the ignition curves obtained by means of direct-current break sparks. Pre-ignition combustion curves of fuels at chosen saturator temperatures were obtained by plotting filament current (in amp.), both increasing and decreasing values, against the corresponding percentages of carbon dioxide as ordinates. No measurable quantity of carbon monoxide was formed in any of these experiments. Very little combustion of toluene took place at filament currents less than that required for ignition, but an abrupt increase took place with ignition. Without exception, the addition of knock suppressors to the fuels mentioned decreased pre-ignition combustion. On the contrary, the addition of a knock inducer caused a marked increase.

H. S. GARLICK.

Propylene from crude oil. A. DOBRYANSKI, B. ARKHANGELSKI, and R. STEPANYAN (Nefte. Choz., 1928, 15, 46—52).—When cracked (0.31 g. per min. through a copper tube at 550—750°), solar oil, of the various fractions examined, gave the highest yield of propylene. The optimal temperature was 600—650°. At higher temperatures the propylene decomposed with formation of ethylene. The yield of propylene could not be increased by using nickel or iron as catalyst.

CHEMICAL ABSTRACTS.

Higher alcohols from petroleum hydrocarbons. III. E. HOLZMANN and S. PILAT (Przemysl Chem., 1929, 13, 455—460; cf. Pilat and others, B., 1929, 743).—The best yields of alcohol from low-boiling benzines produced by the Cross cracking system are obtained by shaking 1 litre of the benzine with 500 c.c. of 85% sulphuric acid and 100 g. of acetic acid, at a temperature below 0°, when 147 c.c. of alcohols are obtained; in the absence of acetic acid the yield is only 88 c.c. The former figure represents a yield of 50% of the olefines present. By treatment of benzine successively with 65 and 85% sulphuric acid, a mixture in the ratio 2:1 of secondary to tertiary butyl, amyl, and hexyl alcohols is obtained. The hydrolysis of the alkyl sulphates should be carried out in an alkaline medium, otherwise olefines are regenerated and largely converted into tarry polymerides. R. TRUSZKOWSKI.

Oxidation mechanisms of the paraffin hydrocarbons. G. EGLOFF, R. E. SCHAAD, and C. D. LOWRY, JUN. (Ind. Eng. Chem., 1929, 21, 785—791).—The theories proposed to account for the course of oxidation of the paraffins are critically reviewed. Particular attention is paid to the hydroxylation theory, which fails to explain why primary alcohols appear not to be primary products of hydrocarbon oxidation, and to the peroxide theory, which satisfactorily explains the prominence of aldehydes among the oxidation products, but cannot be considered proved fully because of the failure to isolate and identify peroxidic oxidation products, nor does it present a complete explanation of knocking, for the tendency of hydrocarbons to knock does not parallel what is known of their ability to form peroxides. The study of flame propagation and the assumption of a chain mechanism for hydrocarbon oxidation make intelligible the checking of knocking by chemical reagents which act as inhibitors of a special

type, and also accords with the stimulation of knocking by unstable reactive substances which, in the engine cylinder, may include oxides of nitrogen and ozone, as well as peroxides.

H. S. GARLICK.

Refining of shale oil. Y. TANAKA and R. KOBAYASHI (J. Fuel Soc. Japan, 1929, 8, 92—93).—The addition of an adsorbent substance, such as clay, before distillation of a shale oil reduces the production of pitch and gas, and improves the quality of the liquid distillate. The process may also be improved by addition of alcohol vapour, as the b.p. of the oil fractions are thereby lowered. Hydrogenation of shale oil can be carried out at 280—290° and 75 atm. in presence of a nickel catalyst, or by nascent hydrogen produced from zinc and acid; in either case the yield of solid paraffins is considerably increased.

R. H. GRIFFITH.

Fuller's earth and acid-treated earths as oil-refining adsorbents. C. W. DAVIS and L. R. MESSER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 207, 18 pp.).—Fuller's earth and earths from which oil-bleaching adsorbents may be prepared vary considerably in composition, but a high silica:alumina ratio is characteristic. The characteristics of the earths are discussed.

CHEMICAL ABSTRACTS.

Determination of unsaturated and aromatic hydrocarbons in gasoline. S. S. NAMETKIN and E. A. ROBINSON (Nef. Choz., 1929, 14, 775—779).—Treatment of gasoline with 98% sulphuric acid at 0° for 30 min. removes the whole of the unsaturated hydrocarbons.

CHEMICAL ABSTRACTS.

Testing of motor spirit. R. BRUNSCHWIG and L. JACQUÉ (Compt. rend., 1929, 189, 486—489).—Potential resins in motor spirit may be determined satisfactorily by passing a current of air through the liquid while refluxing, evaporating, and weighing the residue remaining at 105°. Production of resins by irradiation with ultra-violet light does not give results in agreement with those obtained by storing for nine months.

C. W. GIBBY.

Thermal decomposition of paraffin wax in presence and in absence of hydrogen under high pressure. H. I. WATERMAN, T. W. TE NUYL, and J. N. J. PERQUIN (J. Inst. Petroleum Tech., 1929, 15, 369—371).—A comparison of analogous fractions from the vacuum distillation of the products of thermal decomposition of paraffin wax in the presence (berginisation) and in the absence (cracking) of hydrogen under high pressure shows that the aniline point of the berginisation product is about 20° higher than that of the cracking product, the refractive index of the cracked products is 0.02—0.03 higher, their sp. gr. is higher, and the bromine value of the cracking products is much higher, substitution more readily taking place on cracking than on berginisation. Comparison with the physical constants of the normal saturated aliphatic hydrocarbons indicates the berginised product to consist possibly of 90% of saturated hydrocarbons, whilst the cracking product is strongly unsaturated and contains compounds of a cyclic character.

H. S. GARLICK.

Anti-oxygenic effects of sulphur and selenium on refined transformer oil and paraffin wax. S. MIZUSHIMA and T. YAMADA (J. Soc. Chem. Ind. Japan,

1929, 32, 848—851).—That highly refined transformer oil is far more easily oxidisable than a less refined one is confirmed. The authors' results, contrary to those of other workers, show that sulphur contained in petroleum oils acts as an anti-oxidant. When a refined white transformer oil (originally containing 0.023% S) was mixed with 0.1% of sulphur and oxidised with oxygen at 86 cm. pressure for 100 hrs. at 120°, the oil resisted the oxidation almost perfectly, whilst the same oil without added sulphur was strongly oxidised. By mixing with a small amount of sulphur, paraffin wax resisted the oxidising action of oxygen. Further, isoamyl sulphide and selenium are, equally as is free sulphur, remarkably effective as anti-oxidants for refined transformer oil.

K. KASHIMA.

Physical properties and constitution of mineral lubricating oils. S. KYROPOULOS (Z. physikal. Chem., 1929, 144, 22—48).—The relation between refractive index and mol. wt. is represented by separate curves for normal paraffins, isoparaffins, naphthenes, and aromatic hydrocarbons, and the determination of these two constants for a mixture of hydrocarbons of unknown character enables it to be classified. The method has been applied to a number of mineral lubricating oils in cases where the great complexity of the mixtures makes identification by chemical means impracticable. A typical group of Pennsylvanian lubricating oils ("Valvoline") and some other saturated hydrocarbon oils are shown to be mixtures consisting largely of isoparaffins. The lower lubricating value of oils containing normal paraffins is due partly to the superior lubricating properties of branched-chain as compared with straight-chain molecules, but largely also to the fact that in a mixture of given b.p. the normal paraffins have a lower mol. wt. than the isoparaffins, and consequently lower lubricating power. The removal of normal paraffins by crystallisation from oils used in internal-combustion engines is especially important, since the former are the least readily burned and therefore accumulate in the unburnt residue, thereby greatly lowering the lubricating power and leading to excessive consumption. The relation between the technical value of lubricating oils and their chemical nature, and the question of the origin of mineral oils, are also discussed.

F. L. USHER.

Iodine values of lubricating oils before and after use in automobile engines. W. F. SEYER and J. S. ALLEN (Ind. Eng. Chem., 1929, 21, 793—794).—The iodine value, as determined on a definite sample of dried oil dissolved in carbon tetrachloride and in contact with excess iodine monochloride for 2 hrs. at 0°, shows, with one or two exceptions, that the iodine value of lubricating oil decreases in use in automobile engines in spite of dilution by unconsumed fuel, and that the decrease is greatest during the first 500 miles.

H. S. GARLICK.

Gas purification in relation to coal sulphur. F. W. SPERR, JUN. (Proc. 2nd Int. Conf. Bit. Coal, 1928, 2, 37—64).

Products of decomposition in the distillation of mazout. S. A. VISHETRAVSKI (Nef. Choz., 1928, 14, 769—771).

Mazout distillation in Germany and a new process developed by the Azneft for extracting oil out of heavy bottoms. K. V. KOSTRIN (Neft. Choz., 1928, 15, 658—663).

Distillation of complex mixtures. GAY.—See I. **Gas filter:** COLLES.—See X. **Brewers' pitch.** KUTTER.—See XVIII. **Determination of water [in coal etc.].** PRITZKER and JUNGKUNZ.—See XIX.

PATENTS.

Production of briquettes. F. L. SCHMIDT (B.P. 294,879, 19.7.28. Ger., 30.7.27).—For the production of a hard briquette, the products obtained by fermentation of cellulose or molasses lyes are employed. The sugar substances present are thus decomposed, leaving a material from which a non-sticky substance (similar to tar pitch) may be obtained by evaporation. At the same time such valuable by-products as alcohol, acetone, acetaldehyde, etc. are obtained.

J. A. SUGDEN.

Briquetting of coal, coke, and the like. P. J. and J. P. SPENGLER (B.P. 299,859, 27.4.28. Ger., 4.11.27).—Briquettes may be weak owing to air fissures and striation. De-aeration may be effected during pressing by applying the pressure in steps and by partially or wholly removing the briquette from the mould after each application. In this way a smaller quantity or an inferior binder may be used.

J. A. SUGDEN.

Manufacture of agglomerated blocks of fuel. A. L. J. VOINCHET and A. A. LERCIU (B.P. 297,085, 11.9.28. Fr., 14.9.27).—China or other clay, to which is added an alkaline silicate, is used as the binding agent. The material is compressed, then treated with a solution of a calcium salt (e.g., chloride) containing traces of acids, glycerin, etc. in order to render the binder insoluble and capable of resisting disintegration by water, and finally dried. The product burns without smoke, and leaves no fusible ash; drying of the fuel before briquetting is not necessary. J. A. SUGDEN.

Treatment of coal dust for utilisation in burners. L. LIATS (B.P. 316,389, 21.6.28).—The dried dust particles are covered with a non-absorbent layer of a substance such as coal-tar pitch by mixing 3–6% of the finely powdered pitch with the dried coal dust at the liquefying temperature of the pitch; a small amount (3–5%) of greasy material, e.g., mazout or petroleum oil, may also be added. Risk of blockages in feed pipes due to absorption of moisture and of spontaneous combustion of the coal dust when stored in bins is thereby avoided.

J. A. SUGDEN.

Preparation of granular carbon. H. H. LOWRY, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,722,055, 23.7.29. Appl., 1.6.25).—Comminuted carbonaceous material is heated slowly, so that a uniform product of low porosity is obtained, and this is subsequently heated to a higher temperature. R. H. GRIFFITH.

Carbonisation of briquettes or like agglomerate blocks of fuel. E. GEVERS-ORBAN and J. PIETERS (B.P. 294,581, 26.7.28).—To prevent briquettes from sticking together or altering their shape during carbonisation, it is necessary to embed them in some

powdered material. In the case of briquettes made from peat, lignite, or other non-caking substances, the material must first be carbonised, then agglomerated with a binder, and carbonised again. In the second carbonisation the briquettes are embedded in the powdered raw fuel, which after the carbonisation is ready for briquetting. This process is continuous. In the case of bituminous or caking materials which require only one carbonisation, the briquettes are embedded in coke breeze left over from the previous carbonisation. J. A. SUGDEN.

Carbonising or distilling material. INTERNAT. COMBUSTION, LTD., and S. McEWEN (B.P. 316,734, 8.6.28).—In a modification of the prior patent (B.P. 316,901; B., 1929, 802) the gases as evolved are drawn down through the hot coke in order to increase the yield of permanent gas. J. A. SUGDEN.

Obtaining liquid products from coal and the like. M. MELAMID (U.S.P. 1,723,431, 6.8.29. Appl., 5.12.24. Ger., 17.11.21).—The hydrogenation of coal and similar substances is carried out under pressure in the presence of tin, and at a temperature not above 600°.

R. H. GRIFFITH.

Manufacture of viscous oils from brown-coal tars or their distillation products. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 316,701, 11.5.28).—Good yields of viscous oils can be obtained from oils freed from benzine or paraffin wax, by treatment, at 100–200°, with metallic chlorides (e.g., aluminium or ferric chloride). Heavy-metal salts, particularly those of group VI, may also be added as activators. The yield depends on the fraction employed and its treatment. The formation of low-boiling constituents is almost completely prevented. J. A. SUGDEN.

Gasification of solid fuel. J. M. RUSBY and C. W. ANDREWS, Assrs. to U.G.I. CONTRACTING CO. (U.S.P. 1,720,403, 9.7.29. Appl., 5.12.22).—Bituminous fuel is subjected to a hot air-blast; when the charge becomes plastic the air supply is stopped and the fuel bed is stirred mechanically to break up the coalesced material. Fresh fuel is then added, and the process continued.

R. H. GRIFFITH.

Gas producers. I. G. FARBENIND. A.-G. (B.P. 300,277, 7.11.28. Ger., 10.11.27. Addn. to B.P. 214,544; B., 1924, 549).—In producers for gasifying small-sized fuel, as described in the prior patent, a waste-heat boiler (preferably water-tube) is built into the upper portion of the producer itself, or into the dust separator. When it is not desired to produce water-gas or producer gas, sufficient secondary air for complete combustion may be admitted above the fuel bed.

J. A. SUGDEN.

Gas producers. T. R. WOLLASTON (B.P. 316,749, 5.7.28).—The water-cooled walls of the producer are utilised as a steam boiler. At the hot zone the annular boiler itself serves as the wall, but the upper portion of the boiler (containing the water level and steam space) is protected by the ordinary refractory lining. The water level is maintained by a feed-water regulator. A central column, terminating in a conical distributing head, extends upwards towards the fuel-feed throat and, together with a series of fire bars which can be agitated,

ensures a uniform movement of the fuel. The steam generated may be utilised in heating installations or to saturate the blast.

J. A. SUGDEN.

Decomposition of gas mixtures, more particularly coke-oven gas. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 284,213, 21.1.28. Ger., 24.1.27).—Coke-oven gas is compressed to 10 atm. and the carbon dioxide, hydrogen sulphide, etc. are removed; the purified gas is then cooled in a countercurrent heat exchanger so that the higher-boiling constituents separate. Most of the methane is subsequently condensed in other coils around which circulates additional compressed nitrogen; the cooling effect produced by evaporation of the methane is thus transferred to the nitrogen and carried to another point, when the latter evaporates at a lower pressure. After this treatment the residual gas is rich in carbon monoxide and hydrogen; the former is readily condensed, and the hydrogen is finally purified by washing with liquid nitrogen.

R. H. GRIFFITH.

Cracking of hydrocarbon oils. SINCLAIR REFINING Co., Assees. of E. C. HERTHEL (B.P. 303,879, 3.11.28. U.S., 12.1.28).—An initial charge of oil is brought to cracking conditions of temperature and pressure and the vapours are subjected to a refluxing operation in direct contact with fresh oil. At the same time the reflux condensate and the unvaporised raw oil from the refluxing operation are circulated through the heating zone mixed with the unvaporised oil recirculated from the vaporising zone until a substantial amount of cracked oil has been taken off. Then, before the oil circulating through the heating zone becomes saturated with pitch, the recirculation of unvaporised oil from the vaporisation zone through the heating zone is stopped, while continuing to subject vapours taken off from the vaporisation zone to the refluxing operation and to circulate admixed reflux condensate and unvaporised fresh oil from the refluxing operation through the heating zone until a further substantial amount of cracked oil has been taken off. During the second-mentioned period of operation unvaporised oil from the vaporisation zone is discharged. Finally, discharge of unvaporised oil is stopped and recirculation through the heating zone continued.

H. S. GARLICK.

Catalytic treatment of hydrocarbon oil. G. EGLOFF and J. C. MORRELL, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,705,180, 12.3.29. Appl., 10.5.22. Renewed 5.9.28).—Into the oil raised to cracking temperature are injected regulated quantities of hydrogen chloride, and the mixture is passed into an enlarged zone containing disintegrated aluminium, a pressure above atmospheric being maintained during the cracking process. The vapours produced are condensed and collected.

F. G. CROSSE.

Conversion [cracking] of hydrocarbon oil. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,705,181, 12.3.29. Appl., 4.12.25).—Fuel oil, or other heavy crude oil containing no light fractions, is heated, e.g., at 200 lb./in.² and 440°, and discharged into a reaction chamber simultaneously with kerosene oil heated separately at, e.g., 2000 lb./in.² and 595°. The expansion chamber is connected with the usual dephlegm-

ator, condenser, and receiver, and the phlegms are returned to a separate heating tube and discharged into the reaction chamber at, e.g., 1200 lb./in.² and 540°.

R. BRIGHTMAN.

Distillation of hydrocarbon oils. W. W. HOL- LAND, Assr. to STANDARD OIL Co. (U.S.P. 1,723,698, 6.8.29. Appl., 11.12.24).—Vapours from the still pass into a dephlegmator, in the upper part of which the partly fractionated vapours are in indirect heat exchange with a current of steam at 105–120°. Condensate returns to the still in countercurrent with ascending vapours, whilst the steam heated thereby to, e.g., 160–220° is passed directly into the distilling oil.

R. BRIGHTMAN.

Purification of hydrocarbons. I. H. DERBY (Assr. to P. C. REILLY) and K. R. DAVIS (U.S.P. 1,727,052, 3.9.29. Appl., 13.2.22).—Normally solid hydrocarbon compounds are atomised in a molten condition and subjected to a current of inert gas.

H. ROYAL-DAWSON.

Treatment of carbon from cracking stills. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,705,179, 12.3.29. Appl., 23.4.21. Renewed 20.2.28).—Oil-soaked carbon is discharged from the expansion chamber of a cracking still into a rotating, centrally heated, cylindrical still, preferably provided with steel balls to disintegrate the carbon. The liquid residue distils off at atmospheric pressure and is stored or returned for cracking, and the dry carbon is discharged.

R. BRIGHTMAN.

Production of lubricating oil from petroleum oil. J. B. TERRY and R. A. HALLORAN, Assrs. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,721,719, 23.7.29. Appl., 14.2.23. Renewed 16.5.28).—Petroleum is distilled under reduced pressure until asphalt remains; about one half of this residue is then destructively distilled to leave a brittle pitch, which is then extracted with solvent naphtha in order to recover the lubricating oils that are mixed with it.

R. H. GRIFFITH.

Lubricating composition. A. L. KLEES, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,727,109, 3.9.29. Appl., 16.7.24).—The composition comprises a mixture of cyclic unsaturated non-benzenoid hydrocarbons with naphthenic and paraffin hydrocarbons of similar boiling range.

L. A. COLES.

Separation of hydrogen from gaseous mixtures [coke-oven gas]. G. F. JAUBERT, Assr. to SOC. AMMONIA (U.S.P. 1,723,425, 6.8.29. Appl., 26.12.24. Fr., 6.11.24).—See B.P. 242,583; B., 1926, 440.

Cracking of hydrocarbon oils. E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,721,779 and 1,721,780, 23.7.29. Appl., 12.1.28).—See B.P. 303,879; preceding.

Manufacture of carburetting liquids similar to petrol. E. A. PRUDHOMME, Assr. to SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (U.S.P. 1,711,855, 7.5.29. Appl., 20.1.25. Fr., 19.5.24).—See B.P. 238,931; B., 1925, 950.

Treatment by pressure and heat of heavy mineral oils and carbon material. M. HOFSSÄSS, Assr. to INTERNAT. BERG. COMP. VOOR OLIE- EN KOLEN-CHEMIE

S'GRAVENHAGE (U.S.P. 1,729,943, 1.10.29. Appl., 14.6.22. Ger., 14.6.21).—See B.P. 213,661; B., 1924, 503.

Liquid fuel burning apparatus. ПОМАТРА, LTD., and M. TRACHTENBERG (B.P. 319,468, 18.8.28).

Decohering solid substances (B.P. 292,965).—See I. **Extraction of naphthenic acid** (U.S.P. 1,720,821).—See III. **Emulsion paints** (U.S.P. 1,691,765—8).—See XIII. **Liquors for treating leather** (B.P. 318,070).—See XV.

III.—ORGANIC INTERMEDIATES.

Catalysts for the formation of alcohols from carbon monoxide and hydrogen. IV. **Decomposition and synthesis of methyl alcohol by catalysts composed of zinc and chromium oxides.** D. S. CRYDER and P. K. FROLICH (Ind. Eng. Chem., 1929, 21, 867—871; cf. A., 1929, 153).—The close similarity between the decomposition and the synthesis of methyl alcohol previously noted with zinc-copper catalysts (A., 1928, 1112) is also evident with zinc-chromium oxide catalysts. Catalysts containing less than 50 mol.-% Cr are mixtures of zinc oxide and chromium trioxide, and can be dehydrated and reduced by methyl alcohol at 220°; above 50% the excess chromium is added as the sesquioxide, since then the higher oxide is irregularly reduced. The decomposition experiments, carried out at 1 atm. and 340°, show that a maximum yield of carbon monoxide, equivalent to 85% of the methyl alcohol converted, occurs with a catalyst containing 75 mol.-% Zn. Catalysts richer in chromium gave relatively large yields of carbon dioxide and unsaturated hydrocarbons, whilst those richer in zinc tended to produce methyl formate. The activity of the catalysts, as shown by the amount of alcohol decomposed, has also a sharp maximum at 78% Zn, a result in general agreement with that of Smith and Hawk (A., 1928, 488). In the synthetic experiments, carried out at 204 atm. and 350°, the maximum production of methyl alcohol occurred with a catalyst containing 77 mol.-% Zn, in which case 18% of the carbon monoxide was converted. The optimum temperature for a catalyst of this composition is 370—390°; the liquid product formed is shown by fractionation to be almost pure methyl alcohol. The yield of formaldehyde was almost constant throughout the decomposition experiments, and it is suggested that this is an intermediate product, which either breaks down to carbon monoxide and hydrogen or yields methyl formate as a condensation product.

T. H. MORTON.

Determination of iodoform. A JÁNSKÝ (Časopis Českoslov. Lék., 1928, 8, 215—221; Chem. Zentr., 1929, i, 2454—2455).—A study of published methods.

A. A. ELDRIDGE.

Preparation of isovaleric acid from fusel oil. A. PIOTROWSKI (Przemysł Chem., 1929, 13, 413—418).—Fusel oil obtained from the rectification of potato spirit contains about 80% of isoamyl alcohol, b.p. 128—132°. A mixture of fusel oil vapour with air (820 c.c. of air per g. of alcohol) is passed at a rate of 1.75 m./sec. through a tube containing two copper gauze spirals at 370—390°. A yield of about 75% of oily products, consisting chiefly of unreacted alcohol and of isovaleralde-

hyde, is obtained, and this product gives on oxidation with permanganate a final yield of 39.2% of isovaleric acid. Whilst this yield is smaller than that obtained by direct oxidation by permanganate of the fusel oil, the cost per unit weight of isovaleric acid is considerably lower, using the above indirect method.

R. TRUSZKOWSKI.

Manufacture of benzaldehyde. P. SHORIGIN, I. KIZBERG, N. TROITZKI, and E. SMOLYANINOVA (J. Chem. Ind. Moscow, 1929, 6, 258—260).—For the oxidation of toluene with manganese dioxide the optimal conditions consist in the use of 65% sulphuric acid, ratio of toluene to manganese dioxide 4:1, temperature 18—19°; the yield (calculated on the active manganese dioxide) was 55—56% of the theoretical. A small quantity of nitric acid has a slight accelerating action. Substitution of manganese dioxide by pyrolusite which had been finely divided or heated under pressure with sodium hydroxide was unsatisfactory. Oxidation of toluene vapour by atmospheric oxygen in the presence of a vanadium oxide catalyst gave poor yields of benzaldehyde contaminated with benzoic acid and anthraquinone. Chlorination of toluene vapour followed by treatment with sodium hydroxide and oxidation with bleaching powder and sodium carbonate at 40—60° gives favourable industrial yields of a mixture of benzaldehyde and benzoic acid. The catalytic oxidation of benzyl alcohol or benzyl chloride using a vanadium oxide catalyst is described, yields of 51% and 74%, respectively, being recorded. CHEMICAL ABSTRACTS.

Preparation and conversion [into the nitro- and amino-naphthols] of 5-nitro- α -naphthylamine. N. N. VOROSHTZOV and A. A. KULEV (Bull. Inst. Polytech. Ivanovo-Vosniesensk, 1928, 9, 87—93).—1:5-Dinitro-naphthalene was obtained from the commercial product by means of sulphuric acid or aniline, and was reduced with sodium sulphide and ammonium sulphate to the nitronaphthylamine in 40% yield. The method of Morgan and Jones for the preparation of this substance (J.S.C.I., 1923, 42, 342 r) was modified. The best yield of 5-nitro- α -naphthol obtained was 41.7%, the corresponding aminonaphthol being obtained from it in 88.1% yield. CHEMICAL ABSTRACTS.

Nitration of β -naphthol-6-sulphonic acid. N. N. VOROSHTZOV and N. M. KASATKIN (Bull. Inst. Polytech. Ivanovo-Vosniesensk, 1928, 11, 75—85).—Schaeffer's salt, when treated with nitric and anhydrous sulphuric acids, afforded 13% of 1-nitro- β -naphthol-6-sulphonic acid, and 32% of a red dinitro-compound; dinitration afforded 78% of the latter. CHEMICAL ABSTRACTS.

Meso-derivatives of anthracene and dianthryl. B. P. FEDOROV (Bull. Inst. Polytech. Ivanovo-Vosniesensk, 1928, 9, 103—109).—The action of sodium sulphite on 9-nitroanthracene affords sodium anthracene-9-sulphonate; the SO_3Na group is readily replaced by H, OH, or NH_2 . There is also formed sodium dianthryl-*ms*-sulphonate, from which *ms*-hydroxydianthryl and *ms*-aminodianthryl, m.p. 305° (decomp.), are readily obtainable. CHEMICAL ABSTRACTS.

Oxidation of lignin. HORN. **Propylene from crude oil.** DOBRYANSKI and others. **Alcohols from**

petroleum hydrocarbons. HOLZMANN and PILAT.—See II. Purification of ethyl acetate. VON MIKÓ.—See XX.

PATENTS.

Distillation and vapour-phase extraction of volatile substances [crude pyroligneous acid]. H. SUIDA (U.S.P. 1,715,313, 28.5.29. Appl., 9.2.26. Austr., 21.2.25).—Crude diluted pyroligneous acid flows through a series of distillation retorts at successively lower pressures and temperatures. Steam is injected into the first retort and the vapours from the retort ascend a tower down which a stream of extraction agent descends and carries the acetic acid with it into a collecting conduit at the bottom of the tower. Steam passes from the top of the tower to the succeeding still and thereby distils off the residual liquor from the first still under a reduced pressure. Residual liquor from the final still affords tar and a little acetic acid. R. BRIGHTMAN.

Manufacture of esters [*sec.*-butyl acetate]. H. E. BUC and W. W. CLOUGH, Assrs. to STANDARD DEVELOPMENT Co. (U.S.P. 1,726,945, 3.9.29. Appl., 23.8.23).—A mixture of about 55% of *sec.*-butyl alcohol, 45% of concentrated acetic acid, and 0.1% of sulphuric acid is distilled and the upper layer settling from the condensed liquid is refluxed continuously until stratification practically ceases; the still contents are then distilled in fractions to within a few degrees of the b.p. of *sec.*-butyl acetate and of that of acetic acid, respectively, and water is added to the latter fraction to form with the *sec.*-butyl acetate present a constant-boiling mixture which is then separated by distillation.

L. A. COLES.

Aryl alkyl esters. R. H. VAN SCHAAK, JUN., Assr. to VAN SCHAAK BROS. CHEM. WORKS (U.S.P. 1,700,960, 5.2.29. Appl., 14.9.27).—Aryl alkyl esters of phthalic acid, or, e.g., succinic, citric, or phenylmalonic acid, obtained from saturated alcohols, b.p. below 150°, are used as pyroxylin solvents. *Benzyl ethyl phthalate* has b.p. 199°/3 mm.

R. BRIGHTMAN.

Hexyl [$\alpha\delta$ -dimethylbutyl] esters. R. H. VAN SCHAAK, JUN., Assr. to VAN SCHAAK BROS. CHEM. WORKS, INC. (U.S.P. 1,702,180, 12.2.29. Appl., 20.1.28).—The esters are prepared from methylisobutylcarbinol in benzene in presence of sulphuric acid, e.g., 0.02%, as catalyst. $\alpha\delta$ -Dimethylbutyl propionate has b.p. 163°, d_{25}^{25} 0.87.

R. BRIGHTMAN.

Manufacture of *N*-[hydr]oxyethyl derivatives of 2-amino-1-oxybenzene [*o*-aminophenol]. I. G. FARBENIND. A.-G. (B.P. 292,950, 13.6.28. Ger., 27.6.27).—*o*-Aminophenol is heated at 100° with aqueous ethylene chlorohydrin in presence of an acid-binding agent (calcium carbonate). *o*- β -Hydroxyethylaminophenol, m.p. 80–81°, and *o*-*di*-(β -hydroxyethyl)aminophenol are obtained according to the proportion of chlorohydrin.

C. HOLLINS.

Manufacture of *o*-chloronitrobenzene-*p*-sulphonic acid. H. W. HILLYER, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,701,367, 5.2.29. Appl., 30.1.20).—Chlorobenzene is sulphonated with concentrated sulphuric acid at 90–100°, and the mixture is cooled to 20° and treated at 30–50° with mixed acid (nitric acid 29%, sulphuric acid 65%, water 6%). The product

may be salted out after dilution with water, or isolated by the process of U.S.P. 1,564,239 (B., 1926, 122).

R. BRIGHTMAN.

Manufacture of 6-ethoxy-2:4-dimethylquinoline. H. T. CLARKE and E. R. TAYLOR, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,701,144, 5.2.29. Appl., 7.7.23).—*p*-Phenetidine is condensed with acetone in presence of iodine at 92–96° for 36 hrs. Acetone and water are distilled off until the temperature reaches 140°, the mixture is washed with sodium hydroxide, and the *p*-ethoxyacetoneanil distilled at 165–180°/10 mm. and treated with hydrogen chloride at 180–200° for 8 hrs.

R. BRIGHTMAN.

Manufacture of naphthalene [naphthaquinone] derivatives. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 315,331, 7.2.28).— β -Benzoylpropionic acids, obtained by condensing succinic anhydride or its halogen derivatives with quinol, toluquinol, 3:6-dihydroxy-*o*-xylene, *p*-aminophenol, *p*-chlorophenol, 3:4-dichlorophenol, or other *p*-disubstituted benzene derivatives, are cyclised and at the same time oxidised by means of sulphuric acid in presence of boric acid. The condensation, cyclisation, and oxidation may be performed in one operation. The preparation of naphthazarin from quinol is described.

C. HOLLINS.

Extraction and purification of naphthenic acid. S. P. COLEMAN, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,720,821, 16.7.29. Appl., 30.7.27).—High-boiling organic acids are separated from admixed oil by conversion into the corresponding alkali salts, which are extracted with water. The free acids are then obtained by adding mineral acid, are converted again into their salts, and heated under reduced pressure to remove the final traces of oil.

R. H. GRIFFITH.

Manufacture of condensation products of the benzodiazine [quinazoline] series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,102, 31.12.26).—2:4-Dichloro- or -dibromo-quinazoline is condensed with 2 mols. (*A* and *B*) of compounds containing reactive hydrogen attached to nitrogen, oxygen, or sulphur; the 2 mols. may be identical except in the case of water, ammonia, an alkali hydrogen sulphide, or a monohydric aliphatic alcohol. Examples are described in which *A* = *B* = H-acid, J-acid, γ -acid, S-acid, *m*-aminobenzoyl-J-acid, *p*-nitroaniline-*m*-sulphonic acid (reducible to a diamine), *m*-phenylenediamine-4-sulphonic acid, 5-sulphinosalicylic acid (to give a bis-sulphone), 5-aminosalicylic acid, α -aminoanthraquinone (m.p. above 265°), aniline, sodium phenoxide, thio-*p*-cresol, α -naphthol, or 2:5-diaminobenzoic acid; *A* = H-acid, *B* = J-acid, phenylhydrazine, aniline, methylamine, naphthylamine, 2:5-diamino-4'-methylidiphenyl sulphone, ammonia, phenol, toluene-*p*-sulphinic acid, potassium methoxide, benzyl alcohol, cyclohexanol, *p*-aminobenzenesulphonic acid, dithioglycol (0.5 mol.), resorcinol (0.5 mol.); *A* = J-acid, *B* = sodium hydrogen sulphide; *A* = S-acid, *B* = sodium hydroxide; *A* = *p*-phenylenediamine (0.5 mol.), *B* = H-acid, *m*- or *p*-phenylenediaminesulphonic acid, 4:4'-diaminodiphenyl-3-sulphonic acid, ammonia, *p*-aminophenyl-5-hydroxy-7-sulpho-2-naphthylcarbamide; *A* = phenol, *B*

= sulphanilic acid; *A* = sulphanilic acid, *B* = H-acid; *A* = *m*-phenylenediamine-4-sulphonic acid, *B* = J-acid, *m*-aminofornanilide; *A* = 4-*m*-nitrobenzamido-*m*-anilic acid, *B* = 6-*m*-nitrobenzamido-*o*-toluidine-4-sulphonic acid; *A* = 0.5 mol. of 4:4'-diaminodiphenylamine-2-sulphonic acid or 4:4'-diaminostilbene-2:2'-disulphonic acid, *B* = *m*-phenylenediamine-4-sulphonic acid or 2:6-dichloro-*p*-phenylenediamine; *A* = *p*-nitroaniline-*m*-sulphonic acid, *B* = dimethylamine, aniline, *o*-anisidine, aminophenylpyrazolones, J-acid, γ -acid; *A* = 4-nitro- α -naphthylamine-6-sulphonic acid, *B* = glycol (convertible into sulphato-compound), diethyl- β -methylaminoethylamine; *A* = phenol, *B* = H-acid; *A* = 5-sulphinosalicylic acid, *B* = H-acid, ethyl-H-acid, methyl-J-acid, 1-*m*-aminophenyl-5-pyrazolone-3-carboxylic acid, *m*- or *p*-phenylenediaminesulphonic acid; *A* = 3-*p*-aminophenylureidobenzoyl-K-acid, *B* = sodium sulphite; *A* = acetyl-1:4-naphthylenediamine-6-sulphonic acid, *B* = ammonia (acetyl group removed).

C. HOLLINS.

Manufacture of unsaturated aldehydes.

A. KNORR and A. WEISSENBOERN, ASSRS. to WINTHROP CHEM. CO., INC. (U.S.P. 1,716,822, 11.6.29. Appl., 7.3.27. Ger., 2.7.26).—See B.P. 284,458; B., 1928, 634.

Production of aldehyde-sulphoxylates.

C. SCHUMANN, E. MUNCH, O. SCHLICHTING, and B. CHRIST, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,714,636—7, 28.5.29. Appl., 9.4.28. Ger., 22.4.27).—See B.P. 308,229; B., 1929, 549.

Manufacture of depolymerisation products from carboxylates of high mol. wt. F. LANGE, ASSR. to I. G. FARBEIND. A.-G. (U.S.P. 1,714,565, 28.5.29. Appl., 16.3.27. Ger., 17.3.26).—See B.P. 290,377; B., 1928, 515.

Purification of phthalic anhydride. A. O. JAEGER and F. A. CANON, ASSRS. to SELDEN CO. (U.S.P. 1,702,871, 19.2.29. Appl., 6.9.27).—See B.P. 285,017; B., 1929, 672.

Preparation of 2-aminonaphthalene-3-carboxylic acid [2-amino-3-naphthoic acid]. E. HORTZ and V. LANTZ, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,785, 6.11.28. Appl., 16.12.27. Ger., 17.12.26).—See B.P. 282,450; B., 1929, 467.

Manufacture of α -aminoanthraquinone- β -carboxylic acids. K. WILKE, ASSR. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,698,900, 15.1.29. Appl., 3.3.27. Ger., 8.3.26).—See B.P. 267,164; B., 1928, 361.

α -Di(methylamino)- α -dihydroxyanthraquinone-disulphonic acids and their manufacture. R. E. SCHMIDT and W. TRAUTNER, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,702,022, 12.2.29. Appl., 13.4.26. Ger., 17.4.25).—See B.P. 250,968; B., 1927, 743.

Production of ketones of the anthraquinone series. A. LÜTTRINGHAUS and F. KÄGER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,730,081, 1.10.29. Appl., 26.1.28. Ger., 16.2.27).—See B.P. 289,585; B., 1928, 516.

Preparation of condensation products of the benzanthrone series. G. KRÄNZLEIN and M. CORELL, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,714,677, 28.5.29. Appl., 5.3.26. Ger., 9.3.25).—See B.P. 248,791; B., 1927, 550.

Isatin derivatives and their manufacture. M. P. SCHMIDT and O. HERRMANN, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,700,814, 5.2.29. Appl., 13.9.26. Ger., 6.6.25).—See B.P. 282,863; B., 1928, 151.

Manufacture of isatins [and *N*-arylsulphonyl derivatives thereof]. K. SCHIRMACHER and K. RENN, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,698,894, 15.1.29. Appl., 27.1.27. Ger., 28.1.26).—See B.P. 265,224; B., 1928, 224.

Fatty acid peroxide (U.S.P. 1,718,609).—See XII. **Butyl alcohol and acetone by fermentation** (B.P. 319,079).—See XVIII.

IV.—DYESTUFFS.

Synthesis of alizarin. V. I. MINAEV and B. P. FEDOROV (Bull. Inst. Polytech. Ivanovo-Vosniesensk, 1928, 9, 95—101).—The method of G.P. 241,806 is modified; 5 pts. of sodium hydroxide, 1 pt. of anthraquinone, and water to produce a 28% concentration of sodium hydroxide are kept at 180—200° for 18—30 hrs. after a period of 6 hrs. at 130—138° in the absence of sodium nitrate. Meso-derivatives of anthracene give poor yields of alizarin if the fusion is carried out without addition of sodium sulphite; the presence of sodium nitrate is necessary for the mononitro- but unnecessary for the dinitro-derivative. CHEMICAL ABSTRACTS.

Spectroscopic examination of food dyes. B. V. HEATH (J. Soc. Chem. Ind. Victoria, 1929, 29, 159—165).—A 1:200 aqueous solution of the dye is examined with a direct-vision spectroscope, and the position of the absorption bands noted with reference to the Fraunhofer lines, an accuracy of 50 Å. sufficing. Further readings are then taken after halving the concentration at each stage, and the results plotted. Similar absorption curves are made for solutions of the dye in 10% sulphuric acid and 10% sodium hydroxide, the series of curves thus obtained being characteristic for a particular dye. In order to identify the dye present in a food product, 20 g. of the latter are finely ground and extracted several times with methylated spirit. The residue is macerated with water, an equal volume of methylated spirit is added, and the solution filtered. The filtrate is concentrated, a large excess of absolute alcohol added to precipitate starch, and the filtrate from this added to the original extract. The united extracts are evaporated to dryness, the residue is dissolved in water, and the absorption curves are mapped and compared with those of known dyes. An approximate estimate of the amount of dye present can also be obtained in this way. The method is not suitable for the examination of black dyes, nor are those which show considerable general absorption. H. F. HARWOOD.

PATENTS.

[Manufacture of] vat dyes of the anthraquinone-acridone series. O. UNGER and G. BÖHNER, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,913, 6.11.28. Appl., 25.10.26. Ger., 29.10.25).—See B.P. 260,588; B., 1928, 225.

Azo dyes and their manufacture. E. HOFFA, E. THOMA, and H. HEYNA, ASSRS. to GRASSELLI DYESTUFF CORP.

STUFF CORP. (U.S.P. 1,698,884, 15.1.29. Appl., 11.7.27. Ger., 21.7.26).—See B.P. 296,473; B., 1928, 849.

Monoazo dyes [for acetate silk] derived from naphthylaminocarboxylic [aminonaphthoic] acids. L. LASKA and F. WEBER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,702,832, 19.2.29. Appl., 3.5.26. Ger., 8.5.25).—See B.P. 275,307; B., 1927, 776.

Manufacture of indigoid dyes. E. HOFFA, H. HEYNA, and F. MÜLLER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,730,209, 1.10.29. Appl., 30.11.26. Ger., 4.12.25).—See B.P. 262,457; B., 1927, 772.

Dyes for leather (B.P. 316,822).—See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Structure of vegetable fibres. I. Spiral structure of cotton, wood, manila, straw, bamboo, and sugar cane (bagasse) fibres. M. NAKANO (J. Cellulose Inst., Tokyo, 1929, 5, 202—203).—The spiral structure (spiral cuticle) and the bead necklace effect on swelling in cuprammonium solution, which are both shown by cotton and wood fibres, are obtained with many other vegetable fibres such as manila, straw, bamboo, and sugar cane (bagasse). The spiral structure was successfully shown by swelling the fibres after weak xanthation. B. P. RIDGE.

Hydrolysis of wool by sodium sulphide. II. W. KÜSTER and W. IRION (Z. physiol. Chem., 1929, 184, 225—240; cf. A., 1928, 535).—The acetic acid precipitate from the sodium sulphide hydrolysate of wool contains less histidine than the original wool. Ammonia is produced during hydrolysis, although histidine itself is not deaminated by sodium sulphide. An attempt to prepare a histidine peptide by linking up benzoylhistidine methyl ester with hydrazine hydrate gave a crystalline *azlactone*, m.p. 215°. Of arginine, tyrosine, cystine, and asparagine, only the last splits off ammonia with sodium sulphide. No cystine could be isolated from the acetic acid precipitate, although the latter contained sulphur. This was shown to be present as a new dibasic *diamino-acid*, $C_7H_{14}O_4N_2S$, which crystallised from the neutralised hydrolysate after the tyrosine. When recrystallised from ammonia it formed tetragonal spheroids, m.p. 275° (decomp.). Further attempts to isolate this substance were unsuccessful.

J. H. BIRKINSHAW.

Decomposition of maize stalks by chlorine. E. HORVÁTH (Papier-Fabr., 1929, 27, 626—639).—The cut-up stalks are treated with warm dilute sodium hydroxide solution, then with chlorine, and finally with cold sodium hydroxide solution. At the same temperature the amount of sodium hydroxide used up in steeping the material depends on the concentration of the liquor and on the time of treatment. With $\frac{1}{2}$, 1, or 2% solutions the reaction is completed in 90 min. If equal amounts of material are treated under the same conditions with equal quantities of 1 or 2% sodium hydroxide liquor, the products, after chlorination, consume almost equal amounts of sodium hydroxide, even if the amount of chlorine used is increased 8-fold. The yield obtained depends on the conditions of steeping and chlorination. With increasing

chlorine consumption the yield decreases quickly at first and then gradually. In all the experiments performed, under different conditions, the ash and silica contents of the final products were approximately the same. Loss of lignin increases with increasing chlorine consumption quickly at first, but subsequently reaches an approximately constant value. It is better to use a more concentrated sodium hydroxide solution in the first steeping process, followed by low chlorine consumption, than a dilute solution and high chlorine consumption, since in the former case greater dissolution of lignin results. B. P. RIDGE.

Removal of fermentation-restraining humic substances from the hydrolysis products of hemicelluloses. C. SCHMIDT, M. ATTERER, and H. THALER (Cellulosechem., 1929, 10, 153—155).—Humic substances, formed during the acid hydrolysis of hemicelluloses, which render completely inaccurate the determination of the hexoses by fermentation, may be removed by treatment with a dilute aqueous solution of chlorine dioxide. This treatment is shown not to affect the fermentation value of hexose in solution. The hemicelluloses from archegoniates and phanerogams (cf. Schmidt and co-workers, A., 1929, 1113) may be hydrolysed by refluxing with 5% oxalic acid; the resulting solution is neutralised, treated with a 1% solution of chlorine dioxide, after 12 hrs. extracted with ether, and again neutralised. The hexoses may now be determined by fermentation with *Schizosaccharomyces Pombe* or *Saccharomyces Vördermannii* (loc. cit.). To hydrolyse the hemicellulose from the wood fungus (*Fomes fomentarius*) it is necessary first to treat with 75% sulphuric acid at ordinary temperature, and then to reflux with 5—10% acid for 2½ hrs. The hexose may then be determined as described above. The hexose contents of hemicelluloses from different sources are given: *Pteridium aquilinum* 10.3%, rye straw 9.0%, *Fagus sylvatica* 8.3%, *Fomes fomentarius* 81.0%.

T. H. MORTON.

PATENTS.

Manufacture of artificial textile filaments or fibres. H. DREYFUS (B.P. 317,097—8, 6.2.28).—(A) Hollow filaments of good strength and capable of being drawn out to low deniers may be dry-spun from solutions of cellulose derivatives in organic solvents by introducing into the spinning liquor one or more high-boiling solvents or plasticisers in quantities varying from 3 to 80% (on the weight of the cellulose ester) and depending on the volatility of the high-boiling solvent. Suitable compounds include diacetone alcohol, acetophenone, di- and tri-acetin, ethyl phthalate, alkyl ethers of ethylene glycol, etc. The residual plasticising agent may be removed from the spun filaments by extraction with water or other suitable solvents. For a 24.8% solution of cellulose acetate in 3:1 acetone-diacetone alcohol 83° is a suitable spinning temperature. (B) The same result may be achieved by spinning a solution of cellulose acetate in a mixed solvent the components of which differ in their b.p. by 35—100° or more, the temperature of spinning being below or not substantially above the b.p. of the higher-boiling component of the spinning solution. The less is the

difference between the b.p. of the components of the solvent mixture the greater should be the proportion of the lower-boiling constituent and the lower may be the spinning temperature. Thus hollow filaments are obtained by spinning a 20.7% solution of cellulose acetate in a 1:3 acetone-acetaldehyde mixture at 65°, whilst at lower spinning temperatures cellular filaments result.

D. J. NORMAN.

Packing material for soft soap. H. FREHRS (B.P. 319,517, 15.10.28).—The packing paper is coated with a layer, impervious to gases and liquids, containing lime or a lime salt (*e.g.*, waxes or hard fats with calcium carbonate); on contact with the soft soap an insoluble lime soap layer, impervious to soft soap, is produced.

E. LEWKOWITSCH.

Manufacture of paper. A. L. KENNEDY, ASSR. to PLASTIC, INC. (U.S.P. 1,730,009, 1.10.29. Appl., 25.3.27).—See B.P. 287,538; B., 1928, 668.

Grinding-stones used on wood-pulping machines. H. AKEROYD (B.P. 319,469, 22.8.28).

Stabilisation of lead tetra-alkyl (U.S.P. 1,724,640).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Reflecting power of coloured fabric. CUNLIFFE and FARROW.—See XXI.

PATENTS.

Dyeing of cellulose derivatives. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 308,242, 13.12.27. Cf. B.P. 307,813; B., 1929, 639).—Cellulose esters and ethers are dyed with alkylthiolanthraquinones, *e.g.*, ethylthiolanthraquinone (yellow), 1:4-di(ethylthiol)-anthraquinone (orange), 1-amino-4-ethylthiolanthraquinone (bordeaux), 1-methylamino-2-ethylthiolanthraquinone (pink), 1-amino-2-isopropylthiolanthraquinone (red), 1-methylamino-2-isobutylthiolanthraquinone (pink).

C. HOLLINS.

Dyeing of leather and manufacture of dyes for use therein. J. C. BOTTOMLEY & EMERSON, LTD., and W. D. EARNSHAW (B.P. 316,822, 29.10.28).—Dyes produced by coupling 2 mols. of *o*- or *p*-toluidine, *m*- or *p*-xylydine, or their sulphonic acids, or 1 mol. each of any two of these substances with 1 mol. of resorcinol or α - or β -resorcylic acid in the presence of caustic soda are suitable for dyeing leather in brown shades; those dyes containing sulphonic groups are more soluble in water and yield deeper shades. Dyeing is effected at 45° in an acid liquor on bark-, chrome-, or alum-tanned leather.

A. J. HALL.

Treatment of textile materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,948, 14.9.27).—Hydroxyalkylamines, *e.g.*, "triethanolamine" [tri-(β -hydroxyethyl)amine], cyclohexyl- β -hydroxyethylamine, and cyclohexyldi-(β -hydroxyethyl)amine, are added to aqueous baths for the cleansing, dyeing, carbonising, stripping, scouring, or bowking of textiles (in absence of gums). (Cf. B.P. 270,293; B., 1928, 704.) C. HOLLINS.

Delustring of cellulose acetate silk, films, and the like. SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 316,169, 24.4.28).—Cellulose

acetate silk is treated so as to absorb the leuco-compounds of anthraquinone and such of its derivatives as are colourless in the fully oxidised state, and is then suitably oxidised. The fully oxidised anthraquinone compound is thereby deposited within the silk, which thus becomes reduced in lustre; the resultant delustring effect is fast to soaping. Anthraquinone and 2-chloro-anthraquinone are particularly suitable, and are applied from an alkaline reducing liquor containing ammonia and sodium hyposulphite. The silk may be dyed and delustred simultaneously if use is made of dyes such as Hydron Pink FF, which are capable of colouring cellulose acetate under such conditions.

A. J. HALL.

Delustring of cellulose acetate silk materials and of other materials containing cellulose acetate. SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 316,638, 28.4.28).—The material containing cellulose acetate is treated with aqueous suspensions of esters of aliphatic acids, particularly ethyl oxalate, and then with ammonia, whereby oxamide is formed within the material, thus reducing its lustre and giving to it a scroop handle. The delustring is fast to soaping, and the process can be readily adapted for dyeing or printing methods.

A. J. HALL.

Production of resists in dyeing with vat dyes. J. HÖPKER, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,730,211, 1.10.29. Appl., 30.4.28. Ger., 6.5.27).—See B.P. 290,177; B., 1929, 849.

Preparation of fulling liquors and emulsions [for softening of wool etc.]. K. DAIMLER, G. BALLE, and F. JUST, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,715,410, 4.6.29. Appl., 28.1.26. Ger., 28.1.25).—See B.P. 246,867; B., 1927, 451.

Dye baths. T. PARKINSON (B.P. 319,432, 6.7.28).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Detection of hydrogen peroxide with potassium permanganate solution according to D.A.B. VI. G. FRERICH (Apoth.-Ztg., 1929, 44, 225—226; Chem. Zentr., 1929, i, 1974—1975).—A modification of the procedure is recommended.

A. A. ELDRIDGE.

Manufacture of manganese borate. V. V. ERIN (J. Chem. Ind. Moscow, 1929, 6, 254).—A mixture of finely-divided pyrolusite (9 pts.) and non-caking coal (1 pt.) is heated at 800—900°; the yield of manganous oxide is 85%. The hot mass is boiled with a 5—10% excess of 20% sulphuric acid for 1 hr., the solution is filtered through asbestos, the iron oxidised by boiling the solution with a 10% excess of nitric acid, and precipitated at 50° with sodium carbonate solution. The liquid, after settling, is filtered and treated with 4% borax solution, the end-point being denoted by the disappearance of cloudiness when a manganous salt solution is added to the filtered liquid. After settling, the precipitate is centrifuged, filter-pressed, and dried at 50°, then at 110°.

CHEMICAL ABSTRACTS.

Oxygen and hydrogen in industry. W. P. DOBSON and A. S. L. BARNES (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 11 pp.).—Methods of

production and fields of utilisation of oxygen and hydrogen are reviewed. Further possible outlets for these gases are considered and the prospects of the electrolytic process for producing them discussed.

H. J. T. ELLINGHAM.

Determination of phosphorus in "Phosphor solutus" according to D.A.B. VI. W. BÖTTGER (Apoth.-Ztg., 1928, 43, 1551—1553; Chem. Zentr., 1929, i, 1845).—Determinations based on titration of the acid liberated from an iodide-iodate mixture gave concordant results; iodometric determinations gave results 25% higher.

A. A. ELDRIDGE.

Molybdenum-blue method for micro-determination of phosphate and arsenate ions. G. DENGÈS (Mikrochem., 1929, Pregl Fest., 27—45).—An acid solution of ammonium molybdate gives in presence of phosphate or arsenate ions on treatment with reducing agents stable blue compounds of the composition $(4\text{MoO}_3, \text{MoO}_2)_2, \text{XO}_4\text{H}_3$, where $\text{X} = \text{P}$ or As . A colorimetric method, based on the above reaction, has been devised for the determination of phosphate in wines and other beverages; the determination is carried out on the original sample without previous destruction of organic matter, so that the figure obtained represents the mineral phosphate present. The total phosphate can be found by carrying out a second determination after removal of organic matter. The method is also applicable to the determination of phosphate in urine, milk, and other physiological products, as well as to fertilisers and mineral phosphates. Arsenate may be determined in a similar manner.

H. F. HARWOOD.

Rapid determination of nitrogen. F. M. WIENINGER and M. LINDEMANN (Woch. Brau., 1929, 46, 406—407).—The methods of Lundin and Ellburg (B., 1929, 596) and Kühl and Gottschalk (Z. ges. Getreidewesen, 1929, 114—115) are accurate and rapidly completed, but they require as much attention as the ordinary Kjeldahl method. The use of phosphoric acid in the digestion mixture causes appreciable loss of material from the flasks. Until a rapid accurate method for the determination of moisture is devised such processes are of little advantage in cereal analyses, and in this direction the "D.K." (condenser) method (B., 1929, 373) appears promising.

F. E. DAY.

Peat as a source of nitrogen. LOGVINOVA. Fuller's earth and acid-treated earths. DAVIS and MESSER.—See II. Catalysts for methyl alcohol synthesis. CRYDER and FROLICH.—See III. Selenium and its compounds for ruby glass. KRAK.—See VIII. Precipitation of lead and copper from solutions. OLDRIGHT and others.—See X. Electrolysis of water. SCHNURMANN.—See XI. Antiseptic value of chlorine. HOWARD and others.—See XV. Iron albuminate. OBERHARD.—See XX.

PATENTS.

Catalytic reactions. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 315,900, 13.4.28).—Catalysts for reactions in which hydrogen is employed under pressure, e.g., the synthesis of ammonia, are, prior to the reaction, irradiated by Röntgen rays, rays of radioactive substances, etc. in the presence of inert gases

or of gases, vapours, or liquids of the same nature as those which are to take part in the catalytic reaction, the catalysts being in the same physical and chemical state as that in which their activity is to be exerted.

W. G. CAREY.

Obtaining sodium chloride. G. B. BURNHAM, Assr. to BURNHAM CHEM. CO. (U.S.P. 1,701,295, 5.2.29. Appl., 16.11.25).—The brine is brought to supersaturation by, e.g., atmospheric evaporation, and circulated upwardly to a conical tank and through crystals deposited on a perforated partition a short distance above the bottom (apex) of the tank. Liquor overflowing at the top of the tank is returned to the evaporating pond, and crystals fall through the partition into a vertical collecting conduit parallel with the inlet conduit and are removed to storage by a conveyor.

R. BRIGHTMAN.

Refining of borax. C. F. RITCHIE and W. A. GALE, Assrs. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,724,420, 13.8.29. Appl., 27.1.25).—Liquors containing phosphates and borates are treated with a reagent which by reducing the p_{H} value increases the solubility of the phosphates and thus facilitates the crystallisation of borates free from phosphates.

L. A. COLES.

Manufacture of sodium xanthate. W. HIRSCHKIND, Assr. to GREAT WESTERN ELECTRO-CHEM. CO. (U.S.P. 1,701,264, 5.2.29. Appl., 28.11.24).—Ethyl alcohol (91% denatured) is added to 46% sodium hydroxide solution, and carbon disulphide is added at 25—30°. The crystalline xanthate ($2\text{H}_2\text{O}$) is centrifuged and the mother-liquor treated cold with 55% potassium hydroxide to give a further precipitate of potassium xanthate. The final mother-liquor containing, e.g., 16% of caustic alkali, is useful as an auxiliary froth-floatation agent.

R. BRIGHTMAN.

Manufacture of alkali xanthates. T. W. BARTRAM and H. P. ROBERTS, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,718,937, 2.7.29. Appl., 8.11.26).—Carbon disulphide is added during 50—1000 min. to alcoholic alkali (1:1 mol.) at 0—50°, and the mixture is heated below 70° to decompose by-products.

C. HOLLINS.

Manufacture of inorganic [iron] oxides [for pigments]. E. H. MCLEOD, Assr. to AULT & WIBORG CO. OF NEW YORK (U.S.P. 1,726,851—2, 3.9.29. Appl., 26.10.22).—(A) A solution of ferrous sulphate, e.g., waste pickling liquor, is oxidised with bleaching powder and treated with sodium carbonate to precipitate a mixture of calcium carbonate and ferric hydroxide. The precipitate is agitated with a further quantity of ferrous sulphate to produce, by interaction with the calcium carbonate and ferric hydroxide, a mixture of ferrosiferrous oxide and calcium sulphate of any desired tint. (B) Burnt pyrites cinder is dissolved in hydrochloric acid, sodium carbonate is added in excess, and the mixture treated at 82° with ferrous sulphate.

A. R. POWELL.

Stabilisation of lead tetra-alkyl and its compositions. W. S. CALCOTT and A. E. PARMELEE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,724,640, 13.8.29. Appl., 19.6.28).—Primary, secondary, or tertiary alkylamines or their derivatives are used; e.g.,

1% of methylamine or diethylamine is added to a mixture of lead tetraethyl, ethylene dibromide, and chloronaphthalene.

R. BRIGHTMAN.

Manufacture of silica or masses containing the same. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,675, 16.3.28. Addn. to B.P. 270,040; B., 1927, 481).—A decomposable silicon compound and a decomposing agent are mixed at a low temperature in a single pouring action within a few seconds or by simultaneous confluence in such concentration that a non-alkaline silica sol is produced containing at least 9 g. of silica/100 c.c. of total liquid. W. G. CAREY.

Production of gas strong in sulphur dioxide. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,724,421, 13.8.29. Appl., 26.3.23).—Sulphur burner gases are passed through a confined space in which they are brought in contact with non-reacting surface material and a countercurrent of water, and the solution obtained is subsequently heated and treated in a confined space in the presence of non-reacting material with a countercurrent of the residual gas from the first treatment to yield a gas mixture with a high sulphur dioxide content.

L. A. COLES.

Production of phosphorus. W. H. WAGGAMAN and H. W. EASTERWOOD, Assrs. to VICTOR CHEM. WORKS (U.S.P. 1,728,948, 24.9.29. Appl., 17.1.24).—Briquetted mixtures of natural phosphates, silica, and a solid reducing agent are heated in continuous rotary kilns to not above 1500°, the composition of the mixture being such as not to be readily fusible at this temperature.

C. A. KING.

Production of sodium nitrate. R. GRIESSBACH and K. RÖHRE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,696,197, 25.12.28. Appl., 22.9.27. Ger., 14.10.26).—See B.P. 290,860; B., 1928, 522.

Purification of chemicals [used in preparation of per-salts, etc.]. W. WEBER and M. JACOBI (U.S.P. 1,722,871, 30.7.29. Appl., 7.9.27. Ger., 24.12.26).—See B.P. 282,302; B., 1928, 157.

Protection of pipes (U.S.P. 1,700,995—6). Treatment of ores for separation of salts (B.P. 318,301).—See X. Electrolytic cells (B.P. 292,130).—See XI. Base-interchange agent (B.P. 286,307).—See XXIII.

VIII.—GLASS; CERAMICS.

Volatility of selenium and its compounds in the manufacture of ruby glass. J. B. KRAK (J. Amer. Ceram. Soc., 1929, 12, 530—537).—Glass batches were melted in a gas-fired furnace to determine the amount of selenium lost in making ruby glass, to investigate the conditions for producing "pot" ruby, and to discover whether the use of selenium compounds decreases the loss of selenium. About 75% of the selenium added to a batch was lost by volatilisation, this amount varying with the composition of the batch. Although sodium and barium selenite are stable at 950°, no saving of selenite was effected by substituting either of these compounds for the selenium itself. Methods of preparing sodium and barium selenites and of determining selenium in glass are described.

F. SALT.

Viscosity measurements in glass. H. R. LILLIE (J. Amer. Ceram. Soc., 1929, 12, 516—529).—For low

viscosities (10—10⁴ poises) the method already described (*ibid.*, 505) was used. For higher viscosities (10³—10⁸) the same apparatus was used, but the outer cylinder was turned through a small angle and stopped, the return of the inner spindle towards its point of rest being then timed. The tests were carried out in a molybdenum-wound furnace, in which temperatures between 1400° and 1500° were attained on 110 volts. The glass samples were in the form of cylinders, 5½ in. long and 2¼ in. in diam. The results obtained are compared with those published by English, Stott, and by Washburn.

F. SALT.

Deformation study of cobalt oxide-alumina-silica mixtures. T. M. FELTON (J. Amer. Ceram. Soc., 1929, 12, 548—551).—The study was undertaken to investigate the tendency of cobalt oxide to "spread" in glazes. The various mixtures were made up into cones and the temperatures of deformation and of fusion were noted. The deformation eutectic temperature of the cobalt oxide-silica series was 1325°, the composition being 32% SiO₂ and 68% Co₃O₄. The composition of the alumina-cobalt oxide deformation eutectic was approx. 10% Al₂O₃ and 90% Co₃O₄; that of the cobalt oxide-alumina-silica system was 38% SiO₂, 20% Al₂O₃, and 42% Co₃O₄, the temperature being 1200°. The addition of silica in amounts up to 70% greatly reduced the melting temperatures of mixtures of alumina and cobaltocobaltic oxide. In the presence of alumina and silica, cobalt oxide is an active flux, which may account for the "spreading" tendency in glazes.

F. SALT.

Capillary suction of some ceramic materials. A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1929, 12, 585—595).—A simple experiment with a porous pot, a graduated glass tube, and a flask containing mercury is described, by means of which capillary suction can be demonstrated. Evaporation from the surface of a saturated clay causes compression of the material with the development of tension in the pore water and a retreat of the water-to-air contact surface from the outside of the material toward the interior. Capillary suction is defined as the maximum difference which can exist between the pressure of the pore water and the pressure of the air in contact with the material without causing the water-to-air contact to retreat to the interior. Apparatus is described for determining this maximum pressure by using air at pressures greater than atmospheric and keeping the pore water at atmospheric pressure. The capillary suction of flint, felspar, ball clay, kaolin, and mixtures of these were determined. The addition of non-plastic material to clays caused a marked decrease in capillary suction. The figure obtained for ball clay was three times that for kaolin. Owing to capillary suction, water under atmospheric pressure was made to enter a bomb in which the gas pressure was 200 lb./in.²

F. SALT.

Burning of calcareous clays. O. LECHER (Chem.-Ztg., 1929, 53, 669—671).—In the form of gypsum, the presence of calcium in a clay is definitely undesirable for the production of ceramic ware. Calcium is usually present as the carbonate, and in this form its effect depends largely on its physical condition. It is practically impossible to grind coarse nodules of lime in a

clay to sufficient fineness to render them harmless. When the clay is fired, lime particles, even as small as 1 mm. and less, are not completely "dead-burned," but are partially converted into quicklime. The most effective way of removing coarse grains of limestone is by washing the clay. If in a finely-divided state, lime may be present in considerable amount in clays used in making face-bricks and tiles, stove tiles, etc. It largely destroys the colouring effect of iron oxide in such clays fired to high temperatures, but the bricks produced have great crushing strength and density. The firing of clays rich in lime is somewhat difficult, because the softening point and the m.p. of the clay are near to each other. The temperature of the kiln must therefore be reasonably uniform; hence down-draught kilns are not suitable, and some modification of the annular kiln is necessary to fire these clays successfully. F. SALT.

Torsional apparatus for measuring plasticity [of clays]. C. W. PARMELEE and R. D. RUDD (J. Amer. Ceram. Soc., 1929, 12, 552—555).—Methods and apparatus previously described (cf. Talwalkar and Parmelee, B., 1928, 193) were critically examined and altered to reduce variations due to personal manipulation. The specimens were made in squeeze moulds instead of by hand. The shape of the test-piece was changed so that the ratio of the stress in the outer fibres of the square end-section to that of the circular portion was 1:3.8. The method of applying the load by adding weights to a suspended pan was abandoned in favour of a flowing-water device, which applied the load continuously. Data obtained on a ball clay and a china clay are briefly analysed. F. SALT.

Liquefaction of difficultly workable fireclays. P. P. BUDNIKOV, S. A. SCHICHAREVITSCH, and I. G. SCHACHNOVITSCH (Kolloid-Z., 1929, 49, 174—178).—Measurements have been made of the rate of flow of clays diluted with water with the addition of varying quantities of water-glass. An optimum concentration of water-glass exists for each dilution with water. The minimal amount of water required was with a concentration of water-glass of 0.1—0.125*N*. Hard water must be given a preliminary softening. E. S. HEDGES.

PATENTS.

Tunnel kiln. W. L. HANLEY, JUN. (U.S.P. 1,720,550, 9.7.29. Appl., 11.9.25. Renewed 11.12.28).—The kiln comprises two consecutive firing zones, in addition to the preheating and cooling zones. Each firing zone is heated by separate, directly-fired furnaces, which are on opposite sides of the kiln and can be regulated so as to give a maximum temperature at a point between the two firing zones. The combustion products enter the latter directly and are withdrawn through a series of ports. Two parallel conveyors, movable in opposite directions throughout the entire length of the kiln, are provided. F. G. CLARKE.

Refractory product. J. T. LITTLETON, JUN., Assr. to CORNING GLASS WORKS (U.S.P. 1,728,350, 17.9.29. Appl., 8.6.23).—Suitable material is melted and poured into a mould, unmelted material of approximately the same composition in granular form being then added to the molten material, which is thus cooled, and a heterogeneous crystalline mass is formed. F. SALT.

Repairing of refractory walls. D. W. ROSS and J. M. LAMBIE (U.S.P. 1,727,675, 10.9.29. Appl., 27.7.25. Renewed 12.4.29).—A mixture of a granular aluminous material, a flux, and a bonding clay is applied to the inner surface of glass-tank walls, and the tanks are heated to the point where mullite crystals are formed. F. SALT.

IX.—BUILDING MATERIALS.

Florescence [in building materials]. W. A. MCINTYRE and R. J. SCHAEFFER (Trans. Ceram. Soc., 1929, 28, 363—380).—The term "florescence" is used to cover the general phenomenon of the crystallisation of soluble salts in building materials, the term "efflorescence" being restricted to crystallisations on the surface. The word "cryptoflorescence" is suggested to describe the salts concealed in the body of the material. Most florescences consist of the sulphates of the alkalis and alkaline earths, but they vary in composition within wide limits. The sources of soluble salts which cause florescence are indicated, and the forces causing disintegration are discussed. The chemical action of atmospheric acids in dissolving out certain constituents weakens the structure; salts which crystallise with a decrease in volume may exercise a powerful disruptive force by axial growth of the crystals. The expansion of sodium sulphate on hydration may be a contributory cause of disruption, and soluble salts, such as sodium sulphate, may have a disruptive effect by accelerating the hydration of calcium sulphate present in the body of a brick. The use of chemicals to remove florescences from brickwork is deprecated, periodic washing with water being advocated. F. SALT.

Tensile autogenous heating of Portland cement mixtures. H. J. GILKEY (Engineering, 1929, 128, 450—452).—Broken briquettes made and used for tensile strength tests generally "heal" if the broken parts are refitted and kept in position by suitable binding, and allowed to remain in water. The tensile strength of such healed briquettes is seldom more than 10% of the original strength, but much higher recoveries have been noted. Healing is due to formation of crystalline silicates, hydrates, and carbonates of calcium which form across the break. S. I. LEVY.

Respiratory diseases in a Portland cement plant. L. R. THOMPSON and D. K. BRUNDAGE (J. Ind. Hygiene, 1929, 11, 266—277).

Corrosion of lead pipes in cement etc. BADO.—See X.

PATENTS.

Manufacture and use of oxysalt [calcium oxychloride] composition. C. CATLETT (U.S.P. 1,726,472, 27.8.29. Appl., 26.7.23).—A mixture of calcium chloride and hydroxide is ground with 2% or less of an oleaginous material, e.g., cottonseed oil or beef tallow. The substantially dry product, which is not sensitive to atmospheric conditions, is used to regulate the setting of Portland cement. F. G. CLARKE.

[Bituminous mixture for] the making of roads, paths, etc. J. T. HINES (B.P. 316,017, 31.7.28).—Stone is sprayed with a fluxing oil containing sulphur (2—10%) and then mixed mechanically with commercial bitumen.

and Trinidad bitumen containing clay in a colloidal state; fine mineral filler is subsequently added. Alternatively, the fluxing oil and bitumens are mixed at a suitable temperature and the stone is treated with this mixture.

W. G. CAREY.

Hydraulic cement. C. PONTOPPIDAN (B.P. 293,035, 23.6.28. U.S., 30.6.27).—See U.S.P. 1,722,480; B., 1929, 816.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Heat and material balance of some melts in the Brackelsberg furnace. P. BARDENHEUER and K. L. ZEYEN (Stahl u. Eisen, 1929, 49, 1393—1398).—The advantages of the Brackelsberg coal-dust-fired furnace for melting cast iron and the economics of the process are discussed. The metal becomes coated with a thin layer of slag directly it melts so that absorption of oxide and gases and oxidation of the metal are avoided. When tapped into the ladle the metal remains quiet and the castings are free from pores and internal cavities. Owing to its high degree of fluidity the metal is especially suitable for making thin-walled castings and articles of intricate shape. The consumption of coal dust with 8.2% of ash and a calorific value of 7309 kg.-cal./kg. varied from 9.6 to 14.3% of the weight of metal melted, the slag produced was 5—6—9.2%, and the reduction in the carbon content of the metal about 0.5%. Of the total heat generated, 30—40% was taken up by the iron, 2.5—4.5% by the slag, and 55—45% by the flue gases, the loss by incomplete combustion being 4—8%. Full heat-balance sheets of the three tests made are reproduced.

A. R. POWELL.

Microscopical study of cast iron and its relation to the foundry. E. HOWELL (J. Soc. Chem. Ind. Victoria, 1927, 27, 1397—1407).—Improved physical strength of cast iron is aimed at by both the Lanz and the Emmel-Thyssen processes, the former restraining the formation of graphitic carbon by adopting a low-silicon iron (1% Si) and casting in preheated moulds, and the latter depending on the superheating of the metal together with low carbon content. Addition of steel scrap to the cupola charge does not decrease the total carbon below 3% owing to absorption of carbon from the fuel; for this reason good free-burning coke should be used to obtain a finely-divided graphite together with a close-grained pearlitic structure in the casting.

C. A. KING.

Testing of permanent-magnet steel. W. OERTEL (Stahl u. Eisen, 1929, 49, 1449—1454).—A detailed description is given with reference to photographs and wiring diagrams of the construction, standardisation, and method of operation of a new Bosch magnet-testing apparatus of the type in which a thick soft-iron yoke with a revolving coil is used for measuring the intensity of the lines of force.

A. R. POWELL.

Effect of alumina on the properties of steel-furnace slags. G. MARS (Arch. Eisenhüttenw., 1929—30, 3, 103—116; Stahl u. Eisen, 1929, 49, 1339—1340).—The replacement by alumina of part of the lime in the refining slag of a basic electric furnace and of a Siemens-

Martin furnace reduces the dephosphorising action almost to zero, especially when less than 20% of ferrous oxide is present. On the other hand, an aluminous slag effects a very efficient desulphurising and deoxidising action owing to the formation of aluminium carbide which dissolves in the molten steel, giving up its carbon to the iron and producing aluminium sulphide and oxide which enter the slag; the desulphurising action is, however, obtained only in the electric furnace and not in the open-hearth furnace. Owing to the affinity of alumina for ferrous oxide at high temperatures, the addition of alumina to open-hearth slag results in a more efficient removal of ferrous oxide from the molten iron and thus reduces the tendency to hot-shortness. As a result of these experiments a mixture of 90% of lime and 10% of alumina is recommended as a basic lining for steel-refining furnaces.

A. R. POWELL.

Determination of manganese in steel by Wald's method. J. KASSLER (Chem.-Ztg., 1929, 53, 719).—The following modification of Volhard's method as modified by Wald is recommended for the determination of manganese in chromium, nickel-chromium, and high-speed tool steels containing less than 0.2% Co. A sample of the metal (2.75 g.) is dissolved in 50 c.c. of 1:5 sulphuric acid, the solution is oxidised with 10 c.c. of 1:1 nitric acid, and the iron precipitated with zinc oxide. After dilution to 250 c.c. an aliquot portion (100 c.c.) is filtered through a dry paper, treated with 20 c.c. of 25% sodium acetate solution, boiled, and titrated with a solution containing 0.79 g./litre of permanganate until a slight excess is present, which is then reduced by the cautious addition of 10% alcohol. The aldehyde formed is expelled by boiling for 5 min., and the solution cooled and treated with 10 c.c. of 5% potassium iodide solution and 20 c.c. of hydrochloric acid; the liberated iodine is then titrated with thiosulphate and the amount due to the manganese added as permanganate is deducted. The results are correct to $\pm 0.005\%$ Mn.

A. R. POWELL.

Detection of sulphide segregations in the presence of phosphide segregations [in iron and steel] by Feigl's reagents. M. NIESSNER (Arch. Eisenhüttenw., 1929—30, 3, 157—161; Stahl u. Eisen, 1929, 49, 1378—1379). The mercuric chloride method for detecting sulphide and phosphide segregations in iron and steel does not give characteristic black prints for sulphide when the amount of sulphur present is small, owing to the formation of orange to yellow sulphochlorides of mercury instead of black mercuric sulphide. These spots can be distinguished from phosphide spots on the dried print by placing on them a drop of a solution 0.1N with respect to potassium iodide and 0.2N with respect to sodium azide. If sulphur is present small bubbles of nitrogen will form in the drop within a few seconds (cf. Feigl, A., 1928, 1106); phosphides give no reaction.

A. R. POWELL.

Soil corrosion studies, 1927—8. K. H. LOGAN (Bur. Stand. J. Res., 1929, 3, 275—302. Cf. B., 1929, 56).—Further data on ferrous pipe and lead cable-sheath buried in various soils in 1922, and unearthed in 1928, are given. Previous conclusions are confirmed, the rate of pitting and of loss of weight decreasing with

time. The later corrosion is largely the result of general spreading of the corroded areas, penetration progressing more slowly than loss in weight. Part of the corrosion is due to electrolytic action when a pipe passes through two different soils. No conclusions as to the relative merit of different materials are yet justified.

C. J. SMITHELLS.

[Corrosion of] lead pipes embedded in cement or lime mortars. A. A. BADO (Anal. Assoc. Quím. Argentina, 1929, 17, 5—12).—Instances of corrosion of lead pipes set in damp mortar are described. The corroded metal contains a high proportion of carbonate (13.2% CO_2), and is coloured by oxides of lead. In comparative experiments with pipes set in (a) 1 pt. of artificial Portland cement and 5 pts. of sand, (b) 1 pt. of artificial Portland cement, 1 pt. of lime, and 5 pts. of sand, and (c) 1 pt. of hydraulic cement and 5 pts. of sand, most corrosion occurred with mixture (b) and least with (c). It is suggested that lead pipes in damp positions should be set in a jacket of clay, or be treated with asphalt or pitch varnish.

R. K. CALLOW.

Final report on the relative corrodibilities of various commercial forms of iron and steel. Results of prolonged exposure to the atmosphere, and general conclusions from all the tests. J. N. FRIEND (Iron & Steel Inst., Carnegie Schol. Mem., 1929, 18, 61—71; cf. B., 1927, 843).—The loss in weight and the effect of corrosion on the surface appearance of bars of cast iron, wrought iron, and various plain carbon and alloy steels, after exposure for seven years in the Birmingham atmosphere, and after exposure for six years in the atmosphere of a gas works, have been determined. The wrought-iron specimens showed a tendency to corrode in furrows along the bar, and slight furrowing was also noticed with the plain carbon and nickel-chromium steels; all these metals suffered heavy losses in weight. Steels with 0.12% Si and those with 0.15% Cu had very smooth surfaces, and lost much less in weight, stainless steels were very little affected, manganese steel became pock-marked all over, and cast irons developed a rough surface, but were otherwise in good condition, without any signs of graphitisation. The tests made at the gas works showed similar results, but the losses in weight were generally much more severe. The most important conclusions drawn from all the tests described in the four reports are summarised.

A. R. POWELL.

Metals in dairy equipment; corrosion caused by washing powders, chemical sterilisers, and refrigerating brines. O. F. HUNZIKER, W. A. CORDES, and B. H. NISSEN (J. Dairy Sci., 1929, 12, 252—284).—The effect of solutions of sodium hydroxide, carbonate, phosphate, hypochlorite, chloride, chromate, and silicate, calcium chloride, chloramine-T, and commercial products on aluminium, tinned copper, tinned iron, galvanised iron, tin, nickel, resistant steel, and commercial alloys was studied.

CHEMICAL ABSTRACTS.

Precipitation of lead and copper from solution on sponge iron. G. L. OLDRIGHT, H. E. KEYES, V. MILLER, and W. A. SLOAN (U.S. Bur. Mines Bull. No. 281, 1928, 131 pp.).—Sponge iron is produced by the reduction of magnetite or hæmatite with carbon monoxide at

950°. It resembles porous, coarse sand and offers a large amount of surface. Lead is usually extracted by wet processes as chloride, and the precipitation of lead chloride by sponge iron was therefore studied. It was found that for quick precipitation a temperature of 60° is desirable. Some agitation is necessary, but too vigorous stirring produces balling. Excess of iron has little effect. Percolation methods were unsuccessful, and agitation with air oxidises the iron. The best results were obtained by slowly rabbling sponge iron downward over a series of trays against an upward current of lead solution. Using commercial solutions, complete stripping from lead was obtained, together with a product containing about 70% Pb. For this purpose 30% over the theoretical of iron was used. Comparative results obtained with scrap iron (as generally used) show that a considerable saving in labour cost would be obtained. The precipitation of copper from sulphate solutions was also studied. It was found that sponge iron precipitates copper from a given solution much faster than does scrap iron, but the product is less pure. An excess of 7% over the theoretical amount of iron is usually sufficient. The temperature rises rapidly during precipitation and is not easily controlled. Rate of precipitation increases with temperature. The temperature should later be allowed to fall or redissolution of copper may occur. An increase of acidity increases the rate of precipitation in richer solutions, but decreases it in poorer solutions. Dissolution of copper is associated with the action of air drawn in by agitation; it is negligible if air is excluded. The possibility of a continuous countercurrent method for copper is discussed. The type of precipitator operating on the principle of the MacDougall furnace, and described above as applied to lead, appears satisfactory, but much more thorough agitation is required than with lead.

C. IRWIN.

Bearing metals with a lead-antimony-tin basis. H. MÜLLER (Z. Metallk., 1929, 21, 305—309).—The properties and structure of bearing metals with a high lead content are described, and the effect of adding small percentages of the various copper or nickel antimonides, either alone or together, to eutectic alloys of the system lead-tin-antimony is illustrated by photomicrographs. The compound Cu_2Sb appears unchanged as needles in the ground mass of eutectic, whereas the compounds Cu_3Sb and Cu_5Sb_2 are both converted into Cu_2Sb and the ground mass becomes hypoeutectiferous with small lead areas irregularly distributed throughout. Of the nickel antimonides NiSb alone remains unchanged in the eutectic alloy, Ni_2Sb_3 giving up antimony to form δ -tin-antimony crystals and Ni_5Sb_6 , and Ni_4Sb taking up antimony from the eutectic. Hence, in the systematic investigations of these alloys only the compounds Cu_2Sb and NiSb need be taken into account. The binary system Cu_2Sb - NiSb contains no double compounds, but NiSb crystals retain about 5% Cu_2Sb in solid solution; NiSb affects the temperature of the β - and δ -transformations of Cu_2Sb and also causes a more regular distribution of the copper compound in lead bearing metals.

A. R. POWELL.

Rapid determination of lead. P. F. THOMPSON (J. Soc. Chem. Ind. Victoria, 1929, 29, 154—159).—

Lead in solution may be determined directly by titration with ammonium molybdate without previous separation as sulphate. The ore is dissolved in nitric acid, the solution evaporated, and acetic acid added, followed by ammonia until the liquid is practically neutral. The diluted solution is then heated to boiling and titrated rapidly with standard molybdate solution, using as external indicator a 1:300 solution of tannic acid containing 10% of potassium fluoride and a little acetic acid. Among the ordinary metals only iron, copper, cobalt, and nickel interfere; the effect of iron is eliminated by the addition of potassium fluoride to the tannic acid indicator as above, whilst if copper be present the indicator should contain some potassium cyanide. A determination of lead in galena can be carried out by the above method in 12 min., and the results appear to be as accurate as those obtained by the original long method. (Cf. Dawkins and Weldon, B., 1924, 670.)

H. F. HARWOOD.

Possible use of beryllium in aircraft construction. H. W. GILLET (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 4 pp.).—The physical properties of beryllium are briefly reviewed and possible applications of the metal and its alloys discussed. An outstanding property of beryllium is its high modulus of elasticity.

H. J. T. ELLINGHAM.

Comparison between sodium cyanide and potassium cyanide silver-plating solutions. E. B. SANIGAR (Metal Ind., 1929, 34, 539—540).—The plated articles were indistinguishable; the finishing losses were slightly less for sodium cyanide.

CHEMICAL ABSTRACTS.

Effect of current density on the hardness of electrodeposited chromium. R. J. PIERSOL (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 6 pp.).—Chromium is electrodeposited on steel sheets from a bath containing 250 g. of chromic acid and 3 g. of chromic sulphate per litre at 49°, using various current densities ranging from 16 to 109 amp./dm.². The hardness of the deposits is measured by the time required for an emery wheel, operated under standardised conditions, to cut through a plate 0.025 mm. thick. The hardness is found to increase enormously with current density up to about 62 amp./dm.², after which it falls off somewhat. The maximum hardness observed was 43 times that of a deposit produced at 16 amp./dm.²

H. J. T. ELLINGHAM.

Properties of industrial gold alloys. E. A. SMITH (Metal Ind., 1929, 34, 342—344, 352, 373—374).

PATENTS.

Blast furnace. C. L. T. EDWARDS, Assr. to BETHLEHEM STEEL Co. (U.S.P. 1,727,100, 3.9.29. Appl., 23.10.24).—In the smelting of ores in a blast furnace, the pressure in the shaft is increased by the imposition of back pressure without impeding the free escape of solids entrained by the combustion gases.

A. R. POWELL.

Annealing furnaces [for wire]. INTERNAT. GEN. ELECTRIC Co., Inc., Asses. of ALLGEM. ELEKTRIZITÄTSGES. (B.P. 300,242, 9.11.28. Ger., 9.11.27).—The furnace comprises an electrically-heated tube, inclined downwards at an angle of about 30° to the horizontal, the

upper end of the tube outside the heating zone being bent downwards at right angles to the rest of the tube. A small vertical tube entering the main tube in the underside of the bend provides a means of passing a protective gas, e.g., hydrogen, through the furnace.

A. R. POWELL.

Protecting pipes from corrosion. FREEPORT SULPHUR Co., Asses. of (A) H. S. BURNS and L. S. BUSHNELL, (B) L. S. BUSHNELL (U.S.P. 1,700,995—6, 5.2.29. Appl., [A] 13.7.26, [B] 4.5.28).—(A) The surfaces of pipes for conveying corrosive liquids, e.g., "fermentation" water in the underground fusion system of mining sulphur, are protected by first forcing through the pipes a hot solution of calcium and magnesium salts, e.g., sulphates and carbonates, to deposit a scale on the pipes. (B) Suitable apparatus is described.

R. BRIGHTMAN.

Hardening of tool steels. (SIR) A. HERBERT and A. H. LLOYD (B.P. 318,060, 28.11.28).—Tool steel containing, e.g., 5% Cr, 17% W, 13.5% Co, and 1.25% V is heated at 1310—1340°, cooled in an air blast, reheated, and again cooled as before, and finally tempered at 600° for 30 min.

A. R. POWELL.

Galvanising of steel or iron sheets. G. S. MALIPHANT and F. J. REES (B.P. 318,806, 8.12.28).—A bath for galvanising iron or steel sheets comprises a rectangular vessel filled with molten zinc into which protrudes one arm of a broad U-shaped trough filled with molten lead and terminating inside the zinc bath some distance below the surface of the zinc and outside the zinc bath, either well above the zinc surface or in a second rectangular vessel forming a lead supply bath. The sheets are passed through the lead trough into the zinc, which is covered with a protective flux, thence out of the zinc through a pair of rollers partially immersed in the zinc.

A. R. POWELL.

Welding of rails or the like by means of superheated molten metal. ELEKTRO-THERMIT GES. M.B.H., and A. G. CRÜM (B.P. 318,705, 10.7.28).—Molten steel or iron is poured into a mould surrounding the parts to be welded, and oxygen or chlorine is forced through it to cause superheating by the vigorous exothermic action which ensues. The vigour of the reaction may be increased by adding readily oxidisable substances such as carbon, aluminium, manganese, or silicon to the molten iron.

A. R. POWELL.

Fluxes for joining metals by welding, soldering, etc. K. WERNER, and "PROGRESS" GES. FÜR TEXTILMASCHINEN M.B.H. (B.P. 318,377, 22.8.28).—A flux for hard soldering, brazing, or welding is made by mixing the powdered product obtained by melting equal parts of brass turnings, lead, and borax with one third of its weight of borax, one third of its weight of sodium carbonate, and one fifteenth of its weight of sodium chloride. The borax and sodium carbonate may be replaced by equal weights of potassium and ammonium sulphates.

A. R. POWELL.

Heat-treatment of oxidised copper ores. T. J. and B. TAPLIN, and METALS PRODUCTION, LTD. (B.P. 318,314, 5.6.28).—The gases evolved in the chloridising treatment claimed in B.P. 250,991 (B., 1926, 590) are passed over copper carbonate ores or limestone to absorb

the hydrogen chloride present, and the resulting products are used again in the chloridising treatment.

A. R. POWELL.

Treatment of ores etc. for the separation of metals and their salts. E. A. ASHCROFT (B.P. 318,301, 5.4., 27.7., 20.8.28, and 29.1.29).—Oxidised ores containing lead, zinc, and copper are heated with ammonium chloride at 300–350°, whereby ammonia is evolved and may be collected in water or converted into carbonate, and chlorides of zinc, lead, and copper are produced. The chloridised product is leached with water, the lead chloride collected on a filter, and the solution further purified by treatment with hydrogen sulphide or with zinc dust. Zinc is then precipitated by careful addition of ammonium carbonate; if lime or magnesia is present these separate first during this treatment and may be collected separately. The basic zinc carbonate is calcined to oxide and the carbon dioxide used for the regeneration of ammonium carbonate. The filtrate from the zinc carbonate is evaporated to recover ammonium chloride for use again. The lead chloride residue is extracted with ammonium acetate or with ammonium thiosulphate and the lead eventually recovered by electrolysis of the fused chloride or sulphochloride. The process is applicable to the treatment of mixed sulphide ores; in these cases blende is much more resistant to chlorination than galena, so that by heating with a regulated quantity of ammonium chloride only the galena is chlorinated and may be extracted first. The residual blende is roasted and heated with ammonium chloride until the ammonia is expelled, and the zinc chloride is recovered by distillation at 750–900°.

A. R. POWELL.

Thermal disintegration of chrome ores or minerals containing chromium. BOZEL-MALÉTRA SOC. IND. DE PROD. CHIM. (B.P. 288,250, 27.2.28. Fr., 6.4.27).—The roasting of chromite ores with lime and sodium carbonate is carried out in mechanically rabbled, multiple-hearth furnaces of the type used in roasting pyrites.

A. R. POWELL.

Production of rare metals. J. W. MARDEN and M. N. RICH, Assrs. to WESTINGHOUSE LAMP CO. (U.S.P. 1,728,941, 24.9.29. Appl., 19.2.27).—Tantalum, vanadium, and niobium are prepared in the form of a powder free from hydrogen and nitrogen by heating a mixture of the metal oxide, calcium, calcium chloride, and an alkali metal in a sealed or evacuated container.

C. A. KING.

Production of uranium and uranium-zinc alloys. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,728,940 and 1,728,942, 24.9.29. Appl., [A] 1.3.26, [B] 29.8.28).—(A) Uranium oxide or a mixture of uranium oxide and zinc chloride is reduced by means of calcium in the presence of calcium chloride. (B) A compound of uranium is reduced and alloyed with zinc.

C. A. KING.

Vanadium-aluminium-silicon alloy. B. D. SAKLATWALLA, Assr. to VANADIUM CORP. OF AMERICA (U.S.P. 1,727,180, 3.9.29. Appl., 2.2.28).—The alloy contains 40–90% V, 3–15% Al, 5–30% Si, and the remainder chiefly iron.

H. ROYAL-DAWSON.

Manufacture of annealed thorium. W. B. GERO,

Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,719,975, 9.7.29. Appl., 1.3.26).—Articles of highly reactive, rare refractory metals, *e.g.*, thorium, are annealed by heating them in a gas-tight container packed with a finely-divided, non-contaminating substance, *e.g.*, thorium, which will react with residual atmospheric gas to form stable compounds at the temperature of annealing, but is inert to the refractory metal. F. G. CLARKE.

Ruthenium alloy. M. M. GOLDSMITH, and W. H. FALCK, Assr. to GOLDSMITH BROS. SMELTING & REFINING CO. (U.S.P. 1,730,003, 1.10.29. Appl., 3.1.27).—An alloy comprising about 75% Ru, 17.5% W, and 7.5% Ni is claimed as a substitute for osmiridium.

F. G. CROSSE.

Working-up materials containing precious metals. G. BÜRG (B.P. 317,752, 26.9.28. Ger., 21.8.28).—Ores, minerals, and residues containing precious metals in a colloidal form are subjected to sudden heating or cooling, to electrical conditions, or to pressure so as to set up sudden differences of stress which are claimed to make the precious-metal particles coalesce into visible form, which is recoverable by ordinary methods.

A. R. POWELL.

Electrolytic production of aluminium. ALUMINIUM IND. A.-G. (B.P. 305,458, 31.10.28. Ger., 4.2.28).—In the electrolytic refining of aluminium by the process described in B.P. 265,170 (B., 1928, 412), the anode comprises a plate of crude aluminium with deep corrugations so as to have a low current density at the anode, thus preventing iron and silicon from dissolving in the bath.

A. R. POWELL.

Electrolytic production of aluminium. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 318,431, 15.11.28. Ger., 3.9.28).—In the usual method of obtaining aluminium by electrolysis of alumina in a molten cryolite bath, the active surface of the electrodes is kept equal by using a single anode having a diameter slightly greater than that of the cathode which forms the base of the furnace, and one or more channels through which the charge can be introduced into the furnace. A. R. POWELL.

Roasting of iron carbonate ores. A. APOLD and H. FLEISSNER (U.S.P. 1,729,697, 1.10.29. Appl., 28.11.24).—See B.P. 253,305; B., 1926, 710.

Metallurgical carbons (B.P. 288,551 and 304,676).—See II.

XI.—ELECTROTECHNICS.

Some characteristics of photo-electric tubes. L. R. KOLLER (J. Opt. Soc. Amer., 1929, 19, 135–145).—An account of recent work on photo-electric cells, and of some of their practical applications.

C. A. SILBERRAD.

Pressure electrolysis of water. R. SCHNURMANN (Z. angew. Chem., 1929, 42, 949–952).—Electrolysis of dilute sulphuric acid solution was carried out between platinum electrodes, the acid being enclosed in a sealed glass tube with a space above the liquid in which the gases, hydrogen and oxygen, were collected, thereby increasing the pressure (to about 870 atm.) until it was sufficient to rupture the vessel. The results show that the amount of current which could be passed increased with time, and therefore with gas pressure; until a

maximum was reached. A suggested use for the gas mixture prepared by this method is for the driving of an electric motor. H. T. S. BRITTON.

Testing permanent-magnet steel. OERTEL. **Silver-plating solutions.** SANIGAR. **Electro-deposited chromium.** PIERSON.—See X. **Electro-metric titration [of worts].** EMSLANDER.—See XVIII.

PATENTS.

Electrolytic cells [for producing hydrogen and oxygen]. MONTECATINI SOC. GEN. PER L'IND. MIN. ED AGRIC., Assees. of G. FAUSER (B.P. 292,130, 13.6.28. Ital., 14.6.27).—Flat parallel electrodes are joined by current leads made of nickel-plated copper strip.

J. S. G. THOMAS.

[Uni-directional] electrolytic condenser. A. E. WHITE. From FANSTEEL PRODUCTS CO., INC. (B.P. 319,033, 15.5.28).—One or more filmed electrodes are immersed in a substantially dry electrolytic solution in an alcohol, *e.g.*, glycerin. A film of oil above the electrolyte serves to prevent absorption of moisture.

J. S. G. THOMAS.

Deoxygenation of enclosed atmospheres [of oil-immersed electric transformers etc.]. C. J. RODMAN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,725,160, 20.8.29. Appl., 23.12.22).—The atmosphere above the oil in such apparatus is deoxygenated by treatment with a deoxidising agent, the activity of which increases with rise of temperature.

A. R. POWELL.

[Construction of Faure-type] electric accumulator [positive] plates. H. G. BROWN, and D. P. BATTERY CO., LTD. (B.P. 319,388, 16.6.28).

[Unspillable vent for] electric batteries. M. WILDERMAN (B.P. 319,198, 17.5.28).

[Arrangement of parts in] photo-electric cells. E. G. O., A. E. M., and K. E. H. PRESSLER. ("OTTO PRESSLER" THÜRINGER VAKUUMRÖHRENFABR. U. FABR. WISS. APPARATE) (B.P. 302,183, 10.12.28. Ger., 9.12.27).

Aluminium (B.P. 305,458 and 318,431).—See X.

XII.—FATS; OILS; WAXES.

The fat of barley and of its malting products. K. TÄUFEL and M. RUSCH (Z. Unters. Lebensm., 1929, 57, 422—431).—The fats extracted from barley and various products of its malting and brewing were analysed, using the Twitchell separation of solid and liquid fatty acids after removal of the unsaponifiable matter. The solid fraction was shown to be a mixture of stearic and palmitic acids; the proportions were calculated from the neutralisation value. The liquid fraction was shown to be a mixture of oleic, linoleic, and linolenic acids which was analysed by determining the linolenic acid as the hexabromostearic acid and then determining the oleic and linoleic acids indirectly from the iodine value of the liquid fraction (cf. B., 1929, 564). Barley fat and malt fat were thus found to contain, respectively, oleic acid 26.5, 16.4; linoleic acid 43.7, 49.4; linolenic acid 0.44, 0.82; palmitic acid 7.4, 8.2; stearic acid 2.6, 5.1; and unsaponifiable matter 5.4, 6.1%. The embryo fat contained 23—26% of unsaponifiable matter,

and the fat of the spent malt differed slightly from that of the malt. W. J. BOYD.

Fat from the seed husks of laurel berries. G. WALLRABE (Chem. Umschau, 1929, 36, 293—295).—The fat extracted from the (seed) husks (23.7%) of the laurel fruit was dark yellow and of buttery consistency, optically inactive, and contained no ethereal oils. It had acid value zero, saponif. value 198.2, iodine value 78.72, insoluble fatty acids 94.4%, unsaponifiable matter 1.35%, $[\alpha]_D^{25} +0.82^\circ$. The insoluble fatty acids had m.p. 39—41°, iodine value 85.19, mean mol. wt. 273.9; these yielded 24.15% of solid fatty acids (m.p. 60—61°, mean mol. wt. 257.9) and 74.82% of liquid fatty acids (iodine value 113.9, mean mol. wt. 284.3). The solid fatty acids consisted preponderantly of palmitic acid; the liquid fatty acids contained about 74% of oleic acid and 26% of linoleic acid. The phytosteryl acetate isolated from the unsaponifiable matter had m.p. 123—124° (from alcohol). Corresponding values for fat from the seed-free fruit are also given for comparison (cf. B., 1929, 859).

E. LEWKOWITSCH.

Twitchell fat-splitting reagents. K. NISHIZAWA (Chem. Umschau, 1929, 36, 277—284).—The addition of electrolytes to solutions of fat-splitting reagents ("Idrapid," "Pfeilring," "Kontakt") has considerable influence on the emulsifying power as measured by the Donnan drop method (cf. A., 1900, ii, 102; *i.e.*, the drop number of an oil in reagent solution compared with that in water), the effect varying with the particular reagent employed. Increasing additions of a strong acid or salt, *e.g.*, hydrochloric or sulphuric acid, or sodium chloride or sulphate, up to a certain maximum (when salting-out of the reagent occurs) increases the emulsifying power (to an extraordinary extent in the case of the "Kontakt" reagent). A similar result is obtained with weak acids in much smaller degree, and no salting-out occurs. The emulsifying power of the sodium salts of the reagents increases with concentration, the effect being less if the reagent itself is admixed; the emulsifying power is greater with a mixed solution of 1% of reagent and 1% of its sodium salt than with a 2% solution of the reagent alone. The emulsifying power was found to be increased by the addition of glycerol; a marked increase was also obtained by the addition of fatty acids to the oil, the optimum concentration being at about 70% of fatty acid (1% solution of reagent).

E. LEWKOWITSCH.

Determination of sugar in soaps and soap preparations. K. BRAUN and E. WALTER (Chem.-Ztg., 1929, 53, 778).—A known weight of the sample is decomposed with excess dilute sulphuric acid, the vessel being kept at about 100° for $\frac{1}{2}$ hr. in order to invert the sucrose; after 24 hrs. the acid liquor is filtered, neutralised with soda, and made up to standard volume (200 c.c.). Then 25 c.c. of the sugar solution are added to 50 c.c. (excess) of boiling Fehling's solution and 25 c.c. of water; the whole is boiled for 5 min. and allowed to settle (20 min.). The cuprous oxide is collected, washed with boiled-out distilled water, and dissolved in 50 c.c. of standard ferric sulphate solution (50 g. of ferric sulphate dissolved in 200 c.c. of concentrated sulphuric acid and diluted to 1 litre), and the

ferrous sulphate produced is titrated with 0.1*N*-permanganate (1 c.c. = 0.0029 g. of sucrose). The method is suitable for the determination of sugar in glycerol, and of starches in soaps etc.

E. LEWKOWITSCH.

Polymerisation of linseed oil in stand-oil formation. F. WILBORN and F. KITTLER (Farben-Ztg., 1929, 34, 2942—2943).—Determinations of mol. wts. of stand oils in order to ascertain the degree and type of polymerisation occurring have not led to concordant results. The mol. wts. of a sample of raw linseed oil, before and after heat-thickening, and of the fatty acids derived therefrom were determined by a variety of methods. The influences of solvent used, concentration of solution, etc. on the results obtained lead to the conclusion that it is impossible to investigate the polymerisation problem by mol. wt. determination alone.

S. S. WOOLF.

Drying of tung oil. H. WOLFF (Farben-Ztg., 1929, 34, 2941—2942).—A dried tung oil film was stored for a period of 2½ yrs., light and air being excluded. After about 1½ yrs. syneresis occurred, droplets of oil of lower refractive index than the original oil being formed. After 2½ yrs. three phases were present, a crystalline substance—probably β -elæostearin—having also separated from the film.

S. S. WOOLF.

Determination of organically-combined sulphur in sulphonated oils. R. HART (Chem. Umschau, 1929, 36, 295—297).—The author's revised volumetric method is described (cf. J. Amer. Leather Chem. Assoc., 1927, 22, 588); improvements in the original procedure (cf. B., 1917, 1139) consist in the use of *N*-sulphuric acid, and the addition of ether and salt to facilitate observation of the end-point.

E. LEWKOWITSCH.

Determination of fats in drugs. GLASER and HALBERSTAM.—See XX.

PATENTS.

Extraction of fatty acids, resins, bitter substances, and mucilage from oils and fats. K. K. WILHELM (U.S.P. 1,729,809, 1.10.29. Appl., 15.9.27. Ger., 2.5.27).—A volatile fat solvent is added to the oil or fat and the mixture is stirred (preferably warm) with an aqueous alcohol-ammonia solution; about 1% of Glauber's salt dissolved in 10 pts. of the ammonia solution is added, and the whole is re-mixed. On being kept the oil-solvent layer separates free from mucilage and is drawn off and evaporated. E. LEWKOWITSCH.

Manufacture of sulphonated derivatives of unsaturated fatty acids. I. G. FARBENIND. A.-G. (B.P. 296,999, 10.9.28. Ger., 10.9.27).—Unsaturated fatty acids or esters (*e.g.*, oleic acid, castor oil) are treated with fuming sulphuric acid in the presence of a halogenated unsaturated hydrocarbon (*e.g.*, trichloroethylene); the sulphonated products are neutralised, washed, etc., and the solvent is removed by distillation. The products are resistant to acids and not precipitated by calcareous water.

E. LEWKOWITSCH.

Manufacture of fatty acid peroxide. W. B. STODDARD and V. R. KOKATNUR, ASSRS. to PILOT LAB., INC. (U.S.P. 1,718,609, 25.6.29. Appl., 21.10.27).—A fatty acid chloride is treated with sodium peroxide.

C. HOLLINS.

Extraction of oils from oleaginous materials. R. O. BOYKIN, ASSR. to N. R. VAIL (U.S.P. 1,721,686, 23.7.29. Appl., 22.11.26).—Oily meal is dried and extracted with a water-insoluble oil solvent the sp. gr. of which is not greater than that of the meal; the mixture is then pressed and the residual material again washed with a fresh quantity of the solvent. Suitable apparatus is described.

R. H. GRIFFITH.

Process, steps, and product of reaction of cashew nut-shell oil. Modified cashew [nut]-shell liquid [varnish]. Cashew nut-shell oil condensation product. M. T. HARVEY, ASSR. to HARVEL CORP. (U.S.P. 1,725,795—7, 27.8.29. Appl., [A] 23.12.26, [B] 17.11.27, and [C] 15.6.28).—(A) Cashew nut-shell oil (25 vols.) is dissolved in 50 vols. of light petroleum, 10 vols. of formalin and 5 vols. of concentrated hydrochloric acid are added, and the mixture is kept for 48 hrs. The oily layer is separated and used as a varnish. (B) The oil is heated rapidly to 340°, preferably under pressure, cooled to 230°, and mixed with 1% of litharge and 1% of manganese resinate. The product is suitable for varnishing fabrics and paper, and dries to a flexible film at 135°. (C) The oil is heated rapidly to 340°, mixed with 25% of its weight of raw linseed oil, cooled to 260°, mixed with driers as under (B), cooled to 150°, mixed with an equal weight of a solvent, cooled, treated with 1% of its weight of formalin, set aside overnight, and heated at 140° to expel water and complete the reaction.

A. R. POWELL.

Soap preparation. F. GÜNTHER and J. NÜSSLEIN, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,730,037, 1.10.29. Appl., 29.5.26. Ger., 2.6.25).—See B.P. 253,105; B., 1928, 60.

[Detergent for] washing of textile goods. E. RIEHL and O. LIND, ASSRS. to HENKEL & Co. G.M.B.H. (U.S.P. 1,716,347, 4.6.29. Appl., 15.8.27. Ger., 17.8.26).—See B.P. 276,339; B., 1928, 647.

Packing material for soft soap (B.P. 319,517).—See V. **Liquors for treating leather** (B.P. 318,070).—See XV. **Soaps and oils from sewage** (B.P. 318,849).—See XXIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Viscosity of oil paints. H. WOLFF (Farben-Ztg., 1929, 34, 2990—2991).—Paints of identical oil content but prepared either by grinding to paste and thinning out or by grinding with the total amount of oil show differences in viscosity, the latter type being the more viscous. The difference is accentuated at low oil contents, but diminishes with increase of oil. It is shown, however, that the "second critical point" previously described (B., 1929, 825) is virtually independent of the method of grinding, and may thus, for a particular oil, be regarded as a constant for the pigment.

S. S. WOOLF.

Some optical properties of paints and pigments. F. C. TOY (J. Oil & Col. Chem. Assoc., 1929, 12, 225—239).—The term "covering power" is discussed from the aspect of obliteration rather than of spreading. The theory of measuring instruments of the "cryptometer" type is examined, and it is shown that the physiological characteristics of the eye render such instruments

unreliable, since the matching point (and hence the measured "covering power") varies with the observer and with the intensity and colour of the light used. It is suggested that the grading of paints in order of covering power could be carried out mechanically by making use of a photoelectric cell. A series of curves correlating grain size with obliterating power for light of different wave-lengths is considered. The existence of a maximum in these curves introduces the difficulty of ascertaining on which side of the maximum a determined obliteration lies, whilst the accuracy of measurement depends largely on the actual size. A method for determining grain size is discussed, in which the optical density of a dispersion is observed, undeviated light alone being measured in spite of the fact that a fraction of the scattered light comes forward in the same beam. The extinction coefficient (β) for undeviated light is given by the expression $I = I_0 e^{-\beta M}$, I_0 and I being the intensities of incident and transmitted beams, respectively, and M the mass traversed. The quantity $\beta\rho$, where ρ is the density of the solid phase, is suggested as a criterion of grain size, applicable to all kinds of substances. If the reciprocal of $\beta\rho$ be plotted, a straight-line relation between it and grain size holds over nearly all the range of sizes investigated.

S. S. WOOLF.

Properties of current Finnish turpentine oil. E. PYHÄLÄ (Chem.-Ztg., 1929, 53, 758—759).—The somewhat primitive methods of commercial production of Finnish turpentine oil (from pine stumps) are described: since no refining of the crude oil or after-treatment with deodorisers or decolorisers etc. is undertaken, a pine oil of variable character and odour is obtained. The characteristics (distillation range, flash point, etc.) of typical oils (20—70% of turpentine) are given. It is shown that by suitable treatment prior to distillation a higher yield of a purer turpentine can be obtained.

E. LEWKOWITSCH.

Oil absorption of pigments. W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1929, 34, 2940—2941).—The factors on which depends the determination of oil absorption of pigments are indicated. Oil absorption values obtained by the Gardner method (modified by the use of a short glass rod in place of a flat spatula) are quoted for a range of common pigments. Oil absorptions of mixtures are, in general, additive quantities.

S. S. WOOLF.

White pigments. III. H. WOLFF and G. ZEIDLER (Farben-Ztg., 1929, 35, 24—25; cf. B., 1928, 274).—No appreciable difference in the extent of lead soap formation in paints made from basic carbonated and basic sulphate white lead was detected by optical or analytical methods. In exposure tests paints made from the carbonate show a greater loss of weight than those made from the sulphate. It is considered that the influence of lead soap formation on the durability of paint films is still problematical.

S. S. WOOLF.

Determination of water-soluble arsenious acid in Schweinfürth green. H. M. L. BRÉMOND (Bull. Soc. Pharm. Bordeaux, 1928, 66, 226—231; Chem. Zentr., 1929, i, 2035).—The finely-divided material (1 g.) is boiled for exactly 5 min. over a small flame with

25 c.c. of 12.5% sodium acetate solution, boiling water being added as the liquid evaporates. The cooled liquid is diluted to 50 c.c., filtered, and the arsenious oxide is titrated with 0.1N-iodine solution. The process is repeated with 2 g. and 50 c.c., respectively, of material; the difference in the volumes of iodine solution required multiplied by 0.00495 represents the quantity of arsenious oxide in 1 g. of the Schweinfürth green. The result is accurate when the quantity of water-soluble arsenious oxide does not exceed 3%. A. A. ELDRIDGE.

PATENTS.

Manufacture of emulsion paints. L. KIRSCH-BRAUN (U.S.P. 1,691,765, 13.11.28. Appl., 7.5.25).—Paint base, e.g., pitch, bitumen, which may contain finely-divided mineral powder, is dispersed in an aqueous medium by means of a finely-divided pigment paste.

R. BRIGHTMAN.

Production of [bituminous] emulsions or dispersions. L. KIRSCHBRAUN and H. L. LEVIN, Assrs. to FLINTKOTHE Co. (U.S.P. 1,691,766—7, 13.11.28. Appl., [A] 17.12.25, [B] 12.10.26).—(A) Maximum dispersion of asphalt or other pitches in aqueous media is obtained by adjusting the p_H value of the system to a predetermined value depending on the nature of the pitch. Alternatively, the pitch may be treated to render it suitable for dispersion by a particular emulsifying agent. The emulsion may be subsequently treated to modify its p_H value and the properties of the final system without breaking the emulsion, particularly to increase the resistance of the dried product to water action and re-emulsification. (B) The dispersion is effected in an aqueous solution of bentonite or other insoluble mineral dispersing agent.

R. BRIGHTMAN.

Production of [bituminous] emulsions or dispersions. L. KIRSCHBRAUN (U.S.P. 1,691,768, 13.11.28. Appl., 16.3.28).—The dispersion of bitumen or pitch is effected in an aqueous suspension of a mixture of two or more mineral dispersing agents, previously adjusted to a definite p_H value by addition of an electrolyte as buffer salt.

R. BRIGHTMAN.

Plasticised moulding composition. H. M. WEBER, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,720,406, 9.7.29. Appl., 17.12.24).—Hexamethylenetetramine-triphenol is used as a flux to give greater fusibility to a mixture of a resin with a filler.

R. H. GRIFFITH.

Manufacture of colours or the like capable of being fixed by heat. O. VAN CUYCK (U.S.P. 1,730,178, 1.10.29. Appl., 7.5.26. Belg., 8.5.25).—See B.P. 251,866; B., 1926, 638.

Mills for grinding paints, enamels, inks, and other viscous substances. E. A. WHITE (B.P. 319,544, 4.12.28).

Decohering solid substances (B.P. 292,965).—See I. Aryl alkyl and hexyl esters (U.S.P. 1,700,960 and 1,702,180).—See III. Iron oxide pigments (U.S.P. 1,726,851—2).—See VII. Compositions for paints (B.P. 317,502).—See IX. Resins from oils and fats (U.S.P. 1,729,809). Cashew nut-shell oil products (U.S.P. 1,725,795—7).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Increased stretching capacity of dipped rubber articles (toy balloons etc.) and decrease in number of dippings required for thick-walled dipped rubber articles resulting from the increased viscosity of rubber solutions containing sipalin. R. DITMAR and K. H. PREUSZE (Chem.-Ztg., 1929, 53, 779).—The addition of 5% of sipalin (adipic esters) to rubber solutions greatly increased the stretching capacity of articles made from them by dipping (as determined by the size of balloons at the bursting point). Sipalin MOM was the most effective when vulcanisation was brought about by sulphur chloride; with sulphur vulcanisation the action of the sipalin appeared to depend on the accelerator used, and the choice of the most suitable ester is determined by the conditions. Since the addition of sipalin greatly increases the viscosity of the solutions fewer dippings are needed to produce a given thickness of rubber. E. LEWKOWITSCH.

New microscopical methods in connexion with problems of vulcanisation. E. A. HAUSER, H. MIEDEL, and M. HÜNEMÖRDER (Coll. Symp. Mon., 1928, 6, 207—217).—When the material is undercured sulphur reappears as colloidal globules which grow and form dendrites; zinc oxide retards this process. Accelerators appear to prevent separation of the sulphur except at the surface. Ether-soluble α -caoutchouc vulcanises less readily than whole rubber or β -caoutchouc. It is considered that vulcanisation depends on minute traces of a substance present in crude rubber which adheres to the α -fraction so that it can be removed only after this fraction has been isolated from its original combination in the rubber. CHEMICAL ABSTRACTS.

PATENTS.

Preservation of rubber latex. A. J. SOMER and R. B. R. WALKER (B.P. 318,717, 30.7.28).—Rubber latex is stabilised by the addition of a boron compound, e.g., sodium pentaborate or a mixture of borax and boric acid, so as to impart a p_H value between 6 and 9.2; a small proportion of a germicide may also be introduced. D. F. TWISS.

Preparation of viscous dispersions of rubber and similar materials. ANODE RUBBER CO. (ENGLAND), LTD., Assees. of COMP. GÉN. D'ELECTRICITÉ SOC. ANON. (B.P. 291,805, 9.6.28. Ger., 9.6.27).—Highly viscous aqueous dispersions of rubber, gutta-percha, or balata, which may contain compounding ingredients, are produced by the addition of polysaccharides, proteins, or plant extracts which even at high concentrations form viscous solutions but not jellies. E.g., gum arabic, or partially hydrolysed starch, gelatin, or agar-agar may be used, and such hydrolysis may be effected after introduction of the materials into the latex. D. F. TWISS.

Chemical products [vulcanisation accelerators and anti-oxidants for rubber]. S. M. CALDWELL, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,724,180, 13.8.29. Appl., 2.3.28).—Alkali salts of mercaptobenzthiazoles are heated with chlorodinitrobenzenes, e.g., in alcohol solution. 2:4-Dinitrophenylbenzthiazole sulphide (yellow, m.p. 162—5°) and 4-chloro-2:6-

dinitrophenylbenzthiazole sulphide (yellow, m.p. 167°) are described. R. BRIGHTMAN.

Vulcanisation of caoutchouc. GOODYEAR TIRE & RUBBER CO., Assees. of L. B. SEBRELL (B.P. 297,051, 19.6.28. U.S., 13.9.27).—Vulcanisation is accelerated by the reaction product of a mercaptothiazole, e.g., mercaptobenzthiazole or mercaptanaphthathiazole, and a Schiff base, e.g., ethylidene- or crotonylidene-aniline. The reaction may be effected by heating the mixture under reflux for about 2 hrs. D. F. TWISS.

Manufacture of rubber. E. A. HAUSER, Assr. to K.D.P., LTD. (U.S.P. 1,729,651, 1.10.29. Appl., 16.12.25. U.K., 19.12.24).—See B.P. 250,639; B., 1926, 598.

XV.—LEATHER; GLUE.

Antiseptic value of chlorine with reference to animal skin disinfection. R. C. HOWARD, G. E. ROCKWELL, and W. L. CRIST (J. Amer. Leather Chem. Assoc., 1929, 24, 456—479).—Experiments with Hay bacillus and its spores have shown that the time required for sterilisation by chlorine is increased by increasing the number of bacteria present. Treatment lasting 1 hr. was necessary to sterilise 24 millions of bacteria per c.c. and 3 hrs. for 120 millions, using 120 p.p.m. of chlorine. A longer time was required to sterilise the older spores. The germicidal actions of hypochlorites and chlorine were diminished by the presence of sodium chloride, ammonia, organic matter, and ammonia and ammonium chloride, respectively. The effect of 0.05N-hydrochloric acid was to lower the resistance of the spores to chlorine treatment, but the presence of acid in the chlorine solution lowered its germicidal action. It was shown that such a large amount of chlorine is fixed by raw skins immersed in water containing chlorine, that the concentration of chlorine necessary to effect their complete sterilisation bleaches the hair and retards its loosening. The addition of ammonia to the chlorine water diminished its fixation by the skins. The addition of lactic or formic acid to the chlorinated soak water was not advantageous. Spores were not killed by treatment with lime liquors. The amount of chlorine required to disinfect tannery effluent will depend on the amount of nitrogenous matter and the number of bacteria per c.c. present in the liquor. D. WOODROFFE.

Tanning materials and their determination. G. GRASSER and S. TAU (J. Amer. Leather Chem. Assoc., 1929, 24, 406—427).—Pieces of hide were treated with various tanning agents and the time of hydrolysis, shrinkage temperature, and resistance to boiling were determined on each piece. Iodine was better than bromine or sulphur as judged by the hydrolysis and boiling tests. Bromine, unlike iodine, formed an insoluble compound with gelatin. The salts of chromium, iron, aluminium, thallium, uranium, platinum, and zinc increased the shrinkage temperature of hide to above 65° and, with the exception of zinc, tanned the hide well. All metallic salts except those of barium increased the m.p. of gelatin. Chromium dichromate gave the best tanned leather as judged by the hydrolysis test. The effects of other metallic

dichromates on hide and gelatin respectively showed variations with these materials. No increase in the m.p. of gelatin was observed by the action of dyes, but an increase was noted when they were allowed to act on tanned gelatin; this is attributed to the tanning action of the dyes themselves. Evidently pretreatment of the gelatin with tannin is necessary to obtain the tanning effects of the dyes. The tests with hide were not so favourable. The shrinkage temperature and the hydrolysis and boiling tests showed a difference as the result of the action of gallo-tannic acid on hide, but the m.p. of gelatin was not much affected by it unless sodium sulphate, sodium acetate, or potassium carbonate was added to the solution. The shrinkage temperature of hide was raised by treating it with vegetable tanning materials; the m.p. of gelatin was, however, unaffected. The m.p. of gelatin was raised by treatment with sulphite-cellulose extract, but only when hide had been pretreated with some other tanning materials was its shrinkage temperature increased by subsequent treatment with sulphite-cellulose extract. In an appendix, the effect of ultra-violet light on tanning intensity was studied, and the results are tabulated.

D. WOODROFFE.

Tannin content of dead chestnut trees. R. M. NELSON and G. F. GRAVATT (J. Amer. Leather Chem. Assoc., 1929, 24, 479—499).—It is shown that the tannin content of chestnut trees killed by the blight is not seriously diminished even in trees which have been dead for 30 years. The loss of tannin occurring through the action of decay-producing fungi is not great in the decayed wood, but the amount of available material diminishes. There is no connexion between the sp. gr. and tannin content of green, sound, dead, or decayed chestnut wood. The colour of slightly decayed wood was not worse than that of sound wood. The tannin content of very badly decayed sapwood was low and the colour of the liquors was very dark.

D. WOODROFFE.

Synthetic tanning materials. I. Hydroxybenzene series. G. GRASSER (J. Fac. Agric. Hokkaido, 1929, 24, 73—92).—Various aldehydes were condensed with phenol, sodium phenoxide, or phenolsulphonic acid, and the precipitating power of soluble products towards gelatin was tested. CHEMICAL ABSTRACTS.

South Japanese tanning materials. G. GRASSER (J. Fac. Agric. Hokkaido, 1929, 24, 61—72).—The following values (% of tannin) for Formosan species are recorded: *Quercus castanopsisifolia* (acorn cups) 28.3, *Q. amygdalifolia* (acorns) 6.1, *Q. glandulifera* (galls) 20.4, *Areca catechu*, L., 12.3, *Castanopsis taiwaniana*, Hayata, (wood) 2.0, (bark) 9.2, *Castanopsis kawakamii*, Hayata, (wood) 3.3, (bark) 9.3, *Acacia confusa*, Merr., (bark) 12.0, *Discorea rhipogonoides*, Oliv., (root nodes) 3.0. *Disopyros kaki* tannin is unstable.

CHEMICAL ABSTRACTS.

Solidification of gelatin. M. BERGMANN and B. JACOBI (Kolloid-Z., 1929, 49, 46).—Some properties of stretched gelatin films have been compared with those of unstretched films. The tensile strength of the unstretched gelatin was 4.4 kg./mm.², and it was observed

that immediately before rupture the formerly clear film became milky and opaque. The stretched gelatin had a tensile strength of 9.3 kg./mm.² in the direction of stretching. The middle portion of the strip had the greatest tensile strength, values between 10 and 12 kg./mm.² being obtained. In this case no turbidity appeared before rupture. In the direction perpendicular to that of stretching the tensile strength was only 3.3—4.2 kg./mm.² The fracture was smooth.

E. S. HEDGES.

Purification of tannery effluents. BESSE.—See XXIII.

PATENTS.

Liquors for use in tanning or treating leather, and their application. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 318,070, 27.12.28).—An emulsion produced by agitating one or more oils or fats, e.g., fish oils, rape oil, wool fat, mineral oil, with an aqueous alkylcellulose solution, particularly the methyl or ethyl derivative, with or without the addition of a dye, salt, or other substance used in dyeing, is employed.

D. WOODROFFE.

Treatment of skins or hides with tanning or other liquids. E. LUCKHAUS (B.P. 290,681, 19.5.28. Ger., 19.5.27).—Hides or skins are hung up and stretched in a closed tanning vessel full of tan liquor, which is submitted alternately to a vacuum and air pressure. The vessel is provided at the lower end with an agitating device, and the suspension device fits into a journal in the upper part whereby it can be revolved in the vessel. The tan liquor is circulated by a pump outside the vessel and a filter is provided for it.

D. WOODROFFE.

Adhesive and plastic mass and its manufacture. M. LANDECKER, Assr. to AMER. NUPLAX CORP. (U.S.P. 1,725,805, 27.8.29. Appl., 29.5.26. Ger., 8.4.26).—A mixture of 20 pts. of powdered casein and 100 pts. of carbamide is made into a thick paste with water, and formaldehyde is added as a preservative, and to increase the adhesive power.

A. R. POWELL.

Dyeing of leather (B.P. 316,822).—See VI.

XVI.—AGRICULTURE.

Soils. IV. Classification of their acidic functions. V. Replaceable bases. J. CLARENS and (MME.) PÉRON (Bull. Soc. chim., 1929, [iv], 45, 666—671, 671—674; cf. B., 1928, 869).—IV. In the determination of the acidity of a calcareous clay soil by means of aqueous baryta solution, those acidic constituents which form barium salts hydrolysable by water remain undetermined. They can be determined, however, by the use of alcoholic potash solution. A method for representing geometrically the different types of acidity of a soil is described. V. The different types of acidity present in a soil can also be distinguished by shaking the soil with varying known amounts of 0.1N-hydrochloric acid and determining the amount of free hydrochloric acid remaining in the solution.

O. J. WALKER.

Reclamation of Fresno type of black alkali soil. W. P. KELLY and E. E. THOMAS (Calif. Agric. Exp. Sta. Bull., 1928, No. 455, 37 pp.).—The unproductivity of this soil is due to an excess of soluble salts, particularly

sodium hydrogen carbonate, and the abnormal composition of the clay-like constituents; the productivity can be improved by the use of gypsum, sulphur, ferrous sulphate, or alum, which act simultaneously on the soluble carbonate and clay. Gypsum is effective by reason of its soluble calcium, whilst the other materials act by reason of their acidic nature; the effect of sulphur depends on its slow oxidation. **CHEMICAL ABSTRACTS.**

Hardpans, concretionary layers, and neo-formations of the soils of the more arid parts of the Union [of South Africa]. J. V. CUTLER (Proc. Dept. Conf. S. Africa, Div. Chem., 1929, 421—425).—Extensive areas of hardpan occur in the Union of South Africa. These are of different types, including lime formations, iron formations which appear to be complex silicates and carbonates of calcium, magnesium, and iron coloured different shades of red to brown, and "double" hardpans, which consist of solid hardpan giving place to softer coherent material, readily broken by the spade, deeper in the soil horizon. Two tentative explanations of these formations are presented: (1) that complex soluble salts are washed down to, and deposited at, the pan layer in districts of low rainfall, and (2) that the weathering of parent rock, rich in material which is easily decomposed, but not leached out, gives products which are consolidated *in situ*. In alkali areas the point of maximum concentration of soluble salts does not usually coincide with the layer of maximum development of hardpan. Questions of root penetration in, and methods of breaking up, hardpan are discussed.

E. HOLMES.

Practical sterilisation by heat of small quantities of soil. W. F. BEWLEY (J. Min. Agric., 1929, 36, 623—634).—Various methods for steaming and baking soil are described. A uniform temperature of at least 100° throughout the soil mass is essential, and the treated soil should be in a suitable physical and chemical condition for plant growth. Steaming is the better process.

A. G. POLLARD.

Determination of ammonia in soil and adsorptive power of soil for ammonia. C. OLSEN (Compt. rend. Trav. Lab. Carlsberg, 1929, 17, No. 15, 1—20).—The sample is extracted by shaking with *N*-potassium chloride solution after adjustment of the p_H value of the suspension to 1.0 by the addition of hydrochloric acid. After filtering through a Berzelius filter, the ammonia in the extract is determined by distillation with magnesia in the usual manner. Peat soils have greater adsorptive capacities for ammonia than clays and loams.

A. G. POLLARD.

Determination of the fertiliser requirement of soils. J. KÖNIG (Naturwiss., 1929, 17, 755—758).—In determining fertiliser requirements the nature of the crop must be considered, together with its capacity to utilise nutrients present in soils and fertilisers. Easily soluble nutrients in soil are determined by extraction with 1% citric acid solution. The actual quantity of nutrient assimilable by the crop is calculated from these values by means of a predetermined "percentage utilisation coefficient" for the particular crop. The difference between the values so obtained and

the maximum plant requirement as determined by crop analysis represents the amount to be supplied to the soil artificially. The actual quantity of fertiliser needed is calculated by means of the percentage utilisation coefficient for the particular crop with reference to the nature of the fertiliser to be used.

A. G. POLLARD.

Determination of soil nutrient deficiencies. E. BECKER (Mezőgazdasági Kutatások, Budapest, 1928, 1, 57—94; Bied. Zentr., 1929, 58, 387—390).—Critical trials of the chemical methods of Lemmermann, König and Hasenbäumer, and 'Sigmond, the biological *Azotobacter* method of Niklas and Hirschberger, the chemico-physiological method of Neubauer and Schneider, and corresponding vegetative and field experiments for determining soil nutrient deficiencies are briefly summarised. Practical details are lacking. According to 'Sigmond's method potash may be determined in the filtrate from the phosphoric acid determination; iron, aluminium, calcium, and magnesium are removed by addition of ammonia and ammonium carbonate, sulphate by means of barium chloride, and the potash is then determined by the perchlorate method.

E. HOLMES.

Influence of soil reaction on the conversion of different forms of nitrogen in the soil and their utilisation by plants. K. NEHRING (Landw. Jahrb., 1929, 69, 105—148; Bied. Zentr., 1929, 58, 390—392).—Potash salts are physiologically neutral, superphosphate is certainly not acid, and slag is definitely basic. This physiological nature of fertilisers is of importance from the point of view of nitrification in the soil. Urea and cyanamide act in the first place as physiologically alkaline, but later as acid fertilisers, the net result being that urea is physiologically acid and cyanamide alkaline. Leunaphos and to a smaller degree nitrophoska are acid fertilisers. It is shown that in 1927 in humus, sandy loam pot cultures with artificially adjusted media sodium nitrate on the average gave its optimum effect at p_H 4—5, urea and calcium cyanamide at p_H 6, urea at p_H 7, and ammonium sulphate at p_H 8. The results varied with the crop; barley is acid-sensitive, and oats are acid-tolerant. Below p_H 6.5 nitrate-nitrogen is a better plant food than ammoniacal nitrogen; above that figure they are of equal value.

E. HOLMES.

Peat as a source of nitrogen. LOGVINOVA.—See II.
Micro-determination of phosphate ions. DENIGÈS.—See VII.
Soil corrosion studies. LOGAN.—See X.

PATENTS.

Insecticides. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 317,525, 18.5.28).—The use is claimed of solutions etc. containing mono- or polythiocyanates other than aliphatic monothiocyanates, together with other constituents.

L. A. COLES.

Insect repellent. W. MOORE and H. E. BUC, ASSRS. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,727,305, 3.9.29. Appl., 20.4.28).—The product comprises a volatile solvent and an alkyl phthalate.

L. A. COLES.

XVII.—SUGARS; STARCHES; GUMS.

Special case of corrosion in cane-sugar factory.

T. VAN DER LINDEN (Rec. trav. chim., 1929, 48, 965—968).—Corrosion of brass evaporator tubes, probably initiated by hydrogen sulphide present in the juice entering the evaporator, has been observed. The hydrogen sulphide is presumably formed by microbiological action. H. BURTON.

Velocity of crystallisation of sucrose from its pure solutions. B. G. SAVINOV (Nauch. Zapiski, 1929, 7, 416—429).—The velocity increased with the speed of revolution of the agitator up to 400 r.p.m., thereafter becoming constant. CHEMICAL ABSTRACTS.

Organic acids of sugar-cane molasses. E. K. NELSON (J. Amer. Chem. Soc., 1929, 51, 2808—2810).—The volatile acids present are formic (about 0.1%) and acetic (0.2%), whilst the non-volatile acids consist of aconitic (0.8%), lactic (0.05%), and small amounts of malic and citric acids. H. BURTON.

Determination of sugar in dry [beet] slices. N. FRIZ (Zentr. Zuckerind., 1929, 37, 42; Chem. Zentr., 1929, i, 2482).—The material is soaked for 30 min. in cold water, treated with lead acetate and calcium carbonate, diluted, heated for 0.75—1 hr. at 100°, cooled to 20°, made slightly alkaline to phenolphthalein with sodium carbonate, and the invert sugar determined before and after inversion. A. A. ELDRIDGE.

Improvement of beet-sugar products treated with activated carbon. I. N. KAGANOV and B. E. KRASILSCHIKOV (Nauch. Zapiski, 1928, 6, 344—351).—Not only was the colour decreased, but also the speed of crystallisation and filtration was increased. CHEMICAL ABSTRACTS.

Boiling of [sugar] thin juice, and determination of natural alkalinity. H. DÜWELL and K. SOLON (Deut. Zuckerind., 1929, 54, 237—240; Chem. Zentr., 1929, i, 2595).—For the determination of residual alkalinity carbon dioxide, purified with potassium permanganate, is passed into 205 c.c. of hot juice containing phenolphthalein until the red colour just disappears. After addition of water (50 c.c.) the liquid is boiled for 6 min., filtered rapidly, and the alkalinity of the cooled filtrate and washings is determined by titration with 0.2N-hydrochloric acid. The calcium oxide is determined in 50 c.c. of neutralised juice with soap solution. A. A. ELDRIDGE.

Sugar titration. N. SCHOORL (Z. Unters. Lebensm., 1929, 57, 566—576).—Luff's solution (cupric sodium citrate, 0.1N in cupric salt), the preparation of which is described (cf. B., 1899, 78), is of such concentration that 1 c.c. of the reagent heated with 25 c.c. of 0.1N-hydrochloric acid for 1 hr. on the water-bath should neutralise 19 c.c. of the latter. For the sugar titration the sugar solution is added to 25 c.c. of the reagent and the volume is made up to 50 c.c. The liquid is heated to boiling over a naked flame, quickly attached to a reflux, boiled for exactly 10 min., cooled, and after 5 min. titrated by one of the following methods. (i) The excess of cupric salt may be determined by adding potassium iodide followed either by 25% sulphuric acid, or by a mixture of 25% hydrochloric acid and 20% of thiocyanate solution,

and titrating the iodine in the usual way against 0.1N-thiosulphate. (ii) The cuprous compound may be titrated by adding to the cold solution 50 c.c. of 0.4N-acetic acid, followed, after shaking, by 25 c.c. of 0.1N-iodine; after further shaking, 55 c.c. of 0.75N-hydrochloric acid are poured down the side of the flask on to the surface of the liquid, which is gently agitated till all the cuprous compound is dissolved, and the excess of iodine is then titrated with 0.1N-thiosulphate. A table is given showing the quantity of various sugars (dextrose, laevulose, lactose, and maltose) corresponding with a given amount of reduced copper measured in c.c. of 0.1N-thiosulphate under the above conditions. The influence of time of boiling and of the relative proportion of reagent and sugar has been studied for various sugars. Advantages of Luff's over Fehling's solution are indicated, e.g., the former is more stable on storage and on boiling. W. J. BOYD.

Sources of waste in the manufacture of starch. SPROCKHOFF (Z. Spiritusind., 1929, 52, 306).—The amount of starch left in the water draining from the tanks in which the raw starch settles may be on an average as much as 0.6—0.75% of the total yield, but should never exceed 0.75%. This figure corresponds to 0.08 g. of dry starch per litre of wash-water. The loss of starch during the washing process for the purification of the sludge of raw starch is usually slightly higher, but should not exceed 1% of the final yield. At this stage considerable loss may occur by fermentative action causing a decrease in the size of many of the starch grains so that the filter fabric is unable to retain them. The loss of starch in the form of starch dust during the drying process is unimportant, and in most cases is less than 0.1% of the starch output. C. RANKEN.

Amylases in dry potato starch. SPROCKHOFF (Z. Spiritusind., 1929, 52, 312—313).—In many cases the decrease in the viscosity of starch paste which cannot be attributed to the action of acid produced by bacteria is due to the presence of amylases in the original starch. That the amylases are present can be best proved from the greater viscosity of starch paste made by direct heating with water to 100°, compared with that of the paste made by first heating to 68°, cooling to 45° and retaining at that temperature for 3 hrs., and subsequently heating to 100°. If sodium chloride is present, the liquefying action of the amylases is increased. C. RANKEN.

Influence of temperature of refinery massecuite on hardness and destruction of sugar. M. I. NAKHMANOVICH and I. F. ZELIKMAN (Nauch. Zapiski, 1928, 6, 394—412).

Erratum.—Page 449, col. 1, line 22 from bottom, for "nitrite" read "nitrile."

Determination of sugar in soaps etc. BRAUN and WALTER.—See XII. **Determination of lactose.** SCHULZE. **Melezitose in honey.** NOTTBOHM and LUCIUS.—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Course of fermentation in the fermenting vessel. [Condition of yeast during primary fermentation.] F. WINDISCH (Woch. Brau., 1929, 46, 379—388).—Two

yeasts, "S" and "V," were pitched in a number of 10-litre portions of wort, and at daily intervals the proportion of suspended and sedimentary yeasts and their reproductive and fermentative powers were determined. By the end of the first day the suspended yeast was only in slight excess, and so continued until the fourth day. From the fifth day the proportion of suspended yeast gradually diminished, showing no sign of stronger sedimentation (break) at any particular reaction. Both at the beginning and end of the fermentation period of 8 days the sediment yeast showed a better biological condition than did the suspended yeast; from the third to the fifth day the difference was insignificant (cf. B., 1929, 618, 792).

F. E. DAY.

Fermentation and the surface of vessels. F. EMSLANDER (Woch. Brau., 1929, 46, 401—403).—The local acceleration of fermentation by the cooling coils, and checking by the enamel walls of brewery vessels is associated with negative and positive electrostatic charges, respectively. The possible effects of different lining materials on the colloids of the beer are discussed.

F. E. DAY.

Coagulation of proteins during boiling of unhopped wort. W. WINDISCH, P. KOLBACH, and C. VOGL (Woch. Brau., 1929, 46, 403—406, 417—419).—The amount of protein precipitated from wort by boiling is strongly influenced by the intensity and duration of heating, even in flasks connected to a reflux condenser. The most consistent results were obtained by heating in a bath of boiling salt solution (b.p. 108·8°), but heating for 4 hrs. was insufficient to obtain maximum precipitation. Comparison of boiling a wort to half volume in a beaker (70 min.), for 2 hrs. in salt solution at 108·8°, and for 2 hrs. over a burner with a layer of asbestos paper, gave 3·27—3·92 (mean 3·54), 5·94—6·36 (mean 6·23), and 7·60—9·33 (mean 8·11) mg./100 c.c., respectively, of coagulable nitrogen. Heating at 1·5 atm. for 1 hr. and 2 hrs. and for 1 hr. at 2 atm. in an autoclave gave consistent results, but below those obtained by boiling at ordinary pressure for 4 hrs. The absence of actual ebullition may explain this, as wort heated on the boiling water-bath, though only about 1° lower in temperature than that boiling on the salt-bath, yielded 7·4 mg./100 c.c. against 12·9 mg./100 c.c. of coagulable nitrogen. The optimum hydrogen-ion concentration varies with the duration of boiling from p_H 6·4 or above at 1—2 hrs. to p_H 5·4—6·2 at 5 hrs. in the case of laboratory worts. In an unhopped brewery wort maximum precipitation with boiling for 3 hrs. was found at about p_H 6·5. In the autoclave at 1·5 atm. for 1 hr. practically identical results were obtained at p_H 5·74 and 6·37, though less nitrogen was precipitated than by 4—5 hrs. boiling.

F. E. DAY.

Chemical composition and taste of potato spirit. T. CHRZASZCZ, A. KŁODNICKI, and J. SUCHODOLSKI (Przemysł Chem., 1929, 13, 257—268).—The chemical composition, smell, and flavour of potato spirit are unaffected by the substitution of iron for copper in the distilling and rectifying apparatus. The type of distilling apparatus used appears not to affect the composition and quality of the distillate where the

continuous process is applied, and the quality of the spirit obtained with two-column stills is no better than that using one column only. Installations designed for periodic production yield spirit of inferior quality. Spirit of higher quality is obtained from stills giving more highly concentrated distillates. The use of fermentation vats possessing the smallest practicable surface, which should be as smooth as possible, is advised, as minimising the danger of undesirable bacterial infection. Rotten potatoes yield inferior spirit, often possessing a repulsive taste and odour, these faults depending on the nature of the factors causing decay, and being the greater the more the potatoes are decayed. Potatoes attacked by potato cancer yield spirit of poor quality. The appearance of large quantities of foam during fermentation, as well as hyperacidity of the mash, lead to the production of inferior spirit; the evolution of hydrogen sulphide during fermentation does not involve its presence in the spirit, nor are the taste and odour of the latter to any great extent affected by this type of fermentation. Increase in the acidity of the mash does not involve the production of a more acid spirit, and the potability of the spirit is not adversely affected by its acidity, or aldehyde and ether content. The unpalatability of spirit increases with its fusel oil content, as well as with the velocity with which it decolorises permanganate solution, although no close parallel can be drawn between chemical composition and palatability.

R. TRUSZKOWSKI.

Tyramine as constituent of a Japanese vinegar. K. MIYAJI (Z. physiol. Chem., 1929, 184, 157—162).—From rice vinegar succinic and *l-p*-hydroxyphenyllactic acids were isolated, but no tyramine. All three substances were obtained from sakékasu vinegar. The latter yielded two strains of *B. megatherium*, de Bary, capable of almost quantitative conversion of tyrosine into tyramine.

J. H. BIRKINSHAW.

Relation of temperature of fermentation to quality of sauerkraut. E. A. MARTEN, W. H. PETERSON, E. B. FRED, and W. E. VAUGHN (J. Agric. Res., 1929, 39, 285—292).—The quality of sauerkraut, which depends chiefly on a proper degree of acidity, was highest when the temperature of the fermentation lay between 15° and 18°. Sauerkraut produced at 18—24° was soft and of a pink colour, whilst that fermented at 4—8° possessed a bitter taste, poor texture, and a dark colour. A rise of 1·5—3°, which occurred during the first 8 days in fermentation vats kept at temperatures above that of the surrounding air, was coincident with, and was probably due to, the greatest activity of the bacteria.

C. RANKEN.

[Examination of] foreign wines under the quartz lamp. P. BERG and L. STOCKERT (Z. Unters. Lebensm., 1929, 57, 448—460. Cf. Werder and Zäch, Mitt. Lebensm. Hyg., 1928, 19, 60, 147).—As found by Werder and Zäch (*loc. cit.*) the quartz lamp can be used to determine whether a wine has been made from dried grapes or contains a considerable quantity of such wine, provided it is known by analysis that a concentrated must has not been used. The behaviour of Samos wine in ultra-violet light confirms the view of Günther

("Der Wein," Leipzig, 1918, 660) that it is prepared from fresh grapes. In the case of dessert wines of high extract content and high luminescence it is not at present possible to draw definite conclusions from luminescence phenomena. It appears probable that alcoholic extracts of all sugar-containing dried fruits show strong luminescence. W. J. BOYD.

Behaviour of alcoholic distillates in ultra-violet light. M. RÜDIGER and E. MAYR (Z. Unters. Lebensm., 1929, 57, 561—566).—Various brandies derived from different raw materials, *e.g.*, apples, grapes, rye, maize, potatoes, were brought to an alcohol content of 33 vol.-%, and 100 c.c. of each were distilled into 9 equal fractions and 10 c.c. of residue, which were examined under the quartz lamp. The luminescence was most marked in the case of the fourth fraction and the residue. The brandies differed so little from one another that the method cannot be used for detecting adulteration of brandy. On diluting the liquors the luminescence appeared in an earlier fraction. From various brandies oily droplets of a fatty acid nature were obtained in the fraction distilling between 94° and 97°, which droplets showed strong luminescence. The luminescence of the fourth fraction was removed by precipitation with silver nitrate and restored by decomposition of the precipitate by sodium hydroxide solution. Lauric acid separated as a solid from the fractions poorer in alcohol and does not luminesce (*cf.* Grossfeld and Miermeister, B., 1929, 145). When alcoholic solutions of octoic and decanoic acids are distilled these acids separate as oily droplets which are highly and feebly luminescent respectively. Hexanoic and butyric acids give luminescent solutions without separation of droplets. Luminescence in later fractions than the fourth indicates the addition of an artificial essence to the brandy.

W. J. BOYD.

Is the volume of a saccharine liquid altered in fermentation? C. LUCKOW (Deut. Destillateur-Ztg., 1929, 50, 169; Chem. Zentr., 1929, i, 2596).—Theoretically 62.5 c.c. of dissolved sugar (100 g.) should produce 64 c.c. of alcohol; a 2.5 % deficiency is usually observed, but the volume is increased by the secondary products. In practice the volume is unchanged.

A. A. ELDRIDGE.

isoValeric acid from fusel oil. PIOTROWSKI.—See III. **Removal of fermentation-restraining humic substances from hydrolysates of hemicelluloses.** SCHMIDT and others.—See V. **Micro-determination of phosphate ions.** DENIGÈS. **Determination of nitrogen.** WIENINGER and LINDEMANN.—See VII. **Barley fat and malt fat.** TÄUFEL and RUSCH.—See XII. **Amylases in dry potato starch.** SPROCKHOFF.—See XVII. **Effect of flour on yeast.** MOHS and KÜHL.—See XIX.

PATENTS.

Drying apparatus for hops and like materials. R. D. BATCHELOR (B.P. 319,394, 20.6.28).—The hot gases in their journey from the fire to the hops pass through filter screens which are hinged and readily movable. Each screen consists of a layer of horse hair, woven asbestos, or other fibrous material, supported,

if desired, on wire netting. Spent iron oxide, lime, or other chemical reagent may be present on the screens.

C. RANKEN.

Valuable products from organised substances. R. GRIESSBACH and O. AMBROS, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,724,706, 13.8.29. Appl., 31.3.28. Ger., 6.4.27).—In the production of compounds from organised substances the cell walls are subjected to enzymic degradation before the usual extraction and pressing, so that the products can be readily separated without decomposition. The yield of ergosterol from yeast is increased by adding ethyl acetate, to initiate autolysis, before extracting with alcohol.

F. G. CLARKE.

Production of butyl alcohol and acetone by fermentation. DISTILLERS CO., LTD., and H. B. HUTCHINSON (B.P. 319,079, 25.6.28).—Starchy materials, such as manioc and the like, which have a low content of protein, are fermented in the presence of the non-protein nutrient ammonium phosphate. The process is applicable to every type of organism capable of producing butyl alcohol and acetone from starchy materials.

C. RANKEN.

Cereal beverage and liquid. H. HEUSER, Assr. to UNITED STATES PROCESS CORP. (U.S.P. 1,724,021, 13.8.29. Appl., 1.8.27).—Cerealliquids are stabilised after alcoholic fermentation by the addition of a quantity of pyrocatechol tannic acid not less than that required to precipitate the readily precipitable proteins.

L. A. COLES.

Briquettes (B.P. 294,879).—See II.

XIX.—FOODS.

Bleaching of flour. A. HEIDUSCHKA and A. WARLIMONT (Z. Unters. Lebensm., 1929, 57, 587—592).—The bran of flour is not sufficiently bleached by benzoyl peroxide and Novadelox to remove its undesirable effect on the appearance of the flour. Bleaching with benzoyl peroxide improves the baking qualities of the flour. There is a definite proportion of the agent which gives optimum results for a given flour; excess imparts a bluish tint to the bread. Bleaching at higher temperatures gives no better results. The action of benzoyl peroxide increases the water-soluble material of the flour in proportion to the quantity of the agent used. At the same time the total water-soluble carbohydrate decreases; water-soluble nitrogenous material and acid are unchanged.

W. J. BOYD.

Test for bleaching of flour. J. KULMAN (Chem. Listy, 1929, 23, 333—336, 375—382).—Flour bleached with chlorine or with nitrogen peroxide gives darker coloured benzene extracts than does unbleached flour. Aged flour, however, also yields highly coloured extracts, so that in order definitely to establish whether the flour has been bleached, extraction with benzene-toluene or -xylene mixtures is applied, when brown or red chloro- or nitro-aromatic derivatives are obtained, unbleached flour giving light yellow extracts under these conditions.

R. TRUSZKOWSKI.

Effect of flour on the fermentative power of yeast. K. MOHS and H. KÜHL (Z. Unters. Lebensm., 1929, 57, 443—448).—The toxicity of normal flour

towards yeast is changed to a favourable effect on increasing the degree of milling, owing to the increased proportion of bran, or by the addition of soya-bean flour. This is probably due to the phosphates contained in bran and bean flour. The diastase of germinating wheat has a favourable effect on yeast fermentation, counteracting the toxicity of the flour. High acidity of spoilt flour is not invariably accompanied by a correspondingly increased toxicity such as might be expected if the toxamine were a protein decomposition product. A high toxicity towards yeast does not unfavourably influence the baking qualities of the flour.

W. J. BOYD.

Utilisation of skim milk. A. BICKEL (Z. Unters. Lebensm., 1929, 57, 437—443).—The high nutritive value of "plasmon" (casein) in comparison with vegetable proteins is shown by the results of feeding tests on rats.

W. J. BOYD.

Secretion of lipolytically active "rancid" milk. G. KOESTLER, C. L. ROADHOUSE, and W. LÖRTSCHER (Landw. Jahrb. Schweiz, 1928, 42, 937—966; Chem. Zentr., 1929, i, 2116).—The occurrence of milk naturally containing lipase is discussed; a sample was high in caseinogen, lactose, and calcium, but low in citric acid and chlorine.

A. A. ELDRIDGE.

Determination of lactose [in milk] from the refraction of the calcium chloride serum. G. SCHULZE (Z. Unters. Lebensm., 1929, 57, 460—465).—The observation of Drost (B., 1925, 822) that Ackermann's table (B., 1917, 1025) for finding the lactose content of milk from the refraction of the serum gives results considerably lower than those found by gravimetric methods is confirmed. A new table, prepared by determinations of the refraction and of the lactose by Allihn's method, is provided which gives the true lactose content corresponding to the refraction if the milk is normal. If the content of lactose gravimetrically determined is lower than that given by the table, the presence of milk from diseased udders is indicated. A method of distinguishing diluted milk and milk defective through disease is thus available. The natural variations at the beginning and end of lactation must be considered.

W. J. BOYD.

Melezitose in honey-dew honey from the lime. F. E. NOTTBOHM and F. LUCIUS (Z. Unters. Lebensm., 1929, 57, 549—558).—The trisaccharide melezitose was found in honey-dew honey from the lime as a grey sediment consisting of anhydrous lamellæ (m.p. 153—156°) and rhombic prisms containing water of crystallisation which was completely driven off by heating in the water-oven for 2 hrs. The sugar was much less soluble in water than sucrose. It had a faintly sweet taste and reduced Fehling's solution very slowly. Hydrolysis with 1% sulphuric acid reduced the specific rotation from 89.5° to 66°. More concentrated acid reduced it further to 26.0°, but after destruction of the lævulose formed it rose to 36.0°, or to two thirds that of dextrose. The intermediate products were turanose and dextrose, identified by their osazones. Turanose was shown to consist of lævulose and dextrose. The insolubility of melezitose in water and its great resistance to the action of the ordinary sugar-splitting

enzymes are suggested as the cause of the toxic action on bees which such a honey possesses. Yeast, invertase, and diastase have no effect on it. (Cf. Kuhn and von Grundherr, A., 1926, 1127.)

W. J. BOYD.

Detection of [hydr]oxymethylfurfuraldehyde in honey and artificial honey. J. FIEHE and W. KORDATZKI (Z. Unters. Lebensm., 1929, 57, 468—470).—The honey (5 g.) is stirred in a mortar with 10 c.c. of ether, and the ether is poured off and gently evaporated. The residue is dissolved in 0.5 c.c. of water, and to the solution 0.5 c.c. of 32% hydrochloric acid is added followed by 1 c.c. of 0.625% solution of phloroglucinol in 16% hydrochloric acid. A reddish coloration and a red-brown precipitate are formed in the presence of hydroxymethylfurfuraldehyde. Neither coloration nor precipitate occurs when pure honey is used. By comparison with synthetic mixtures of natural and artificial honey the height of the layer of sediment can be used as a measure of the adulteration of any sample. Genuine honey heated at 80° for 2 hrs. and then warmed for 2 hrs. on the boiling water-bath gives no precipitate, but with Fiehe's resorcinol reagent an evanescent red coloration is obtained and with phloroglucinol a strong lemon-yellow coloration which gradually fades. The latter reaction is characteristic for heated honey and is not due to hydroxymethylfurfuraldehyde, for the production of which the presence of acid is necessary.

W. J. BOYD.

Determination and content of water in dried fruit. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1929, 57, 520—524).—The apparatus described is suitable for the determination of water in dried fruits by distillation with tetrachloroethane, xylene, or other volatile liquid lighter or heavier than water. It can also be used for the determination of water in coal or other commodities. With 75 c.c. of tetrachloroethane and 25 g. of dried fruit a determination can be completed in 20 min. Good agreement was found between the tetrachloroethane and the xylene methods. The drying-oven method requires 10—14 hrs. and gives less uniform results. Moisture contents found for various dried fruits are given. Dried apples, apricots, and peaches should contain not more than 30% of water.

W. J. BOYD.

Determination of husk in cocoa and cacao products. M. WAGENAAR (Z. Unters. Lebensm., 1929, 57, 525—537).—The sclereide tissue in 1 g. of fat-free cocoa powder is isolated by heating with 20 c.c. of 5% potassium chlorate solution and 10 c.c. of 4N-hydrochloric acid for 10 min. on the water-bath. Then 25 c.c. of 4N-sodium hydroxide are added, and the heating is continued for 10 min. more. After settling, the clear liquid is drawn off, the sediment washed repeatedly by decantation, and recovered by centrifuging. The whole or an aliquot portion is suspended in a weighed quantity of glycerin, 6 drops of known total weight are placed on slides under calibrated cover-slips, and the total area of sclereide tissue in them is measured, using polarised light (1 g. of fat-free powdered husk contains 2100 mm.² of sclereide tissue.) The content of husk in 100 g. of fat-free cocoa is obtained by dividing the area of sclereide tissue in 1 g. by 21.

W. J. BOYD.

Chemical and physical examination of flesh and flesh fluid of various animals : the Feder ratio. A. GRONOVER and E. WOHLICH (*Z. Unters. Lebensm.*, 1929, **57**, 592—604).—Efforts to establish chemical and physical constants (*e.g.*, f.p. depression, sp. conductivity, sp. gr., and chlorine content) for the flesh sera of different animals were unsuccessful. Not only different individuals but different portions of the same individual gave widely varying values. Analyses are given for flesh from different parts of various animals. That of calves only a few weeks old has usually a Feder value above 4, whereas that of normally-fed cattle is usually below 4 or only slightly above. Variations in the water content of different parts were inconsistent and sometimes even reversed in different individuals. Nevertheless, contrary to the opinion of Bongert and Muchlinsky (*ibid.*, 1929, **57**, 248), determinations of the Feder value are of great use in detecting addition of water to minced flesh, but suspicions founded thereon must be confirmed otherwise. Naturally-occurring moisture in fat has little influence on the Feder value. W. J. BOYD.

Spectroscopy of food dyes. HEATH.—See IV. **Micro-determination of phosphate ions.** DENIGÈS. **Determination of nitrogen.** WIENINGER and LINDEMANN.—See VII. **Metals in dairy equipment.** HUNZIKER and others.—See X. **Quality of sauerkraut.** MARTEN and others.—See XVIII.

PATENTS.

Reduction of grain and its subsequent conversion into bread or like product. E. RABINOWITSCH (B.P. 317,571, 8.6.28).—The cleaned and dried grain is washed for $\frac{1}{2}$ — $\frac{3}{4}$ hr. in 8% sulphuric acid, which is then removed thoroughly by running water. The grain is allowed to steep for about 24 hrs., when germination begins and lasts for 24—36 hrs., during which the grain is exposed on a perforated tray to a current of air and a stream of water. When the decomposition of the gluten reaches the requisite stage, 2.5% of salt, 0.3% of lime (added as 10% lime water), and 0.5% of glycerin are added and the mixture is kneaded to a coarse dough, which is fermented in a closed space at 86—95° for 12—14 hrs., the pressure rising to about $4\frac{1}{2}$ atm. When fermentation ceases the pressure is suddenly released, yeast is kneaded in, and the dough baked into wholemeal bread. E. B. HUGHES.

Removing caffeine and unsavoury substances from roasted coffee. J. PÄFFGEN (B.P. 302,332, 16.8.28).—Coffee liquor as prepared for drinking is filtered through absorbent material such as active carbon, whereby it is claimed that caffeine, pyridine, etc. may be removed, the carbon being preferably treated with fumes from the roasting of coffee to render it a non-absorbent of the volatile (aroma) constituents of the liquor. In another example an absorbent, obtained by treating suitable organic substances with zinc chloride, is treated with smoke gases of aromatic resins. Suitable apparatus is described. E. B. HUGHES.

Production of edible paste from the liver of cod fish and the like. H. J. EGGE (B.P. 319,529,

29.10.28).—The liver is treated with dilute acid to remove surface (oxidised) oil, then heated to 70°, converted into a paste with casein etc. as binder, canned, and sterilised. E. B. HUGHES.

Cereal beverage (U.S.P. 1,724,021).—See XVIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Purification and preservation of ether for anæsthetic use. S. PALKIN and H. R. WATKINS (*Ind. Eng. Chem.*, 1929, **21**, 863—867).—The tendency of ether to develop peroxide and aldehyde during storage is entirely stopped by the addition to the container of a few g. of a preservative consisting of fibrous asbestos moistened with (a) a saturated solution of potassium permanganate in 30% caustic soda or potash, or (b) 1 g. of pyrogallol in 15 c.c. of 30% caustic potash. Comparative tests, lasting over a year, showed that deterioration apparent in the untreated samples was thereby completely inhibited, even when the ether was exposed to direct sunlight and elevated temperatures, and no contamination of the ether resulted by such addition. T. H. MORTON.

Purification of ethyl acetate for the evaluation of opium and opium preparations. G. VON MIKÓ (*Ber. Ungar. pharm. Ges.*, 1928, **4**, 392—396; *Chem. Zentr.*, 1929, i, 1846).—Ethyl acetate which has suffered decomposition is shaken with 0.15% calcium hydroxide solution; it is preferably preserved over neutral potassium tartrate. A. A. ELDRIDGE.

Determination of chlorine and cyanogen in chloral cyanohydrin. G. FAVREL (*Bull. Soc. Pharm. Bordeaux*, 1928, **66**, 139—142; *Chem. Zentr.*, 1929, i, 2088).—The chloral cyanohydrin (0.5 g.; cf. Carré, *Prod. pharm. ind.*, 1909, **2**, 37) is boiled for 0.5 hr. under reflux with amyl-alcoholic sodium hydroxide; the cold liquid is extracted several times with water. The extracts are diluted to 200 c.c.; in 100 c.c. the cyanide is determined with silver nitrate by Denigès' method, whilst the remainder is boiled with nitric acid and silver nitrate to determine the chlorine gravimetrically or volumetrically. The method is inapplicable to nitriles. A. A. ELDRIDGE.

Synthesis of camphor. Purity of isobornyl acetate. M. T. LACRUE (*Bull. Inst. Pin.*, 1929, Special No., 15—30; *Chem. Zentr.*, 1929, i, 2303).—Losses occur in the "borneol oil," the amount of which is diminished when purer (97—99%) acetate is employed. The fractionation of the 90% product is discussed. A. A. ELDRIDGE.

Preparation of a solution of iron albuminate. I. A. OBERHARD (*Farm. Zhur.*, 1928, 702—705).—Dilute solutions of ferric chloride and albumin at 50° are mixed and the solution is neutralised with 15% sodium hydroxide solution at 50°. The precipitate is washed free from chlorine, dissolved by the addition of alkali, and the remaining pharmacopœial components are added. CHEMICAL ABSTRACTS.

Behaviour of some colloidal silver preparations in aqueous solution. O. TOMIČEK (*Časopis Českoslov. Lék.*, 1928, **8**, 196—202; *Chem. Zentr.*, 1929, i, 2443).—

The proportion of ionised silver in commercial preparations has been determined, usually potentiometrically.

A. A. ELDRIDGE.

Titration of mercurial preparations used in pharmacy. A. WØLLK (Dansk Tidsskr. Farm., 1929, 3, 225—244).—(a) In the analysis of tablets of mercuric chloride containing sodium chloride, 20 c.c. of an aqueous solution of the tablets (containing about 2.5% HgCl_2), 50 c.c. of 0.1*N*-caustic soda, and 5—6 c.c. of 3% hydrogen peroxide are boiled until the precipitated mercury has agglomerated. The solution is filtered and the excess caustic soda in the filtrate titrated against methyl-red with 0.1*N*-hydrochloric acid. (1 c.c. of $\text{N-NaOH} \equiv 0.01356$ g. of HgCl_2 .) (b) Rupp's method (cf. B., 1908, 1179) gives satisfactory results with solutions of mercuric chloride (from 1% upwards). (c) In the examination of the solution of mercuric chloride (1 pt.) in 5% acetic acid (300 pts.), 50 g. of the solution, 15 g. of Liq. kalii arsenicosi, and 30 c.c. of 2*N*-caustic soda are boiled for 5 min., cooled, and diluted with 100 c.c. of water. After addition of 8 c.c. of 30% acetic acid and 3 g. of sodium bicarbonate, the solution is titrated with 0.1*N*-iodine, using starch indicator. (d) For solutions containing not less than 1% of mercuric chloride the following method is recommended. Mercury is precipitated by potassium iodide, the end-point being indicated by the liberation of iodine by hydrogen peroxide. A mixture of 20 c.c. of the mercurial solution, 0.5—1.0 c.c. of 3% hydrogen peroxide, 5 c.c. of dilute sulphuric acid, and 3—5 c.c. of starch solution is titrated with 0.1*N*-potassium iodide until the mercuric chloride turns brick-red. Blue rings form above the precipitate and, on shaking, the whole liquid turns blue. (e) Mercuric oxide can be determined (i) by dissolving in hydrochloric acid and, after adding sodium chloride, titrating residual acid with caustic soda; or (ii) by dissolving the oxide in water containing excess of sodium thiosulphate (10 times the weight of oxide), maintaining the temperature below 35° to prevent decomposition of the complex, and titrating the resultant sodium hydroxide with hydrochloric acid. (f) For the determination of calomel iodometric methods are recommended. An indirect method is to boil the material with hydrogen peroxide and caustic soda (cf. a). (g) Mercuric amidochloride may be determined by decomposition with potassium iodide or sodium thiosulphate followed by titration of the liberated base, or by titration with hydrochloric acid in the presence of sodium chloride. (h) Mercuric cyanide and oxycyanide may be determined by decomposition with sodium thiosulphate or potassium iodide. The alkali cyanide is titrated with acid against methyl-orange. (i) For the determination of mercuric oxide or mercuric amidochloride in ointments, the ointment base is removed by dissolving in benzene or ether-acetone, the residue being then dissolved in acid and the excess titrated back after addition of sodium chloride.

H. J. DOWDEN.

The tannin of the purple digitalis. P. BOURCET and A. FOURTON (Bull. Soc. chim., 1929, [iv], 45, 776—778).—The green coloration given by the digitalis extracts from the fresh purple digitalis is due to caffeic acid and not to the presence of gallic acid or tannins in the soluble glucoside complex.

R. BRIGHTMAN.

Determination of fats in drugs. E. GLASER and A. HALBERSTAM (Arch. Pharm., 1929, 267, 526—532).—Sulphuric acid (d 1.81; 10 c.c.) is introduced into a Gerber butyrometer, a small amount of water added cautiously, then 0.4—1.0 g. of the powdered drug, 1 c.c. of amyl alcohol, and sufficient water to make a total volume of 22 c.c. The mixture is agitated, heated at 60—70° for a short time, centrifuged, and re-warmed. The volume of fat which separates is converted into weight by means of a factor which varies somewhat for the substances used. This factor is determined by the usual ether extraction method. If the fat layer is contaminated with the powder used, it is advisable to carry out the determination with a smaller quantity (7—8 c.c.) of sulphuric acid.

II. BURTON.

Application of "critical solution temperature" to pharmaceutical investigations. F. WRATSCHKO (Pharm. Presse, 1929, 34, 143—145).—Values for a number of pairs of organic substances are recorded.

CHEMICAL ABSTRACTS.

Application of various micro-methods in qualitative toxicological and pharmaceutical analysis. L. FUCHS and A. MAYRHOFER (Mikrochem., 1929, Pregl Fest., 106—126).—The application of microchemical methods, especially sublimation and determination of the refractive index of the crystalline sublimate, to the identification of various pharmaceutical products has been studied, and the results obtained are critically discussed.

II. F. HARWOOD.

Transparent emulsions of some essential oils. W. F. WHITMORE and R. E. LINEHAN (Ind. Eng. Chem., 1929, 21, 878—880).—The properties of transparent emulsions prepared by dispersing the oil in an aqueous solution of invert sugar having a refractive index equal to that of the oil are described. Of gelatin, gum-arabic, agar-agar, and tragacanth, the first was the best peptising agent and the last two were unsatisfactory. Transparent emulsions of orange, peppermint, and rose oils were easily made by this method, but for the preparation of emulsions of wintergreen, anise, and cinnamon oils the refractive indices of the oils themselves had to be lowered by the addition of coconut oil ethyl esters to a value low enough to permit the use of an aqueous sugar solution of sufficiently low concentration to pass through the colloid mill without difficulty. These emulsions developed a coconut oil taste on keeping. Graining of certain of the emulsions was obviated by using a mixture of 2 pts. of invert sugar and 1 pt. of sucrose instead of invert sugar alone.

E. H. SHARPLES.

Constituents of East Indian lemon-grass oil. F. ELZE (Riechstoffind., 1929, 4, 23—24; Chem. Zentr., 1929, i, 2248).—Methylheptenol, methylheptenone, geraniol, nerol, and farnesol were isolated.

A. A. ELDRIDGE.

Essential oil of *Juniperus excelsa*. G. V. FIGULEVSKI and Y. N. LOVYAGIN (Trans. Sci. Chem. Pharm. Inst. Moscow, 1927, No. 17, 151—158).—The b.p. and yields of fractions are tabulated. Pinene and cedrol were isolated.

CHEMICAL ABSTRACTS.

Essential oils from Irish-grown plants. V. Oil of dill. J. REILLY, P. J. DRUMM, and C. BOYLE (Econ.

Proc. Roy. Dublin Soc., 1929, 2, 415—418).—Following a review of the cultivation of *Anethum graveolens* and the chemical properties of dill oil produced in Europe, India, and the East Indies, it is shown that *A. graveolens* grown near Cork gave in 1926—27, 7450 lb. of fruit and 258 lb. of redistilled oil per acre, a 3½% yield of oil. It had $d_{20} 0.9135$, $n_D^{20} 1.48513$, $\alpha_D^{20} 70^\circ 54'$, percentage of carvone 37.8, and solubility in 70% alcohol 1:6. Similar values were obtained in 1927—28, except that the percentage of carvone was 43—44. F. HOLMES.

Modified determination of total geraniol content in citronella oil. M. VAN DER SLIK and J. VERMEULEN (Chem. Weekblad, 1929, 26, 482—483).—The use of anhydrous sodium acetate involves in a damp climate repeated preparation of fresh material, and is laborious and slow. Anhydrous sodium carbonate is found to give equally satisfactory results. The data obtained with 97 samples, using both acetate and carbonate, are tabulated. S. I. LEVY.

Nerol and its esters. A. RECLAIRE (Deut. Parfüm. Ztg., 1929, 15, 71—73; Chem. Zentr., 1929, i, 2249).—The following limiting data for neryl esters are recorded: formate, $d_{15}^{25} 0.9163$, $n_D^{20} 0.9169$, $n_D^{20} 1.4558$, $n_D^{20} 1.4578$; acetate $d_{15}^{25} 0.903$, $n_D^{20} 0.906$, $n_D^{20} 1.451$, $n_D^{20} 1.454$; propionate $d_{15}^{25} 0.9044$, $n_D^{20} 1.4550$; isobutyrate $d_{15}^{25} 0.8915$, $n_D^{20} 0.8936$, $n_D^{20} 1.4508$, $n_D^{20} 1.4527$; butyrate $d_{15}^{25} 0.8968$, $n_D^{20} 0.8986$, $n_D^{20} 1.4539$, $n_D^{20} 1.4536$; isovalerate $d_{15}^{25} 0.8898$, $n_D^{20} 1.4531$.

A. A. ELDRIDGE.

Evaluation of Liquor cresoli saponatus according to D.A.B. VI. K. KRAFFT and G. ZEITLER (Süddeut. Apoth.-Ztg., 1928, 68, 613—615; Chem. Zentr., 1929, i, 2212).

Determination of iodoform. JÁNSKÝ.—See III. **Determination of hydrogen peroxide.** FRERICHs. **Determination of phosphorus in "Phosphor solutus."** BÖTTGER.—See VII. **Vitamins in canned foods.** KRAMER and others.—See XIX.

PATENTS.

Manufacture of pharmaceutical products. I. G. FARBERIND. A.-G. (B.P. 282,453, 19.12.27. Ger., 20.12.25. Addn. to B.P. 267,169; B., 1927, 379).—Amino-derivatives of the quinoline, di- and tri-arylmethane, azine, oxazine, thiazine, acridine, and xanthen series are prepared in which the amino-group carries a substituent comprising a nitrogenous alicyclic or heterocyclic residue linked to the nitrogen directly or through an aliphatic carbon chain. From 8-aminoquinoline and 1-β-chloroethylpiperidine is obtained 8-(β-1-piperidylethyl)aminoquinoline, m.p. 59—60°, b.p. 180—182°/1 mm. (hydrochloride, m.p. 190—191°). 2-Bromocyclohexyldimethylamine, prepared from the alcohol and hydrogen bromide, gives with 8-amino-6-methoxyquinoline 8-(2-dimethylamino-1-cyclohexyl)amino-6-methoxyquinoline, b.p. 192—195°/1 mm. Methyltriacetonealkamine is converted by hydrogen bromide into 4-bromo-1:2:2:6:6-pentamethylpiperidine, which reacts with 8-amino-6-methoxyquinoline to yield 8-(1:2:2:6:6-pentamethyl-4-piperidyl)amino-6-methoxyquinoline, b.p. 215—218°/0.5 mm. 1-β-(N-Methylanilino)ethylpiperidine, obtained from methylaniline and 1-β-chloroethylpiperidine, is converted

by means of nitrosodimethylaniline and sodium thiosulphate into the thiazine. C. HOLLINS.

Manufacture of unilaterally acylated diamines of therapeutic activity. O. Y. IMRAY. From Soc. CHEM. IND. IN BASLE (B.P. 307,719, 6.12.27).—Monoacylated diamines having depressant or other therapeutic action are obtained from alkylenediamines, especially NN-diethylethylenediamine, and heterocyclic acids. The following are described: N-β-diethylaminoethylamides of 2-phenylquinoline-4-carboxylic acid [m.p. 70—75°; dihydrochloride, m.p. 185—186° (decomp.); isomeric 1:2:3:4-tetrahydro-derivatives, m.p. 132° and 97.5°, respectively (dihydrochlorides, m.p. 192° and 225°, respectively, both decomp.)], of 2-ethoxyquinoline-4-carboxylic acid (m.p. 97°), of 3-β-hexyl-2-naphthaquinoline-1-carboxylic acid (dihydrochloride, m.p. 274—275°), of acridine-*ms*-carboxylic acid (m.p. 105—106°; 1:2:3:4-tetrahydro-derivative, m.p. 97—98°), of 1-phenyl-5-pyrazolone-3-carboxylic acid (hydrochloride, m.p. 220°), of 5:6-methylenedioxy-8-phenyl-2:3-dihydro-isoquinoline-2-carboxylic acid (oxalate, m.p. 206—207°), of 6-ethoxy-2-phenylquinoline-4-carboxylic acid (m.p. 127—128°), of quinoline-3-carboxylic acid (b.p. 245°/4 mm.), of 2-methoxyquinoline-4-carboxylic acid (m.p. 94°), of 2:3-diphenylquinoline-4-carboxylic acid (m.p. 146—147°), of 2-β-phenylethylquinoline-4-carboxylic acid (m.p. 98—99°), and of 2-styrylquinoline-4-carboxylic acid (m.p. 98—99°; hydrochloride m.p. 122°); N-β-diethylaminoethyl-N-ethylamides, NEt₂CH₂CH₂NEt.COR of 2-phenyl-1:2:3:4-tetrahydroquinoline-4-carboxylic acid, of 1:2:3:4-tetrahydroacridine-*ms*-carboxylic acid (b.p. 189—191°/0.015 mm.), and of 2-β-phenylethylquinoline-4-carboxylic acid (b.p. 187—188°/0.007 mm.); 6-aminoethylamide of 2-phenylquinoline-4-carboxylic acid (m.p. 124—125°); 3-N-piperidinoethylamides of 2-phenylquinoline-4-carboxylic acid (hydrobromide, m.p. 102°), of pyridine-3-carboxylic acid (hydrochloride, m.p. 182—183°), and of quinoline-4-carboxylic acid; and acridine-*ms*-carboxylic N-ε-diethylamino-*n*-amylamide, m.p. 84°. NNN'-Triethylethylenediamine has b.p. 160—165°; 2-β-phenylethylquinoline-4-carboxylic acid melts at 221—222°. C. HOLLINS.

Anæsthetic compound. R. ADAMS and O. A. BARNES, Assrs. to ABBOTT LABS. (U.S.P. 1,724,248, 13.8.29. Appl., 13.1.28).—ω-Piperidylalkyl *p*-aminobenzoates, prepared as already described (A., 1927, 672), are claimed. R. BRIGHTMAN.

Manufacture of 4-amino-3-hydroxyphenylarsinic acid. I. E. BALABAN, and MAY & BAKER, LTD. (B.P. 309,249, 24.1.28. Cf. Balaban, A., 1928, 655).—4-Nitro-3-hydroxybenzenearsinic acid, obtained by hydrolysis of the 3-chloro-compound with alkali, is reduced with alkaline glucose, hyposulphite, or ferrous sulphate solution. C. HOLLINS.

Manufacture of pyridines. I. OSTROMISLENSKY, Assr. to PYRIDIUM CORP. (U.S.P. 1,724,305, 13.8.29. Appl., 23.9.27).—Benzenediazonium salts are coupled in weakly acid medium with 2:6-diaminopyridines, and the *py*-diazoamino-compound, e.g., 2-phenyldiazoamino-6-aminopyridine, m.p. 117°, yellowish-brown, is converted quantitatively into, e.g., 4-benzeneazo-2:6-

diaminopyridine, m.p. 137°, by boiling its hydrochloride with water. B. BRIGHTMAN.

Obtaining and separating physiologically active substances from male generative organs. Soc. CHEM. IND. IN BASLE (B.P. 298,092, 1.10.28. Switz., 30.9.27).—The male generative organs, *e.g.*, bull testicles, are extracted with water-soluble solvents, *e.g.*, alcohol, and with water-insoluble solvents, *e.g.*, ether, in either order, the two solutions are mixed, preferably after concentration of the alcoholic extract, the mixture with water is shaken, and, after settling, the aqueous and ethereal layers are worked up separately into water-soluble and lipid-soluble extracts, respectively. L. A. COLES.

Extracting nicotine and ammonia from tobacco and its products. F. R. ABLETT (B.P. 317,564, 5.6.28).—The tobacco etc. is heated at 60–205° on perforated trays in an oven provided at the top with an outlet pipe conveying vapours etc. to a "scrubber" filled with substances, *e.g.*, charcoal, lime, tannic acid, phosphotungstic acid, gypsum, etc., having an affinity for the deleterious constituents in the vapour, and the residual gases are pumped from the scrubber into the lower end of the oven. At the end of the treatment the tobacco is cooled in the oven after the addition of sufficient water to restore its normal moisture content. L. A. COLES.

Manufacture of a chlorinised and ozonised topical remedy. B. E. CLARKE (U.S.P. 1,724,562, 13.8.29. Appl., 10.12.25).—A vehicle, *e.g.*, vaseline or cacao butter, is impregnated with chlorine and ozone by passing the latter mixed with pure dry air into it in melted condition. On contact with the body nascent oxygen and chlorine are liberated. F. G. CLARKE.

Pharmaceutical products. H. HAHN and L. SCHÜTZ, ASSRS. to WINTHROP CHEM. CO., INC. (U.S.P. 1,723,695—6, 6.8.29. Appl., [A] 27.8.23, [B] 24.3.27. Ger., [A, B] 6.11.22).—See G.P. 406,151; B., 1925, 426.

Manufacture of arseno-compounds of the pyridine series. A. BINZ and C. RÄTH (U.S.P. 1,702,334, 19.2.29. Appl., 8.11.24. Ger., 19.11.23).—See B.P. 250,287; B., 1926, 512.

Removing caffeine from coffee (B.P. 302,332).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photography and the wave-theory of light. Clash between ray and wave theories of image formation. T. SMITH (Proc. 7th Internat. Congr. Phot., 1928, 276–292).—The ray and wave theories of image formation are discussed. J. W. GLASSETT.

Photographic method of investigating the colour of light sources, and the reflecting power of coloured fabric and other substances. P. W. CUNLIFFE and F. D. FARROW (Proc. 7th Internat. Congr. Phot., 1928, 327–333).—The light from the source to be investigated is divided into a series of spectral bands by transmission through a number

of colour-filter strips arranged side by side, and the intensity in each wave-length band is compared by the normal photographic wedge method with the intensities in the corresponding bands of a standard light source. J. W. GLASSETT.

Function of gelatin in photographic emulsions. S. E. SHEPPARD (Phot. J., 1929, 69, 330–336).—It is suggested that the protective action exerted by gelatin on the silver halides depends on the formation of an oriented adsorption layer on the surface of the halide of certain groups in the gelatin containing active nitrogen atoms. Evidence is given for the existence of the :C:NH and $\cdot N < \begin{smallmatrix} C \cdot C \\ C \cdot C \end{smallmatrix} > N \cdot$ groups in gelatin, and

an attempt is made to correlate the reversible effect on the sensitivity of emulsions caused by changes in p_H with the modification of this adsorption layer produced by the tautomeric changes taking place under the influence of p_H changes in compounds possessing such groups. J. W. GLASSETT.

Photographic sensitivity of photographic layers. F. WEIGERT (Proc. 7th Internat. Congr. Phot., 1928, 87–91).—It is suggested that the changes which produce photodichroism in photochloride layers also cause developability of the silver halide. The silver halide crystals are not homogeneous, but contain complexes of gelatin, primary silver, and silver halide, the structure of which is altered by the action of the light in such a way that the action of the reducing agents during development is promoted by the resultant change in absorption properties. J. W. GLASSETT.

Theory of photographic light sensitivity. A. STEIGMANN (Proc. 7th Internat. Congr. Phot., 1928, 258–262).—The sensitiveness of high-speed emulsions is not explained merely by the presence of ripening nuclei, for it has been shown that there are a large number of ripening nuclei in a high state of dispersion upon the surface of the fine grains of emulsions of low sensitivity. The high sensitivity of the rapid emulsion is due to the fact that the relatively few active ripening nuclei strongly orient the crystallisation of the silver formed by the decomposition of the silver halide, thereby causing a rapid growth of the nuclei to a size sufficient to induce development. J. W. GLASSETT.

Preparation of a paper film for negatives and positives. L. HERVÉ (Bull. Soc. Franç. Phot., 1929, 16, 182–188).—A linen-rag paper of medium strength is bathed in a solution composed of 60 g. of soft gum copal dissolved in 500 g. of benzene, dried, and mounted on a flat glass plate. A coating of a 10% gelatin solution is then applied, allowed to dry slowly, and the emulsion poured on. When dry the paper film is stripped from the glass support. J. W. GLASSETT.

Contrast of photographic printing paper. L. A. JONES (Proc. 7th Internat. Congr. Phot., 1928, 216–235).—The contrast of the photographic paper is dependent on (a) the rate factor, which is given by the value of the gradient of $dD/d \log E$ of the characteristic curve ($D = \log E$), and (b) on the extent factor, which is the difference between the maximum and minimum

densities. A contrast value, which comprises both these factors, is given by the area under the curve obtained by plotting the density (D) against the gradient ($dD/d \log E$) at those densities. J. W. GLASSETT.

Expression of [photographic] plate speed in terms of minimum useful gradient. L. A. JONES and M. E. RUSSELL (Proc. 7th Internat. Cong. Phot., 1928, 130—143).—The specification of speed in terms of inertia is not altogether satisfactory, and fails particularly in those cases where the foot of the characteristic curve alone is being used. A more useful expression of speed, especially from the point of view of tone reproduction requirements, may be derived from the exposure (E) at which a fixed minimum useful gradient ($dD/d \log E$ of the D -log E curve, where D is the measured density) is obtained, measured at a definite degree of development. The most suitable value of the minimum useful gradient to cover all classes of work is the subject of further experiments. J. W. GLASSETT.

Dependence of the resolving power of photographic materials on the contrast in the test object. O. SANDVIK (Z. wiss. Phot., 1929, 27, 60—76, and Proc. 7th Internat. Congr. Phot., 1928, 243—256).—With low contrast values the resolving power was found to be very small, but it rapidly increased with increasing contrast. The relation between the resolving power (R) and the density (D) of the opaque parts of the test object, i.e., the measure of the contrast, can be expressed by means of the exponential equation $R = C(1 - e^{-\alpha D})$, where C and α are constants. The resolving power varied widely with the exposure, but was almost independent of the degree of development. J. W. GLASSETT.

Relation between time and intensity in photographic exposure. L. A. JONES and V. C. HALL (Proc. 7th Internat. Congr. Phot., 1928, 115—128).—With rapid emulsions the density developable for a fixed exposure varies very little over a wide range of intensities, whilst for slow emulsions the density diminishes rapidly at low intensities. The optimal intensity, at which the density produced is greatest, tends towards higher values for slow emulsions. The behaviour of emulsions above and below the region of optimal intensity may be represented by the equation $\log E = \log [(I/t_0)/2] [(I/I_0)^a + (I/I_0)^{-a}]$, which expresses the relation between the exposure (E) required to produce a constant density at any intensity I , and I_0 and t_0 , the values of intensity and time at optimal intensity for the density in question. J. W. GLASSETT.

Mechanism of latent image formation. F. C. TOY (Proc. 7th Internat. Congr. Phot., 1928, 14—34).—Existing literature dealing with the photo-conductivity effects exhibited by silver halides and similar substances is reviewed. The similarity of the position of maximum photo-conductivity effects shown by the same halides of different metals seems to suggest that the anions are the light-sensitive ions. The results obtained by Coblenz with silver bromide layers, which showed a maximum effect approximately at λ 4600 with very low effects in the violet and ultra-violet regions, are shown to

be due to the thick silver bromide layer used, which favoured a much stronger absorption of blue light than of light of shorter wave-length. A series of experiments, using layers the thickness of which varied from about 660μ to 20μ , showed that as the thickness decreased so the effects in the violet and ultra-violet regions increased relatively to those in the blue, until the maximum in the blue finally disappeared. At the same time an increase in the magnitude of the effects was observed. With such very thin layers the three curves representing (i) the relative photo-conductivity effects, (ii) the relative photographic effects as measured by a determination of the number of centres formed by exposure of single-layer plates of a silver bromide emulsion, and (iii) the relative light absorption, each plotted against the wave-length for equal incident energy, are very nearly the same. These facts indicate that in all probability the primary stage of the photographic mechanism is intimately connected with that producing conductivity changes on illumination, consisting in the liberation of an electron from the anion of the silver halide crystal and the formation of neutral silver and free halogen. J. W. GLASSETT.

Principles and practice of [photographic] fixation. F. F. RENWICK (Phot. J., 1929, 69, 310—314).—The theory of fixation and the factors influencing the rate of fixation in sodium thiosulphate solutions are outlined. J. W. GLASSETT.

PATENTS.

Photographic films. MIMOSA A.-G., and W. NAEWIGER (B.P. 316,811, 29.9.28).—Material suitable for the production of positives, which may be viewed both by transmitted and reflected light, is prepared by coating both sides of a semi-opaque film base, cast, for example, from a mixture of cellulose nitrate and tinted barium sulphate, with sensitive emulsion, the rear coating being the more sensitive. Chloro-bromide and bromide emulsions may be used for the front and rear sides, respectively. The image produced on the rear side gives the increased density required when the print is used as a transparency. J. W. GLASSETT.

Light-sensitive films or carriers therefor. Subtractive colour photography. (A) W. T. L. BECKER and L. W. OLIVER; (B) W. T. L. BECKER, L. W. OLIVER, H. D. MURRAY, and COLOUR PHOTOGRAPHS (BRITISH & FOREIGN), LTD. (B.P. 317,909 and 317,911, [A] 26.5.28, [B] 26.5.28 and 18.1.29).—(A) To facilitate registration in the preparation of composite prints on cellophane or similar materials, the dried sheets are brought to a known state of expansion before use by either of the following methods. (i) The materials immersed in the different sensitising baths are brought to the same known state of expansion and are then mounted on a stiff support, such as celluloid, and dried. (ii) The solid contents of the different baths are adjusted by the addition of experimentally determined amounts of a neutral substance, e.g., sugar, such that each bath will then cause the same swelling and contraction of the material during immersion and drying. (B) Water-permeable, homogeneous, transparent substances which retain their mechanical properties while wet, as, e.g., cello-

phane or specially treated cellulose derivatives, are used in the form of films as the support and medium for the light-sensitive substances, which are introduced into the body of the film by absorption from aqueous solutions of the sensitisers. J. W. GLASSETT.

Providing photographic raw film with visible reproducible inscriptions. I. G. FARBENIND. A.-G. (B.P. 295,383, 27.7.28. Ger., 12.8.27. Addn. to B.P. 287,124; B., 1928, 914).—A colloid, preferably a cellulose derivative, which is insoluble in any reagent used in the subsequent treatment of the film, is admixed with the dye solution or pigment used for the printing. For printing on nitrocellulose film, a 2:1 mixture of cold saturated solution of cerasine-red in glycol acetate and a 5% solution of acetylcellulose in the same solvent may be used. J. W. GLASSETT.

Photographic and like screens. T. C. ERWIN (B.P. 315,681, 13.4.28).—Reticulated gelatin screens are prepared by coating a transparent support (glass, celluloid) with a gelatin solution hardened, to render it insoluble in hot water, by treatment, either before or after coating, with chrome alum, formalin, or similar tanning agents. After drying at room temperature the film is treated with a warm acid bath; preferably a bath consisting of 1 c.c. of hydrochloric acid (*d* 1.27) in 1150 c.c. of water is used at 57–60°. J. W. GLASSETT.

Producing even coatings on transparent plates, films, etc. to serve as colour screens and for other photographic purposes. F. H. HAUSLEITER (B.P. 316,719, 23.5.28).—The base is coated with a layer of a colloid mixture, such as dichromated gelatin, which is rendered insoluble by the action of light, given a uniform exposure through the rear side of the base, and treated to remove the soluble portion. The even coatings so formed are free from surface skin formation, but this may be produced where necessary by treatment with a tanning vapour, such as bromine, or by the precipitation of a film of lead or copper ferrocyanide on the surface. J. W. GLASSETT.

Production of screen negatives for producing photomechanical printing surfaces. H. A. GILL. From AMIRA TRUST (B.P. 316,364, 14.5.28).—A silver halide emulsion on a transparent support is exposed through the rear side behind a screen, and is developed in a hardening developer or treated after development by any known method for hardening the gelatin in proportion to the silver deposit. Without fixation, the negative is washed in hot water and the silver dot image intensified or reduced by the usual methods. High dot formation may be prevented by lowering the penetration of the light by the addition of a suitable dye to the emulsions or by destruction of the surface latent image by means of copper chloride, bromine–iodine tincture, etc. J. W. GLASSETT.

Colour photography. R. RUTH (B.P. 317,712, 20.2.28).—A multi-coloured gelatin screen, supplied with a detachable transparent support, is coated with sensitive emulsion. The exposure is made through the support and the screen is hardened at those parts in contact with the light-affected silver halide by the use of a

hardening developer such as pyrocatechol. The image is reversed, using a non-hardening developer, transferred together with the screen to a suitable base, and the original support is then stripped, the unhardened parts of the screen being removed by hot water.

J. W. GLASSETT.

Development and fixing of photographs. ETABL. E. BELIN (B.P. 299,466, 25.10.28. Fr., 28.10.27).—The developing and fixing solutions and the wash-water are sprayed in turn through atomising jets on to the surface of the sensitive material. The solutions may be used at 50–80° for rapid work, and although this temperature is considerably lowered by the cold emulsion surface, the working temperature obtainable is much higher than that possible by the ordinary bathing methods. J. W. GLASSETT.

Manufacture of photographic prints. LUXOR-FILM GES.M.B.H. (B.P. 289,858, 3.5.28. Ger., 4.5.27).—A gelatin film, which has been sensitised by bathing first in a strong dichromate solution (up to 10%) followed while still wet by a short treatment in a weak solution (up to 2%), is exposed behind the negative and then left in a warm atmosphere supersaturated with water vapour and containing a small quantity of volatile organic acid (acetic acid) or carbon dioxide. During this treatment the plate is given a uniform general exposure and later removed, washed with cold water, and dyed. J. W. GLASSETT.

N-Hydroxyethyl derivatives of [p]-aminophenols [photographic developers]. G. REDDELEIN and W. MUELLER, AssTs. to AGFA ANSCO CORP. (U.S.P. 1,712,716, 14.5.29. Appl., 2.11.27. Ger., 19.11.26).—See B.P. 280,873; B., 1929, 38.

[Registering devices for] production of [multi-colour] kinematograph film positives. J. E. THORNTON (B.P. 316,338—9, 25.4.28).

[Registering devices for] manufacture of two-colour kinematograph positive films. J. E. THORNTON (B.P. 316,367, 18.5.28).

XXII.—EXPLOSIVES; MATCHES.

Explosive properties of "Chloratit 3" containing various amounts of petroleum. A. HAID and H. SELLE (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 251–252).—"Chloratit 3" with 8–12% of petroleum is the most sensitive in the fall-hammer test. When "Chloratit 3" contains 3% of woodmeal and 1–2% of petroleum, it is insensitive to friction in the porcelain mortar test, a content of 3–5% of petroleum causes it to crackle, whilst 7–10% causes an explosion. Sensitiveness to initiation is greatest when the petroleum content is 3–5%. The values in the lead-block test increase linearly with the petroleum content until the latter reaches 10%, and then decrease. The maximum detonation velocity (3500 m./sec.) occurs with a content of 7% of petroleum. On storage, a cartridge of "Chloratit 3" with 3% of woodmeal and 9% of petroleum loses 1% in weight at 20° in 5 months.

S. BINNING.

Testing the chemical stability of smokeless powders and explosives by measuring their hydrogen-ion concentrations. L. METZ (*Z. ges. Schiess- u. Sprengstoffw.*, 1929, 24, 245—250, 291—296, 335—340).—Hansen has proposed to test the stability of smokeless powders by measuring the change in their p_H when they are heated at 110° for 8—10 hrs. His data were checked by determinations on various types of nitrocelluloses and nitrocellulose powders, and his method was then extended to nitroglycerin powders, and also to explosives such as trinitrotoluene, tetryl, nitroglycerin, dinitroglycol, and gelatine-dynamite. The test was found to have decided advantages in giving in a few hours results that were in agreement with the present tests, which require many hours for performance. The relatively low temperature at which the test is carried out is also in its favour. The apparatus is easy to work, even by relatively unskilled users.

S. BINNING.

Influence of gel structure on the technology of smokeless powder manufacture. F. OLSEN (*Coll. Symp. Mon.*, 1928, 6, 253—264).

XXIII.—SANITATION; WATER PURIFICATION.

Determination of hydrogen cyanide in fumigation experiments. C. O. EDDY and E. N. GEDDINGS (*J. Econ. Entomol.*, 1929, 22, 366—378).—Samples of the air are passed through 3.5% sodium hydroxide solution and the cyanide is determined turbidimetrically with silver iodide.

CHEMICAL ABSTRACTS.

Equipment for the treatment of feed water for modern steam boilers. J. D. YODER (*Ind. Eng. Chem.*, 1929, 21, 829—834).—Descriptions are given of hot-process lime-soda softeners, sodium phosphate feeding equipment, sulphuric acid feeding equipment in connexion with zeolite softeners to maintain the sulphate ratio, and continuous blow-off apparatus.

C. IRWIN.

Examples and precepts of water conditioning. R. E. HALL (*Ind. Eng. Chem.*, 1929, 21, 824—829).—A number of analyses of boiler-feed waters are given and softening methods used are described. For boiler pressure over 200 lb. "direct" treatment with phosphate with or without a partial preliminary lime-soda treatment is preferred. In one case where water polluted with organic matter was used ammonia was eliminated from the steam by treatment of the water with chlorine. Analyses of boiler scales include examples where the chief component was (a) calcium hydroxide, (b) sodium sulphate. If alkalinity falls too low, phosphate in solution ceases to be effective in preventing the formation of calcium sulphate scale. Some superheater deposits contained sulphides. Experiments with unsaturated asphaltic hydrocarbons as anti-foaming material, whilst successful in some cases, did not indicate that its use is generally desirable.

C. IRWIN.

Zeolite-deconcentrator combination for boiler-water purification. E. W. SCARRITT (*Ind. Eng. Chem.*, 1929, 21, 821—823).—In the case reported the untreated water contained 320 p.p.m. of carbonate hardness and 45 p.p.m. of permanent hardness. After 14 days,

using zeolite-softened water alone for make-up, the alkalinity had reached 2050 p.p.m. This necessitated excessive blowing down, and the low ratio of sulphate to total alkalinity involved danger of embrittlement. The make-up was therefore altered to 4 pts. of hard water with 1 pt. of softened water, and a "deconcentrator" or settling tank outside the boiler and connected with it for the removal of sludge was fitted. With this arrangement the alkali formed was sufficient to remove the permanent hardness, no scaling took place, the blow-down was reduced from 6.9% to 1.46%, and the alkali content and sulphate:alkali ratio in the boiler water remained satisfactory.

C. IRWIN.

Purification of tannery effluents with argillaceous colloids. J. BESSE (*J. Soc. Leather Trades' Chem.*, 1929, 13, 503—507).—Brick clay is agitated with a dilute solution of sodium carbonate (0.2 g./litre) to disperse it, the heavier particles and sand are allowed to settle out, and the milky liquor is decanted. The tannery effluent is acidified with sulphuric acid, mixed with four times its volume of the colloidal solution of clay, and the mixture is agitated; the clay thereby flocculates and carries down the impurities. The dry residue in the supernatant liquor is considerably less than that in the untreated effluent, and the treated liquor does not froth on shaking.

D. WOODROFFE.

Antiseptic value of chlorine. HOWARD and others.—See XV.

PATENTS.

Liquid preparation for sanitary purposes, eliminating the smell, and decomposition of offensive matter. S. S. PALEY (*B.P.* 319,113, 24.8.28).—A liquid containing chromic acid (1 pt.), sulphuric acid, *d* 1.84 (1 pt.), water (16 pts.), and as much zinc as will dissolve therein, is claimed.

C. JEPSON.

Obtaining soaps and oils from sewage. E. VON SPRINGBORN (*B.P.* 318,849, 6.6.28).—The vapours produced by heating sewage sludge either alone or in conjunction with combustible filtering material, *e.g.*, coal, coke, peat (*cf.* *B.P.* 307,582; *B.*, 1929, 418), are deodorised by potassium permanganate, chloride of lime, or charcoal; after passing through a suitable alkali for the production of soaps, the remaining oils are fractionally cooled to separate them into various grades.

C. JEPSON.

Softening of water and removing iron and manganese therefrom by base-interchange, and preparing the requisite agent therefor. A. ROSENHEIM (*B.P.* 286,307, 2.3.28. *Ger.*, 4.3.27).—The water is brought into contact with a metal oxide gel, silica gel, or mixtures thereof, into which have been incorporated alkali oxides, alkali metals, or ammonium ions which enable base-interchange to take place. The incorporation may be accomplished by treatment of the gels with aqueous solutions of (i) caustic alkalis and ammonia, (ii) alkaline reacting alkali-metal or ammonium compounds, (iii) basic reacting substances the cations of which are not those of an alkali metal or ammonium, with subsequent treatment by a solution of an alkali compound, *e.g.*, common salt.

C. JEPSON.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 6, 1929.

L—GENERAL; PLANT; MACHINERY.

Determination of gas temperatures by measurement of pressure differences. H. SCHMICK (Z. tech. Physik, 1929, 10, 146—147; Chem. Zentr., 1929, i, 2800).—The method is based on the fact that when a gas is passed through a valve at a certain speed the pressure difference is approximately proportional to the absolute temperature. A. A. ELDRIDGE.

Measurement of absolute viscosity by the use of concentric cylinders. H. R. LILLIE (J. Amer. Ceram. Soc., 1929, 12, 505—515).—A stationary cylindrical spindle is suspended by a torsion member in the centre of a cylinder containing the liquid under test, the cylinder is rotated at a measured rate, and the torque thus exerted on the spindle is measured by means of a mirror, a telescope, and a straight scale. With castor oil as a test fluid, spindles of different lengths but of the same radius were used to give values for apparent viscosities. These values were plotted against reciprocals of the length of the spindles, and the "true" viscosity of the oil was found by averaging the extrapolated values of a number of such curves. By this method the viscosity was found to be 9.67 poises at 20°. Using this value, a correction, to allow for the torque on the ends of the spindle, was determined. For a spindle such as is used in measuring the viscosity of glasses this factor was 0.44 cm. The effect of variations of temperature of the spindle and the fluid was also studied. F. SALT.

New laboratory materials. H. A. J. PIETERS and J. A. MEYLINK (Chem. Weekblad, 1929, 26, 495—497).—Nickel and special steel crucibles increase in weight irregularly when heated at 700—1000°, but nickel crucibles may be used for smelting minerals with potassium hydroxide, though not with sodium hydroxide. Small nickel crucibles are suitable for determination of the gas content of coal. Among refractories, "Weta" material and alundum are suitable for igniting precipitates, determining ash in coal, etc.; the former must first be ignited till constant in weight. S. I. LEVY.

Optical measurement of [furnace] temperature. G. AECKERLEIN (Z. tech. Physik, 1929, 10, 129—136; Chem. Zentr., 1929, i, 2799).

See also A., Nov., 1233, **Velocity of elutriation and particle size** (ANDREASEN and LUNDBERG). 1234, **Ultracentrifuging** (LAMM). 1261, **Removal of dissolved gases from liquids** (HIBBEN).

PATENTS.

Furnaces or plant for the heating and heat treatment of metals or other materials or goods.

J. SUMMERS & SONS, LTD., and C. BOTTOMLEY (B.P. 319,300, 20.6.23).—Chambers for goods to be heated (*e.g.*, bars to be rolled into plates) are situated on each side of a fire chamber, and behind these are larger chambers (containing, *e.g.*, plates to be rolled into sheets) at a lower temperature. Means for controlling the distribution of heat and admission of secondary air are described. B. M. VENABLES.

Shaft furnaces and reaction chambers for treating solid materials with gases. A./S. NORSK STAAL (ELEKTRISK-GAS-REDUKTION), and E. EDWIN (B.P. 295,999, 18.8.28. Norw., 22.8.27).—The furnace consists of a stack of interchangeable sections each having a perforated bottom to support the material under treatment; the detachable sections are terminated by top and bottom sections for inlet and exhaust of gases. These end sections are not removable, but have sufficient vertical movement (alone or with the whole stack) to permit insertion and withdrawal of the detachable sections one at a time; the latter work downwards (or upwards) through the whole height of the tower. Other similar towers for preheating and cooling may be arranged adjacent the reaction tower.

B. M. VENABLES.

Heat interchanger. F. C. REED and F. A. ERNST (U.S.P. 1,730,293, 1.10.29. Appl., 1.8.27).—An inner cylindrical casing is clamped between two headers of larger diameter, the internal space being idle. This unit is inserted into a larger cylindrical container having a fixed flange on the lower end to afford support for the unit and inlet and outlet connexions for the outer fluid. Spiral tube coils extending from header to header in parallel are situated in the annular space and convey the inner fluid.

B. M. VENABLES.

Heat-exchanging apparatus. J. M. HARRISON (U.S.P. 1,730,139, 1.10.29. Appl., 16.5.28).—A number of elements, each comprising a pair of side plates secured together along all four edges, are assembled with distance pieces between the elements. Inlet and outlet connexions are formed from transverse conduits welded to the edges of the units, with suitable openings into the interiors of the units.

B. M. VENABLES.

Boilers for evaporating or heating liquids. SPENCER-HOPWOOD, LTD., and A. L. HITCHCOCK-SPENCER (B.P. 319,380, 23.3 and 7.12.28).—In a vertical boiler of circular section provided with cross-tubes, the segmental dead spaces at each side of the bundle of tubes are filled by hollow members which may be either of solid refractory material or of metal; the latter may serve as heaters for air or vapour to be used internally or externally for any useful purpose. B. M. VENABLES.

Drying of steam or vapours under pressure. A. VALDER (B.P. 319,567, 30.1.29).—A priming trap for use inside steam boilers is described. B. M. VENABLES.

Carrying out chemical reactions. H. T. BUCHERER (B.P. 296,376, 30.8.28. Ger., 30.8.27).—A gaseous substance is caused to pass at high velocity at right angles to an orifice which delivers a liquid substance; the speed of the gas current produces a reduction of pressure which assists in promoting the flow of liquid and causes the latter to be finely sprayed. Both conduits are calibrated to maintain the stoichiometric relation according to the pressures used. Inert gases may be added to promote desired effects such as evaporation, and the apparatus may be used for purely mechanical purposes such as spray-painting.

B. M. VENABLES.

Extinguishing fire. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 319,083, 27.6.28).—Burning material, especially that in a finely-divided state, *e.g.*, coal dust, straw, cotton, is sprayed with a dilute aqueous solution of a wetting agent, *e.g.*, soap, saponin, Turkey-red oil, a sulphuric ester of or a sulphonated organic compound. Alternatively, the wetting agent is sprayed on to inflammable material liable to ignite and water is subsequently played on to it in case of ignition.

L. A. COLES.

Disintegrating machines. MAGNET-WERK G.M.B.H. EISENACH SPEZIALFABR. F. ELEKTROMAGNET-APPARATE (B.P. 308,347, 1.3.29. Ger., 22.3.28).—The preliminary disintegration is effected in an annular hopper-like space provided with fixed and rotating knives, but the final disintegration takes place in a zone that is not very narrow, the necessary retardation of the downward flow of material being brought about by a change of direction in the passage, the fine-grinding zone being bounded above by a renewable ring of fixed knives or serrations and below by a flat cone attached to the disintegrator shaft. The width of the fine-grinding passage remains about the same throughout.

B. M. VENABLES.

Apparatus for separating minerals or other materials. R. E. TROTTER (U.S.P. 1,729,913, 1.10.29. Appl., 2.5.25. Fr., 12.5.24).—A conical drum with horizontal axis is provided on the interior with a continuous helix which decreases in depth and pitch towards the narrower end. The drum is rotated in such a direction that the helix tends to work the heavier material up the slope, whilst water delivered to the interior of the drum washes the lighter material downwards.

B. M. VENABLES.

Separation of materials of different sp. gr. H. M. CHANCE (U.S.P. 1,730,123 and 1,730,189, 1.10.29. Appl., [A] 5.12.25, [B] 15.4.29).—(A) A mass of materials is passed through a reciprocating conduit, and a positive longitudinal movement is given to the material by means of a slope and by forming the bed in steps; stratification is thereby produced, the upper layer consisting of the larger lighter particles. A high-velocity gas current is provided in the same direction as the travel of material. In (B) a particular form of (A) is described, the bottom of the conduit being formed of trays in step-like formation with spaces between.

B. M. VENABLES.

Obtaining granular solids [from viscous liquids]. A. J. COLLIER, F. HEYWOOD, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 319,893, 27.8.28).—Droplets of a viscous liquid, such as solutions of calcium nitrate or mixtures of concentrated ammonium nitrate and calcium carbonate, are picked up by the spikes of a rotary, brush-like member and flicked off into a chamber in which they are solidified by contact with a gas before they reach the walls or bottom of the chamber.

B. M. VENABLES.

Apparatus for uniformly evaporating liquids, especially for uniformly charging gases with vapours. R. MEZGER (B.P. 305,089, 12.10.28. Ger., 30.1.28).—The apparatus for purposes such as delivering xylene into a gas main comprises an electrically-heated vaporising chamber with warning thermometer and thermo-elements to cut off the current in the event of overheating. The vaporising chamber being well lagged, after the initial heating, the amount of liquid evaporated per unit of time will be proportional to the watts of electricity supplied. The level of the liquid in the vaporiser is kept constant by a separate bird-fountain device. Should this leak, an emergency overflow (with tank) is provided to prevent flooding of the vaporiser.

B. M. VENABLES.

Electrically heated apparatus for evaporating, distilling, etc. J. A. REAVELL (B.P. 319,849, 6.7.28).—The vessel is surrounded by more than one concentric jacket containing oil or other suitable fluid, which is heated electrically and circulated by an internal pump or propeller.

B. M. VENABLES.

Device for interconnecting the evaporation elements of evaporation plants. B. BOULOGNE (B.P. 292,915, 18.6.28. Holl., 25.6.27).—The liquor spaces of a number of evaporating elements are connected by U-shaped conduits. A similar U-shaped conduit connects at both ends with the later of the evaporators concerned, and the rising branch is common to both U-shaped conduits. Modifications, some including non-return valves, are described. The object is mainly to prevent, by cooling, too sudden ebullition of incoming weak liquor.

B. M. VENABLES.

Distillation. E. H. LESLIE (U.S.P. 1,730,892, 8.10.29. Appl., 19.4.23).—Liquid to be distilled is vaporised and the vapours are led through a fractionator to a reflux cooler in which heat control is effected by a liquid cooling medium that boils under the conditions of the operation, thereby enabling the return of an exactly predetermined weight of the partial condensate to the fractionating system.

H. S. GARLICK.

Acid-concentrating and like towers. P. H. EVANS and R. C. BOWDEN (B.P. 319,839, 3.7.28).—The baffles, shelves, or partitions are constructed from acid- and heat-resisting bricks in such a manner to be individually supported against collapse, one form of construction consisting of alternate arches and pairs of half-arches. A cement consisting of glass, sodium silicate, and pumice powder may be used.

B. M. VENABLES.

Apparatus for [comparing the rate of] separation of particles suspended in a liquid. K. T. R. LUNDGREN (B.P. 319,629, 19.6.28).—The samples, *e.g.*,

blood for medicinal tests, are contained in a number of tubes which are supported in a framework so that they are in one vertical plane but may be all inclined equally to the horizontal.

B. M. VENABLES.

Gas mixer. L. J. O'NEIL and L. B. BECKER (U.S.P. 1,730,288, 1.10.29. Appl., 26.3.28).—A device for insertion in a pipe-line comprises a spiral baffle (twisted flat strip) supported at its ends by discs of perforated material of different mesh; the discs are forced into grooves formed on the interior of a tube, which is provided with an atomising nozzle at one end.

B. M. VENABLES.

Gas analyser. W. KEMP (U.S.P. 1,727,544, 10.9.29. Appl., 15.9.28).—A sample of the gas is supplied under a steady head by means of a water-operated injector terminating in a water-seal which permits escape of excess gas. The gas is led to one side of a diffusion plate, on the other side of which is an absorbent (*e.g.*, caustic potash for flue gases) for one constituent. The pressure in the absorption chamber is less than that of the gas supplied, by an amount depending on the proportion of absorbable constituent, and is measured by a known type of gauge.

B. M. VENABLES.

Viscosimeter. I. G. FARBERIND. A.-G. (B.P. 303,853, 28.12.28. Ger., 11.1.28).—A viscosimeter of the type in which a body is rotated in the liquid is constructed so that the pull of the thread by which the rotating force is applied comes directly opposite one of the bearings of the rotating body, that bearing being of the needle-point and cup type. Any parts subjected to friction may be constructed of stainless steel.

B. M. VENABLES.

Heating and/or chemical treatment of liquids and molten materials by direct contact with combustion products. S. C. SMITH (U.S.P. 1,730,440, 8.10.29. Appl., 4.5.25. U.K., 12.5.24).—See B.P. 242,680; B., 1926, 32.

Apparatus for evaporation of liquids. E. MORTERUD (U.S.P. 1,731,146, 8.10.29. Appl., 13.12.26. Norw., 19.12.25).—See B.P. 263,132; B., 1927, 800.

[Centrifugal] apparatus for precipitation of particles suspended in liquids. K. T. R. LUNDGREN (U.S.P. 1,730,776, 8.10.29. Appl., 16.12.27. Swed., 22.12.26).—See B.P. 282,681; B., 1928, 507.

Furnaces with mechanical stokers. M. RATEL (B.P. 293,468, 6.7.28. Fr., 8.7.27).

Machines for grinding ores, minerals, stones, etc. J. R. BROADLEY (B.P. 319,897, 29.8.28).

Rectification of gas and/or vapour mixtures in absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 298,574, 11.10.28. Ger., 11.10.27).

II.—FUEL; GAS; TAR; MINERAL OILS.

Chemical composition of peat. V. Rôle of micro-organisms in peat formation and decomposition. S. A. WAKSMAN and K. R. STEVENS (Soil Sci., 1929, 28, 315—340; cf. B., 1929, 545).—Micro-organisms are most important factors in the production of peat from plant residues. The nature and numbers of organisms

occurring at various depths in different peat bogs were determined. In low-moor peats aerobic bacteria, fungi, nitrifying and cellulose-decomposing bacteria occur mainly in surface layers, being steadily displaced by anaerobic organisms as the depth from the surface increases. Acid sphagnum peat has an abundant bacterial flora capable of growing in media of p_H 4.0. The decomposition of peat is slower than that of fresh plant residues, and the ratio of carbon, eliminated as carbon dioxide, to nitrogen, liberated as ammonia and nitrate, increases with the depth from the surface. The decomposition of peat is accelerated by the removal of waxes by suitable solvents.

A. G. POLLARD.

X-Ray study of vitrain and durain and of their constituents. C. MAHADEVAN (Indian J. Physics, 1929, 4, 79—98).—Vitrain shows the two most prominent graphite-carbon rings when examined by the X-ray diffraction method. Durain behaves as a colloid with vitrain as the dispersion medium and ash and vegetable detritus as the disperse phases. The α - and β -compounds of vitrain appear to be identical and similar to untreated vitrain. The pyridine extract, the benzene-soluble and the benzene-insoluble fractions gave similar patterns for vitrain and durain, respectively.

W. E. DOWNEY.

Moisture content of petrographic varieties of Polish coal. J. PFANHAUSER (Przemysl Chem., 1929, 13, 449—455).—The highest values for moisture content of coal are given by Schläpfer's xylene distillation method. Drying at 105° in nitrogen gives somewhat lower results, whilst in the presence of oxygen the values obtained are lower still. In the former case the drying curve has a halt after 30—45 min., after which it again ascends, the apparent moisture content eventually exceeding that obtained by Schläpfer's method; this is due to loss of volatile constituents. In the presence of air the drying curve similarly reaches a maximum after 30—45 min., but thereafter the apparent moisture content declines in the cases of vitrain and durain, as a result of absorption of oxygen; in the case of fusain the apparent moisture content attained after 30 min. thereafter varies very little. The most reliable method of determining the moisture content is that of Schläpfer; if ordinary drying methods are used, the coal should be kept at 105° for 45—60 min. in a stream of nitrogen, or for 30—45 min. in air.

R. TRUSZKOWSKI.

Determination of water in lignite with calcium carbide. R. VON WALTHER and G. BENTHIN (Braunkohlenarch., 1929, No. 23, 110—122; Chem. Zentr., 1929, i, 2260).—Piatscheck's method is modified in detail to give more concordant results.

A. A. ELDRIDGE.

Classification of North American coals. A. C. FIELDNER (Fuel, 1929, 8, 473—481; cf. B., 1929, 155).—The first year's work of the three technical committees organised by the American Society for Testing Materials for dealing with the standardisation of coal classification is summarised. The committees deal respectively with (a) scientific classification, (b) use classification, and (c) marketing practice. Their work up to the present has consisted in the collection and correlation of data concerning existing systems of classification.

A. B. MANNING.

Errors due to the substitution of "ash" for "mineral constituents" in coal analyses. K. MAYER (Brennstoff-Chem., 1929, 10, 377—382).—For coals of low ash content the calorific values as calculated from the ultimate analyses by Vondracek's formula (B., 1927, 179) agree closely with the experimental values. The deviations exhibited by coals of greater ash content are due to the assumption that the "ash" may be taken as equivalent to the "mineral constituents" when calculating the "pure coal" content. A simple procedure for determining more accurately the mineral constituents of coals, except those containing hydro-silicates, consists in extracting the coal with hydrochloric acid, ashing the extracted coal at a dull red heat, and determining the ferric oxide in the ash. The mineral constituents are then given by the sum of the acid-soluble constituents, the pyrites, calculated from the ferric oxide, and the remainder of the ash.

A. B. MANNING.

Tests of bituminous caking coal in a large low-pressure heating boiler. P. NICHOLLS, C. E. AUGUSTINE, and B. A. LANDRY (Fuel, 1929, 8, 482—486).—The design of the boiler used in earlier tests (cf. Blizard and Houghton, Bur. Mines Tech. Paper 303, 1922) has been modified by the provision of tuyères for admitting secondary air at the rear of the bridge wall. The boiler and furnace efficiency averaged only 1% higher in tests with the new arrangement than when the secondary air was admitted through slots in the fire doors. Less carbon monoxide was present in the flue gases in the tests with tuyères, and less smoke was measured by the Ringelmann chart, but the smoke was not materially reduced. Owing to the increased resistance to the flow of gases through the boiler with tuyères for supplying secondary air, about 15% additional draught was required to obtain the same steam output as before.

A. B. MANNING.

Various methods of calculating the calorific value of solid fuels. P. JARRIER (Chaleur et Ind., 1929, 107; Fuel, 1929, 8, 486—491).—The methods that have been used for calculating the calorific value of a solid fuel from (a) the ultimate analysis, (b) the proximate analysis, and (c) the oxygen used for combustion are summarised and compared. Whilst none of the formulæ is strictly satisfactory, some of them permit the evaluation of the calorific value with an accuracy that is often sufficient.

A. B. MANNING.

Phases of the organic sulphur problem in the manufacture and utilisation of gas. W. J. HUFF (Proc. 2nd Int. Conf. Bit. Coal, 1928, 2, 814—825).—The amount of carbon disulphide produced on distillation of coal is larger when the coal is heated rapidly. Carbon-sulphur complexes may be important intermediates in the production of sulphur compounds found in gas; their elimination as hydrogen sulphide is desirable.

CHEMICAL ABSTRACTS.

Low-temperature carbonisation of a coal in the presence and absence of tetrahydronaphthalene. D. J. W. KREULEN (Brennstoff-Chem., 1929, 10, 382—383).—A 20-g. sample of coal was saturated with tetrahydronaphthalene, heated at 175—200° until the bitumen had passed into solution, and the whole then car-

bonised in the Fischer aluminium assay apparatus. The yields of coke, tar, liquor, and gas, and the quality of the coke, were the same as obtained by carbonising the coal directly.

A. B. MANNING.

"Water-gas" equilibrium in higher-limit methane-oxygen-nitrogen flames. H. F. COWARD (Fuel, 1929, 8, 470—471).—An analysis of the results of Burgess and Wheeler (B., 1914, 1194), giving the composition of the gaseous mixture "snatched" from the centre of "higher-limit" flames of methane, oxygen, and nitrogen, shows that the water-gas equilibrium was attained either in the flame or closely behind it.

A. B. MANNING.

Determination of phenols in effluent from the carbonisation of lignite, and their extraction. P. ROSIN and H. JUST (Z. angew. Chem., 1929, 42, 965—968, 984—987, 1002—1007).—Ulrich and Kather's bromination method of determining phenols (B., 1926, 306) when applied to lignite effluents must be performed with 1.6 pts. of 20% quinoline-benzene mixture, the treatment must last 2 hrs., and the liquor sample must be fresh. The bromine number so obtained, however, does not permit the phenol content to be calculated accurately, as dibasic phenols are present and their bromination proceeds irregularly, especially in the case of quinol. The error involved is not, however, great. Much lower results are obtained by extraction of phenols with ether, distillation of the latter, and bromination. Apparently the extraction is incomplete. The authors' gravimetric method is to extract with quinoline and benzene, treat with alkali, acidify the phenoxide solution, and then extract with ether. The crude phenols (containing fatty acids) are dissolved in alkali and brominated to determine the percentage of pure phenols. For extraction on an industrial scale quinoline is too expensive, but tests show that benzene alone has only inferior extracting power. The most satisfactory medium for large-scale work is a 20% aniline-benzene mixture. The determinations carried out in this connexion were performed by Ulrich and Kather's method, blank tests being run to correct for the bromination of aniline etc. By working on the countercurrent principle, 1 pt. of aniline + benzene will extract 75% of the phenols from 4 pts. of liquor. It is impossible to separate the extracted phenols by distillation owing to the presence of aniline, but alkali treatment is suitable. The entrained solvent must be recovered from the effluent by distillation. Lignite carbonisation effluents usually contain too little ammonia for its recovery by distillation to be economical, but this could be attained, and one distillation might serve both purposes, if the lignite were thoroughly dried before carbonisation.

C. IRWIN.

Formation of liquid hydrocarbons from acetylene. II. Catalytic hydrogenation and condensation of acetylene. F. FISCHER, K. PETERS, and H. KOCH (Brennstoff-Chem., 1929, 10, 383—385; cf. B., 1929, 703).—By passing a mixture of acetylene and hydrogen over an iron-copper-alkali catalyst at 250°, 40—70% of the acetylene has been converted into oil. The catalyst was not readily poisoned, and a high degree of purity in the gas mixture was unnecessary. If the copper:iron ratio exceeded 1:10 the tube became

stopped up by a deposit of cuprene. The addition of nickel appeared to diminish the production of cuprene. An iron-nickel catalyst (10 Fe:1 Ni) containing no copper brought about a 60% conversion of acetylene into oil. With iron alone it was necessary to raise the reaction temperature to 300–340°, and the oil production was then accompanied by carbon deposition. Considerable fluctuations in the temperature and rate of passage of the gas were without effect on the yield and quality of the oils produced. The product consisted of a heavy oil which collected in the receiver and a light oil, of boiling range approx. 50–150°, which was absorbed by activated charcoal. The heavy oil contained no paraffin wax. From 1 cub. m. of coke-oven gas, which was first subjected to an electric discharge and then to the above process, 85 g. of total oil were produced, of which 75% was light oil.

A. B. MANNING.

Testing of bitumen. H. BÖSENBERG (Petroleum, 1929, 25, 1275–1279).—The determination of the dropping point, solidification point, and stability value of a bitumen is described in detail, together with diagrams of suitable apparatus. The physical properties of pitches of the Central Institution of Asphalt and Tar Research and of the American Asphalt Association are given, also of mixtures of these. It has been found possible, by mixing given grades of bitumens, to obtain any desired degree of penetration value and m.p., numerical data for several mixtures of which are appended.

W. S. E. CLARKE.

Cracked residues and cracked oil distillates. A. SACHANEN and M. BESTUSCHEV (Petroleum, 1929, 25, 307–309).—The sp. gr., viscosity, and flash point of cracked oil residues are recorded. Balachany mazout having d_{40}^{15} 0.908, viscosity (E_{50}) 6.17, setting point –20°, flash point (open) 161°, was cracked at 425° under 15 atm. pressure for 32 min., 1 hr. 32 min., 3 hrs. 2 min., and 6 hrs. 2 min., respectively. The cracked residue, viz., that remaining after removing benzene and kerosene fractions to 300°, was distilled at 6–15 mm. It is shown that the difference between the cracked oil distillates and oil distillates of the original substance increases with longer cracking. Determinations of mol. wt. and elementary analyses show that the hydrocarbons present are chiefly aromatic and hydro-aromatic, and that cracking brings about dehydrogenation and decomposition.

W. S. E. CLARKE.

Analysis of motor fuels. O. SMETANA (Petroleum, 1929, 25, 1061–1067).—The b.p. curve of a liquid fuel reveals, in some cases, its method of preparation, but does not measure its volatility; the latter can only be accurately determined by passing an inert gas over the liquid. A shorter method is described in which a filter paper is soaked with the fuel and the rate at which it loses weight is measured, under conditions which may be varied to suit the type of liquid. The action of bromine is considered to give more information as to the tendency of a fuel to polymerise than treatment with sulphuric acid, but the test must be carried out in the dark, and with only slight excess of halogen. The mutual solubility of aniline, benzene, and non-aromatic light petroleum has been examined, and the results are shown in a diagram. The volume change which occurs

on shaking a fuel with aniline can be used to determine its aromatic content. A special vessel is employed, consisting of two bulbs connected by a graduated neck; 48.3 c.c. of aniline and 25 c.c. of the fuel are shaken and warmed slightly, and then allowed to cool to 20°. The contraction is directly related to the aromatic content in a way which is shown graphically, and varies slightly with the b.p. of the liquid. Better results are obtained if the aniline be previously saturated with non-aromatic hydrocarbons of the same type. Practical recommendations for the application of the method to various cases are made, and the accuracy of measurement is considered.

R. H. GRIFFITH.

“Formolite” analysis of mineral oil. J. HOŠEK (Chem. Obzor, 1929, 4, 67–70; Chem. Zentr., 1929, i, 2606).

See also A., Nov., 1237, **Equilibrium $\text{CO}_2 + \text{C} \rightleftharpoons 2\text{CO}$** (DENT and COBB). 1275, **Solubility of humic acids in phenol** (SOUK and PODBREZNIK). **Humic acids in coal and lignite** (PODBREZNIK). **Optical activity of hydrocarbons derived from resin acids** (ZELINSKI and SEMIGANOWSKI).

PATENTS.

Coke ovens. C. A. BAGLIN (B.P. 318,448, 17.12.28).—In a coke oven composed of U-shaped elements in which combustion takes place in two stages (cf. B.P. 259,078; B., 1926, 1004), the two adjacent flues composing each element are provided at their lowest part, and below the point at which combustion commences in the ascending flue, with a number of canals allowing the passage of a controllable volume of products of combustion from the descending to the ascending flue. The flame is thereby increased in length and the heating made more uniform.

A. B. MANNING.

Coke ovens. F. TOTZEK, Assee. of H. KOPPERS A.-G. (B.P. 300,278, 8.11.28. Ger., 10.11.27).—Regenerators are provided extending longitudinally under the oven chambers, and each is divided into two portions by a partition wall transverse to the length of the chambers. A horizontal extension channel leading from each regenerator portion passes above the companion regenerator portion. Each portion and its corresponding extension channel are directly connected with the heating wall above, so that each heating wall is supplied over the whole of its length with heated gases rising from one of the regenerator portions.

A. B. MANNING.

Coking retort ovens. KOPPERS Co., Assees. of J. VAN ACKEREN (B.P. 304,743, 26.3.28. U.S., 26.1.28).—A coking retort-oven battery with alternate coking chambers and heating walls has two or more separate heating systems, each with its own intake and offtake regenerators. Each heating wall comprises a group of flame flues of one system superposed on a group of flues of another system, each group being connected in series with a corresponding group in another heating wall. The intake regenerators for all the heating systems are disposed on one side of the battery and the offtake regenerators on the other side. The regenerators extend to about half the height of the battery and are connected to the heating walls at about their middle

point. Provision is made for heating with a rich gas if desired.

A. B. MANNING.

Carbonising apparatus. G. W. WALLACE, ASST. to S.E. Co. (U.S.P. 1,728,582, 17.9.29. Appl., 15.3.27).—A vertical shaft kiln has an inclined bottom. An opening in the lower part of the side wall adjoining the lower edge of the bottom is provided with a sliding door which comprises a grate presented to the interior of the kiln, and a vapour-collecting compartment outside the grate. Means, operable when the door is in position, are provided for covering the opening through which liquids and vapours from the collecting compartment pass to a point outside the kiln.

A. B. MANNING.

Rotary retorts for the distillation or heat treatment of carbonaceous materials. H. NIELSEN and B. LAING (B.P. 319,223, 18.5.28).—Surrounding the lower end of a rotary retort is an annular drum. A discharge door in the retort communicates with the interior of the drum and a second door leads from the drum to the atmosphere. The doors are slidably mounted in suitable frameworks and are operated by two motors supplied with current in such a manner that they intermittently and alternately open and close each door.

A. B. MANNING.

Regulation of furnaces working with different fuels. C. LÖBBECKE (B.P. 300,204, 1.11.28).—A simple mechanical device, comprising two fixed scales, a moving scale, and a pulley system which transmits the motion of the pointer of one fixed scale to the moving scale with a suitable ratio of transmission, is used to indicate the volume of air which must be supplied to a furnace working with two different fuels, corresponding with any given rates of supply of the respective fuels.

A. B. MANNING.

Operation of internal-combustion engines with pulverulent fuel. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,853, 14.7.28).—Engines of the Diesel type are run with pulverulent fuel and with oxygen or with air enriched with oxygen.

A. B. MANNING.

Dryer and carboniser for fuel agglomerates. F. M. CROSSMAN (B.P. 318,506, 24.5.28).—A central driving shaft supports a number of flat, circular trays within a vertical cylindrical chamber. Each tray is divided into compartments the perforated floors of which are hinged radially. The floors are normally supported by a horizontal circular rail, in which, however, a gap is so arranged that after an almost complete revolution the floors fall open and discharge the contents of the corresponding compartments on to the tray below. A current of hot gas or superheated steam is passed up through the trays and the material contained therein.

A. B. MANNING.

[Low-temperature] carbonisation of coal. A. SMALLWOOD and J. FALLON (B.P. 319,464, 14.8.28).—The apparatus consists of a long horizontal retort or heating chamber constructed of heat-resisting steel surrounded by brickwork, a horizontal cooling chamber in line with the retort, and a continuous conveyer which carries the coal through the two chambers in succession. A feed hopper is situated at one end of the apparatus and a discharge outlet at the other end. The conveyer

returns through a long narrow conduit below the retort and is so arranged as to be completely enclosed. The retort, the underside of which is arched, is heated externally from below.

A. B. MANNING.

Treatment of carbonaceous material for fuel. TRENT PROCESS CORP., ASSEES. of W. E. TRENT (B.P. 293,454, 25.6.28. U.S., 7.7.27).—The size of the coal-oil agglomerates formed as described in B.P. 183,430 (B., 1923, 392 A) is controlled by varying the water dilution and the temperature of the materials. In the first stage of the process these conditions are adjusted to give agglomerates of a size, e.g., $\frac{1}{4}$ in., capable of being carried by the moving liquid. These agglomerates are then further treated by agitation in clean cold water in a second apparatus, whereby they coalesce into larger masses which may be baked directly to form a solid fuel.

A. B. MANNING.

Distillation of solid carbonaceous materials and manufacture of briquettes. H. NIELSEN and B. LAING (B.P. 319,895, 28.8.28).—Caking coal is mixed with a non-caking carbonaceous material and heated to a temperature, e.g., 350–400°, at which the former becomes plastic. The mixture is then briquetted and the briquettes are subjected to a further heat-treatment at a higher temperature, e.g., 850–1000°. The non-caking material may be a non-caking coal, a caking coal which has undergone a preliminary heat-treatment in an oxidising atmosphere, high- or low-temperature coke breeze, etc.

A. B. MANNING.

Distillation of fine granular bituminous material. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES. M.B.H. (B.P. 301,907, 27.11.28. Ger., 9.12.27).—Coal etc. is distilled in layers of less than 2 mm. thickness and at temperatures only slightly above the dew point of the tar, e.g., 380°, for pit coal and lignite. The process is advantageously carried out on a revolving, annular, horizontal hearth as described in B.P. 289,137 (B., 1928, 436).

A. B. MANNING.

Manufacture of carbon. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,140, 17.9.28. Addn. to B.P. 286,845; B., 1928, 357).—The yield and quality of the carbon produced by heating carbon monoxide in the presence of small amounts of iron carbonyl etc., as described in the main patent, are improved by the addition of small quantities (0.5–5%) of water vapour to the gas.

A. B. MANNING.

Production of light hydrocarbons from carbonaceous materials and similar bodies containing complex organic compounds. J. M. F. D. FLORENTIN and A. J. KLING (B.P. 302,354, 30.11.28. Fr., 15.11.27).—Low-temperature tars, lignites, or coals are heated with hydrogen under high pressures in the presence of dehydrating catalysts (cf. B.P. 263,082 and 276,007; B., 1927, 836), non-hydrogenating substitutive catalysts (cf. B.P. 253,507; B., 1927, 696), and hydrogenating catalysts such as reduced iron, nickel, or cobalt, or unstable compounds, e.g., suboxides, sulphides, or nitrides, of tungsten, vanadium, molybdenum, uranium, iron, manganese, etc. The mixture may be heated successively at different temperatures, each corresponding with the optimum conditions for the action of one of the catalysts.

A. B. MANNING.

Manufacture of fertilisers and hydrocarbons from coal and other carbonaceous materials. W. W. HARRIS (B.P. 318,404, 26.9.28).—Coal, or similar carbonaceous material, is mixed with quicklime and common salt and is subjected to the action of steam under a pressure of at least 800 lb./in.²

A. B. MANNING.

Gas apparatus. F. D. MOSES (U.S.P. 1,727,892, 10.9.29. Appl., 22.11.24).—A vertical reaction chamber comprises an upper tapered portion which can be heated externally and constitutes the carbonising zone, and a lower cylindrical portion which is supplied with an air-blast and forms the gasification zone. The upper part of the carbonising zone is more sharply tapered than the lower part.

A. B. MANNING.

Apparatus for making oil gas. A. E. DICKERMAN, ASSR. to GASCO POWER CORP. (U.S.P. 1,728,400, 17.9.29. Appl., 25.8.22).—An oil-gas retort comprises a chamber which can be heated at one end, an oil-vaporising tube extending within the chamber, and gasifying tubes leading from the chamber, outwardly spaced from the vaporising tube and exposed to further heat. The vaporising tube is adapted to receive oil and water and to deliver the vapours near to the heated end of the chamber, on which, however, they are prevented from impinging directly by a baffle. The gasifying tubes lead to an exit conduit for the gases.

A. B. MANNING.

Regenerative water-gas producers. GAS U. TEER GKS.M.B.H. (B.P. 296,374, 30.8.28. Ger., 30.8.27).—The total chamber space of a regenerative water-gas producer is divided into as large a number of smaller chambers as possible; these are connected in pairs, and the phases at which change-over is effected are so arranged that the individual pairs of chambers are changed over consecutively, preferably at regular intervals. The gas, air, and exhaust valves of each chamber communicate with the corresponding valves of the remaining chambers by means of common pipings.

A. B. MANNING.

Utilisation of the heat of the waste gases of alternately-working gas generators and that of the produced gas. A. BREISIG (U.S.P. 1,728,720, 17.9.29. Appl., 11.6.26. Austr., 13.6.25).—In the operation of a plant comprising a gas generator, a heat accumulator serving as a vaporiser, a recuperator for superheating the steam, and a waste-heat boiler, a step is included which consists of simultaneously passing part of the waste gases leaving the recuperator into the vaporising accumulator and another part into the boiler.

A. B. MANNING.

Gas-purification process. C. J. RAMSBURG, ASSR. to KOPPERS Co. (U.S.P. 1,727,559, 10.9.29. Appl., 8.9.23).—The gas is passed through a mass of inert solid material which is drenched with a solution inactive to the impurities in the gas. The solution discharged from the inert material is regenerated by heating and aeration, and the material is drenched intermittently with the regenerated solution.

A. B. MANNING.

Purification of coal gas. MANCHESTER OXIDE Co., LTD., and R. H. CLAYTON (B.P. 319,382, 23.5.28).—The hot gas drawn from the hydraulic main is passed up through a tower filled with a loose packing material,

e.g., Raschig rings, the whole being maintained at a temperature above the dew point of the gas for water. "Tar fog" is thereby eliminated, and in particular the higher tar acids are removed. A considerable portion of other oxidisable materials are removed, and a further reduction in the contamination of the ammonia-still effluent liquor is effected, if dephenolated tar is passed down the tower countercurrent to the gas. The ammonia in the gas after passing through the tower may be recovered by the "direct" process.

A. B. MANNING.

Recovery of sulphur from gases. C. STILL (B.P. 297,062, 13.9.28. Ger., 13.9.27).—Gases containing hydrogen sulphide are washed with a suspension of a metal oxide in a dilute alkaline solution containing a soluble foam-producing agent, *e.g.*, soap, saponin. On regeneration of the spent liquor by oxidation the sulphur separates in a particularly pure form.

A. B. MANNING.

Purification of gas containing sulphuretted hydrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,396, 20.6.28).—Gases containing hydrogen sulphide, and preferably also ammonia, are purified by catalytic oxidation of the hydrogen sulphide, the catalyst consisting of a heavy metal, preferably one capable of combining with sulphur, to which has been added up to 10% of an alkali or alkaline-earth salt, or of lead, bismuth, copper, or their compounds. One or more oxygen-transferring metals, group IV, V, or VI (*e.g.*, chromium, vanadium), may also be added. The sulphur dioxide is recovered in the form of a sulphite-bisulphite mixture by treatment with a suitable alkaline agent.

A. B. MANNING.

Separation of by-products and of other secondary constituents from coke-oven gases and from other gas mixtures. CONCORDIA BERGBAU-A.-G., J. I. BRONN, and G. FISCHER (B.P. 318,319, 9.6.28).—Gases, the principal constituents of which are to be separated by compression and cooling to a very low temperature, are pretreated at an intermediate temperature, *e.g.*, —60°, and at a comparatively low stage of compression, by washing successively with concentrated ammonia of low f.p. or with liquefied ammonia gas, and an organic solvent for acetylene, *e.g.*, acetone. The washing may be followed by cooling to about —90° in order to bring about complete condensation of the vapours of the washing liquids, or these vapours may be absorbed in acid, alkali, and/or solid adsorbents.

A. B. MANNING.

Apparatus for detecting the presence of explosive or combustible gases. C. MCLUCKIE (B.P. 319,231, 16.6.28).—A metallic vessel is divided into two chambers; in one the gas to be tested can be subjected to the action of a hot platinum wire, whilst the other serves as a compensator for variations in the temperature and pressure of the atmosphere. By means of a multiple-way cock the chambers may be connected, respectively, to the source of gas and to the air, both chambers may be closed, or they may be connected, respectively, to the two limbs of a U-tube manometer. The diminution in pressure after combustion gives a measure of the combustible gas, *e.g.*, methane, present in the gas tested.

A. B. MANNING.

Apparatus for indicating the presence of inflammable vapours or gases. H. T. RINGROSE (B.P. 319,530, 30.10.28).—The diminution in pressure produced within a porous vessel by the combustion of the inflammable constituents of a gas which has passed into the vessel is used to break the circuit of a lamp when the percentage of the inflammable constituents reaches a predetermined quantity. The lamp circuit and the heating filament of the vessel are connected in series. The circuit is broken automatically by the action of the diminished pressure on a collapsible diaphragm.

A. B. MANNING.

Volumetric determination of explosive gases or gaseous mixtures, e.g., eudiometric measurement of firedamp. J. G. DALOZ (B.P. 300,281, 10.11.28. Belg., 10.11.27).—The apparatus includes two chambers of identical volume and a small hand-pump by means of which they may be filled with the gaseous mixture. The chambers are connected by a U-tube manometer which serves to measure the contraction resulting from the combustion of the mixture in one chamber, brought about by passing a current through a small heating resistance therein. From the contraction the percentage of combustible gas in the mixture may be deduced. The reading may be confirmed by subsequently burning the gas in the other chamber.

A. B. MANNING.

Distillation of tar. BARRETT Co., Assees. of S. P. MILLER (B.P. 286,278, 315,368, 315,373—4, 2.3.28. U.S., 2.3.27).—(A) The tar is introduced into a still, through the vapour space of which are passed the hot gases from a number of coke ovens, the temperature of the gases being sufficiently high to effect rapid distillation of the tar. A small proportion of the tar is circulated continuously, in the form of spray, through the hot gas current. The still may be run intermittently or continuously. The gases and vapours leaving the still may be used to preheat the entering tar, and are then subjected to fractional condensation. (B) The gases and vapours leaving the still are passed through an electrical precipitator in order to remove suspended material therefrom before condensing the oils. The material so removed may be drawn off as a separate pitch product or may be returned to the still. (C) The gases and vapours leaving the still are scrubbed with tar before passing to the condensing system. They are thereby cleaned, whilst the tar used for scrubbing undergoes a partial distillation in the process. The tar leaving the scrubber is preferably passed into the still. (D) The hot gases for carrying out the distillation are formed by combining the gases from a number of ovens or retorts in order to equalise the fluctuations in the temperature, volume, and composition of the gases from individual ovens. The gases from the remaining ovens, comprising the greater part of the battery, are combined and cooled for the recovery of tar therefrom, which is subsequently distilled as above described.

A. B. MANNING.

Dehydration of tar. BARRETT Co., Assees. of S. P. MILLER (B.P. 287,084, 5.3.28. U.S., 14.3.27).—Tar is dehydrated, or dehydrated and distilled, by passing hot gases from the carbonisation of coal through a body of tar contained in a still, which is not otherwise heated.

A. B. MANNING.

Manufacture of liquid or pasty emulsions from natural or artificial bitumen or tar. H. PLAUSON (B.P. 319,101, 25.7.28).—Bituminous materials are emulsified in water in the presence of animal or vegetable fats or oils, an alkali carbonate, calcium hydroxide, and, if desired, a water-soluble organic compound such as aniline, furfural, etc. The oil or fat is first emulsified in water containing the alkali carbonate, and the heated bitumen, to which the aniline etc. has been added, is then emulsified therein, before or after the addition of the calcium hydroxide.

A. B. MANNING.

Treatment of liquids containing tar acids. KOPPERS Co., Assees. of J. A. SHAW (B.P. 299,837, 12.4.28. U.S., 2.11.27).—The heated liquid is passed down a tower filled with suitable contact material, and is exposed therein to a rising current of inert gas which volatilises and carries away the tar acids. These are recovered from the gas by treatment with alkali, and the purified gas is recirculated for the treatment of further quantities of liquid. The gas becomes saturated with the vapours of the volatile constituents of the liquid other than tar acids, and the temperature and other conditions are so adjusted that no undesirable condensation of these vapours will occur. A gas liquor may be subjected to this treatment between the free and fixed ammonia distillation stages, or the tar acids may be driven off with the free ammonia and recovered from a first condensate obtained by cooling to a temperature which will effect the condensation of the tar acids with as little as possible of the ammonia.

A. B. MANNING.

Distillation of petroleum. E. H. LESLIE and E. M. BAKER (U.S.P. 1,730,891, 8.10.29. Appl., 20.3.23).—Petroleum is heated and passed to a flash vaporiser, from which the vapours are led to the bottom of a fractionator, consisting of a number of sections placed one above the other, up which the vapours pass counter-current to condensate from a reflux. Either liquid products or vapours can be withdrawn as desired from any of the sections.

H. S. GARLICK.

Catalytic cracking of petroleum oils, tar, and the like. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 319,341 and Addn. B.P. 320,286, [A] 19.12.28, [B] 20.12.28. Fr., [A] 21.9.28, [B] 2.10.28).—(A) In a process for cracking the oils etc. at atmospheric pressures, they are vaporised as completely as possible, and the vapours are led first to a catalyser, and thence to a dephlegmator. The light products are removed, and the residual liquors led to a second vaporiser-catalyser-dephlegmator group followed, in series, by other similar groups. These are arranged to be used in parallel or in a closed circuit of any number of cycles. (B) Between the catalyser and dephlegmator is placed a purifier containing divided oxides or metals on a suitable carrier, this being maintained at a temperature sufficient to avoid condensation.

W. S. E. CLARKE.

Manufacture of light hydrocarbons by catalytic cracking of petroleum oils, tars, or other similar starting materials. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 320,287, 21.12.28, Fr., 24.10.28).—In a further elaboration of the apparatus described in B.P. 319,341 and 320,286 (cf. preceding abstract) a connexion is made to the

dephlegmators of groups of purifiers for the refining of light products issuing from the dephlegmators. These groups consist of a heater for the light products, followed by one or more purifying chambers for retaining sulphur compounds, and one or more catalytic reaction chambers, and finally a condensing apparatus. Non-condensed products are collected in absorbers before the evacuation of the permanent gases. The vaporiser of each main group receives oil heated in successive heaters at a progressively increasing temperature, additional moisture being supplied as steam. W. S. E. CLARKE.

Distilling and cracking or decomposing bodies such as oils and the like by contact with salt or metal melts. T. SEIFER (B.P. 320,126, 28.6.28).—The melt is circulated under thermodynamic action through a heating chamber disposed in a furnace to a vertical treating chamber, to the bottom of which the liquid to be treated is supplied and from the top of which the products are withdrawn, the melt returning to the heating chamber by means of a downwardly-directed pipe outside the furnace. H. S. GARLICK.

Converting solid carbon into liquid hydrocarbons. P. DANCKWARDT (U.S.P. 1,730,997, 8.10.29. Appl., 13.1.28).—Carbon or carbonaceous material is forced together with hydrogen under high pressure through a quantity of molten metal hydroxide which is being electrolysed, using finely-divided nickel as the cathode and an anode consisting of an electrical conductor through or near which fixed hydrocarbon gas is injected. The carbonaceous material is allowed to escape into another vessel where the light hydrocarbons are separated from the heavier ones, the latter being returned with fixed carbonaceous material to the electrolyser for re-treatment. H. S. GARLICK.

Purifying and cooling the gaseous cloudy substance obtained by heat-transformation of heavy oils. C. CHILOWSKI (B.P. 288,305, 4.4.28. Fr., 8.4.27).—The gas produced by cracking heavy oils as described in B.P. 255,423, 271,899, and 271,907 (B., 1927, 805, 867) is maintained at a high temperature (above 500°) until it issues from the gas producer, and is then suddenly cooled by sprays of water or heavy oil to below 100°. This brings about the condensation of the suspended particles of tar and carbon in the gas, which would otherwise form a deposit on the walls and in the pipes of the intermediate apparatus leading to the engine. [Stat. ref.] A. B. MANNING.

Dehydrator [for crude oil etc.]. V. L. GIBSON (U.S.P. 1,727,504, 10.9.29. Appl., 16.2.27).—The separation of water from a liquid of differing sp. gr. is effected by gravity while the mixture is flowing slowly in thin layers. The mixture passes generally upwards with a number of horizontal passes through passages enclosed on all sides and heated on all sides by thermostatically controlled steam passing upwards through a containing tower. The horizontal pans are provided with riffles into which the water (or liquid of higher sp. gr.) settles, and from which it is drained by automatic valves. Subsequent to the separation, the oil (or liquid of lower sp. gr.) is cooled in similar towers provided with an air draught instead of with steam, and in which the zig-zag passages are not provided with riffles. B. M. VENABLES.

[Fuel for] airship. J. I. BRONN (U.S.P. 1,731,840, 15.10.29. Appl., 6.3.28. Ger., 14.3.27).—In an airship of the type in which the heat energy for operating the engines is obtained by burning buoyant fuel gas and liquid fuel in such proportions that the buoyancy is practically independent of the fuel consumption, the fuel gas consists of a mixture of hydrocarbons free from components liquefiable at atmospheric temperatures, and comprises methane mixed with a smaller proportion of hydrocarbons of higher b.p. F. G. CROSSE.

Treatment of carbonaceous materials in electric furnaces or the like. J. J. NAUGLE (U.S.P. 1,731,473—4, 15.10.29. Appl., [A, B] 21.4.23).—See B.P. 267,240—1; B., 1927, 694.

Distillation of carbonaceous material. O. H. HERTEL (U.S.P. 1,731,165, 8.10.29. Appl., 15.3.24).—See B.P. 247,639; B., 1926, 350.

Making infusible asphalt masses of high elasticity. R. LICHTENSTERN (B.P. 320,579, 16.1.29).—See U.S.P. 1,720,487; B., 1929, 769.

Feeding frames and lids for retort-bench producers, furnaces, and the like. C. D. DRURY and F. P. FALL (B.P. 319,872, 28.7.28).

Machines for crushing coke, coal, and other materials in the form of lumps. I. H. PILOT (B.P. 319,864—5, 21.7.28).

[Coke-extractor gear for] coke chambers of vertical retorts for carbonisation of coal and the like. E. WEST, and WEST'S GAS IMPROVEMENT CO., LTD. (B.P. 320,232—3, 11.10.28).

Gas burners. J. MILES (B.P. 320,083, 2.7.28). BASTIAN-MORLEY CO. (B.P. 312,981, 6.7.28. U.S., 5.4.28). F. KING, and F. KING & CO. (BATH), LTD. (B.P. 320,453, 15.8.28).

[Head for upright] incandescent mantles. ALADIN INDUSTRIES, LTD., and C. W. DAVIS (B.P. 294,536, 10.7.28. U.S., 25.7.27).

Extinguishing fire (B.P. 319,083). Charging gases with vapours (B.P. 305,089). Distillation (U.S.P. 1,730,892). Gas analysis (U.S.P. 1,727,544).—See I. Treatment of partial oxidation products (U.S.P. 1,721,959). Dibasic acid salts (U.S.P. 1,721,958).—See III. Revivification of potassium ferrocyanide (B.P. 319,147).—See VII. Bituminous coatings (B.P. 320,123).—See IX. Cultivation of micro-organisms (B.P. 318,649).—See XVIII.

III.—ORGANIC INTERMEDIATES.

Synthesis of carbamide from ammonia and carbon dioxide. G. JAKOVKIN (Trans. Sci. Inst. Sci.-Tech. Dep. U.S.S.R. [Russia], Papers Chem., 1928, No. 2, 207—224).—Carbon dioxide and ammonia are preferably caused to react continuously under pressure without cooling. The conversion of the ammonium carbamate into carbamide and water by heating in an autoclave is effected at temperatures not above 160—170° during 6 hrs. The yield of carbamide is then 40% of the theoretical at 75—80 atm. Catalysts are not advantageous. Nickel and non-corroding chromium-nickel steel (Krupp V4A) are the best available materials. A. A. ELDRIDGE.

Chlorination of benzene in the presence of iron and ferric chloride. P. W. KARLASCH (Ukraine Chem. J., 1929, 4, [Tech.], 145—151).—Results obtained by this method were compared with those obtained by former investigators (cf. Bourion, B., 1920, 480 A). The results obtained with anhydrous ferric chloride (1% by wt. of benzene) and iron were substantially the same, and agreed with the former data, yielding 65—75% of monochlorobenzene, and 10—15% of polychlorobenzenes (of which about 30—40% was *p*-dichlorobenzene). In commercial practice, therefore, the method of chlorination in the presence of metallic iron is expedient, since it is considerably cheaper than that in which ferric chloride is used; the latter method necessitates a preliminary drying of the benzene and purification of the catalyst by sublimation. M. ZVEGINTZOV.

Electrochemical oxidation of toluene. R. W. MITCHELL (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 17 pp.).—An indirect method, in which toluene was sulphonated and a solution of the product electrolysed, was investigated, as well as the direct method of electrolysing an aqueous solution of an electrolyte containing toluene in suspension. In the former method the highest current efficiency in the production of *p*-sulphobenzoic acid was 21·8%, using a 40% aqueous solution of *p*-toluenesulphonic acid at 100° as electrolyte and an anode constructed of lead-wool. Further anodic oxidation of *p*-sulphobenzoic acid keeps the current efficiency low. In the direct method, using an emulsion of toluene with 2*N*-sulphuric acid or sodium hydroxide, hardly any oxidation occurred; increasing the degree of dispersion of the toluene by means of various emulsifying agents caused no improvement, nor did this procedure increase the yield or velocity of the purely chemical reaction between toluene emulsions and oxidising agents such as ammonium persulphate solution. In the electrolytic process increasing the surface of contact between the anode and the toluene by using a lead-wool anode in a well-stirred emulsion yielded only traces of desirable oxidation products, although the low current densities employed must have kept the oxygen overvoltage low. Experiments in which a catalyst was added to the toluene emulsion were then conducted under various conditions, using a sheet lead cathode. With a rich toluene emulsion in 55% sulphuric acid containing manganous sulphate at 50°, benzaldehyde was produced at a current efficiency of 61% when the toluene phase was frequently or continuously removed from the cell, and the oxidation products were extracted. With chromic instead of manganous sulphate benzoic acid was produced with an efficiency as high as 54% under similar conditions. In these processes the oxidation of the toluene is brought about by manganic sulphate and chromic acid, which are the respective primary anode products, but a certain amount of cresol is produced by direct anodic oxidation of the toluene. H. J. T. ELLINGHAM.

1-Chloronitronaphthalenes. R. ASAOKA (Bull. Tokyo Ind. Res. Inst., 1928, 23, 1—26).—The following eutectics were observed for the 1-chloro-mono- and -di-nitronaphthalenes: 1:4 (57%)—1:5, 74—75°; 1:4 (57%)—1:8, 52—53°; 1:5 (45%)—1:8, 66—

67°; 1:4:5 (34%)—1:4:8, 117—118°. Methods of preparation of the pure materials are described.

CHEMICAL ABSTRACTS.

Phenols from carbonisation of lignite. ROSIN and JUST.—See II. **Perfumes etc. from furfuraldehyde and its derivatives.** GILMAN and WRIGHT.—See XX.

See also A., Nov., 1247, **Electrochemical preparation of phenylhydrazine** (McCLURE). 1261, **Apparatus for distillation at very low pressures** (WATERMAN and ELSBACH). 1276, **Electrolytic reduction of acetone** (HAGGERTY). 1288, **1-Methylnaphthalene derivatives** (VESELÝ and others). 1289, **Synthesis of aniline** (SMIALOWSKI). 1291, **Electrolytic preparation of 2:4-diaminophenol** (BRADT and BROWN). 1302, **Derivatives of naphthaquinones** (LANTZ and WAHL). 1303, **Partly hydrogenated naphtha- and anthra-quinones** (DIELS and others). 1304, **Benzanthrone** (LÜTTINGHAUS and others). 1310, **Aldehydo-fluorescein** (SEN and BANERJEA). **Condensation of esters with resorcinol, dimethylaniline, and *m*-diethylaminophenol** (SEN and MUKHERJI). 1341, **Bacterial oxidation of phenanthrene** (TAUSSON).

PATENTS.

Treatment of partial oxidation products [of hydrocarbon oils]. J. H. JAMES (U.S.P. 1,721,959, 23.7.29. Appl., 18.5.23).—Aldehydic partial oxidation products obtained by catalytic vapour-phase oxidation of hydrocarbon oils are further oxidised to mono- or di-basic fatty acids, *e.g.*, with chromic acid, chlorine, 22% nitric acid below 50° in presence of an air current, or further catalytic oxidation. *E.g.*, a catalytic oxidation mixture from Pennsylvania kerosene, containing 81% of aliphatic aldehydes, 10% of aldehydic fatty acids, and 9% of hydrocarbons, is converted at 300—330°, with uranyl molybdate catalyst screen, into a mixture containing 65% of aldehyde fatty acids, 31·5% of aliphatic aldehydes, and 3·5% of hydrocarbons.

R. BRIGHTMAN.

Manufacture of highly polymerised products of unsaturated hydrocarbons. H. STAUDINGER and H. A. BRUSON (U.S.P. 1,720,929, 16.7.29. Appl., 15.3.27).—Unsaturated diolefines containing at least four carbon atoms are treated with stannic chloride in chloroform or other inert solvent. R. BRIGHTMAN.

Manufacture of emulsions of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,296, 1.6.28. Cf. B.P. 312,201; B., 1929, 612).—Emulsifying agents are produced *in situ*, *e.g.*, by dissolving oleic acid in the diolefine and ammonia in water and mixing the two solutions. Polymerising agents, oxidants, vulcanisation accelerators, driers, proteins, pectins, resins, plasticisers, and stabilisers may be added to one or other of the two solutions. An artificial latex is obtained, for example, by agitation of a solution of benzoyl peroxide (5 pts.), cobalt oleate (0·5 pt.), olein (12·6 pts.), an accelerator, and linseed oil (10 pts.) in β -methylbutadiene (400 pts.) with a solution of trisodium phosphate (10 pts.), 5% glue solution (25 pts.), and 10% hydrogen peroxide (5 pts.) in 0·5% aqueous ammonia (500 pts.). C. HOLLINS.

Manufacture of ketones [from glycols]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,124,

15.5.28).—Glycols are passed in vapour form at 210–220° over a catalyst comprising a heavy metal of group I or VIII (copper) together with an alkaline promoter and, if desired, a difficultly reducible metal oxide. Examples are: methyl ethyl ketone from γ -butylene glycol, methyl *n*-propyl ketone from $\beta\delta$ -amylene glycol, methyl isopropyl ketone from trimethylethylene glycol, phenylacetone from γ -phenyl- $\alpha\beta$ -propylene glycol.

C. HOLLINS.

Manufacture of homologues of dioxan. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,758, 24.9.28).—Homologous glycols, *e.g.*, the mixture from cracked gases, or $\alpha\beta$ -propylene glycol, are boiled with a little concentrated sulphuric acid or sodium hydrogen sulphate.

C. HOLLINS.

Extracting [fatty] acids from solutions. H. E. MARTIN, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,717,719, 18.6.29. Appl., 3.12.27).—Aqueous solutions of acetic acid or other fatty acids of low mol. wt. are concentrated by extraction with methylene chloride or mixtures containing at least 80% of methylene chloride and less than 20% of ether, *e.g.*, by counter-current processes with methylene chloride in liquid or vapour phase.

R. BRIGHTMAN.

Manufacture of dibasic acid salts. J. H. JAMES (U.S.P. 1,721,958, 23.7.29. Appl., 4.4.22. Renewed 5.12.28).—Organic acids obtained, *e.g.*, by the partial oxidation of mineral oil are oxidised to dibasic acids, preferably with chlorine or hypochlorite in alkaline solution, and the dibasic salts or soaps are salted out. The dibasic acids may be liberated from the salts and converted into a mixture of the sodium ammonium or potassium ammonium salts.

R. BRIGHTMAN.

Manufacture of pure *m*- and *p*-cresols. SCHERING-KAHLBAUM A.-G. (B.P. 297,083, 29.8.28. Ger., 14.9.27).—The compound of *m*-cresol and carbamide separates readily (especially on seeding) in a pure state when carbamide is added to crude *m*-cresol or to a mixture of *m*- and *p*-cresols. A solvent (carbon tetrachloride or a small amount of water) may be used. The carbamide-cresol compound may be decomposed by distillation in a vacuum or by treatment with acid.

C. HOLLINS.

Purification of phthalic anhydride. P. C. BOWERS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,728,225, 17.9.29. Appl., 27.10.22).—Phthalic anhydride obtained by the oxidation of naphthalene vapour is heated with a condensing agent capable of rendering the impurities less volatile than the anhydride, *e.g.*, an alkali hydroxide, a metal chloride, or an alkali hydrogen salt of a polybasic mineral acid.

L. A. COLES.

Separation of α -naphthylamine from a mixture of α - and β -naphthylamines. H. J. WEILAND and I. GUBELMANN, Assrs. to NEWPORT Co. (U.S.P. 1,722,026, 23.7.29. Appl., 28.6.26).—The β -naphthylamine is dissolved out with dilute acid from mixtures containing less than 15% of β -naphthylamine, *e.g.*, by heating at 50° with 3–4 mols. of hydrochloric acid per mol. of β -naphthylamine. The latter is recovered quantitatively from the cold filtrate by addition of sulphuric acid.

R. BRIGHTMAN.

Manufacture of 5:7-dialkoxy-3-[hydr]oxythio-naphthens [thioindoxyls] and of dyes therefrom. I. G. FARBENIND. A.-G. (B.P. 291,361, 30.5.28. Ger., 30.5.27).—5:7-Dialkoxythioindoxyls are converted into thioindigos by oxidation or are condensed with isatins etc. 2-Nitro-3:5-dimethoxybenzoic acid, m.p. 232°, is reduced to the amine, m.p. 189–190°, and converted into 3:5-dimethoxy-1-carboxyphenyl-2-thioglycollic acid, m.p. 105–107° (or 128–130°, anhydrous), which on alkaline fusion gives 5:7-dimethoxythioindoxyl, m.p. 143°. This is oxidised to the thioindigo (bluish-grey), or is condensed with the *p*-dimethylaminoanil of 6-chloro-4-methylthioisatin (violet), or with 5:7-dichloroisatin α -chloride (bluish-grey).

C. HOLLINS.

Production of butyric aldehyde. M. MUGDAN and J. WIMMER, Assrs. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,730,587, 8.10.29. Appl., 30.3.27. Ger., 15.5.26).—See B.P. 271,103; B., 1928, 151.

Manufacture of chlorinated aldehydes of the aromatic series. G. KALISCHER, H. SCHEYER, and K. KELLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,717,567, 18.6.29. Appl., 30.1.28. Ger., 2.2.27).—See B.P. 311,208; B., 1929, 747.

Manufacture of chlorine-substituted product of 1-amino-2:4-dimethylbenzene [*m*-4-xylydine]. E. HOFFA, E. RUNNE, and E. THOMA, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,730,729, 8.10.29. Appl., 21.9.27. Ger., 6.10.26).—See B.P. 278,729; B., 1928, 440.

Manufacture of nuclear-substitution product of 1-aminonaphthalene-8-carboxylic acid in the open or anhydride form. R. HERZ and F. SCHULTE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,728,995, 24.9.29. Appl., 29.11.26. Ger., 4.12.25).—See B.P. 296,010; B., 1928, 780.

Concentration of nitric acid (B.P. 320,125). **Sodium glutamate** (B.P. 290,657).—See VII. **Butyl alcohol and acetone by fermentation** (B.P. 319,642).—See XVIII.

IV.—DYESTUFFS.

New derivatives of *p*-phenylenediamine and their application for hair-dyeing. H. MEYER (Chem.-Ztg., 1929, 53, 765–766).—The injurious action of diamines on the skin is related to their basicity and diminishes, *e.g.*, in the order: *p*-phenylenediamine, 2:5-tolylene-diamine, *p*-aminodiphenylamine. If both amino-groups are neutralised with acid, the salts are unsuitable for dye production. With carboxylic acids these bases form only mono-acid salts, which are crystalline substances, oxidised readily to dyes, and free from harmful action on the skin; they do not, like the diamines, combine with butyric acid to give powdery salts which close the skin-pores. The use of these compounds in hair-dyeing is protected by patent application. Examples are: *p*-phenylenediamine salicylate, m.p. 137° (decomp.), gallate, m.p. 180° (decomp.), benzoate, *o*-nitrobenzoate, acetate, and lactate; 2:5-tolylene-diamine salicylate; *p*-aminodiphenylamine salicylate.

C. HOLLINS.

See also A., Nov., 1231, **Absorption of dyes by growing crystals** (MILLIGAN). 1248, **Colour selectivity in dye systems** (WEIGERT and NAKASHIMA).

1251, Ultramarine (HOFFMANN). 1262, Scopometer (EXTON). 1310, Aldehydofluorescein (SEN and BANERJEA). Condensation of esters with resorcinol, dimethylaniline, and *m*-diethylaminophenol (SEN and MUKHERJI). 1313, Reaction products from indigotin (POSNER and others).

PATENTS.

Azo dyes from rubber derivatives. II. L. FISHER, Assr. to B. F. GOODRICH Co. (U.S.P. 1,724,270, 13.8.29. Appl., 28.12.26).—The product from caoutchouc dibromide and phenol (2 mols.) is coupled with a diazotised arylamine, *e.g.*, sulphanilic acid, aniline, or benzidine. The dyes are maroon or purple in acid and yellow in alkali.

C. HOLLINS.

[Manufacture of] disazo dyes. H. JORDAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,724,663, 13.8.29. Appl., 25.8.27).—Disazo dyes of the type, $A \rightarrow B \rightarrow C$, containing at least two sulphonic or carboxylic groups but no free hydroxyl, and carrying, in the end-component only, a free amino-group, are condensed with *p*-nitrobenzoyl chloride and the reduced product is further condensed with an aliphatic or aromatic acid chloride or anhydride. The *p*-benzamidobenzoyl derivative of the dye amido-G-acid \rightarrow *m*-toluidine \rightarrow *m*-toluidine (yellow on cotton, wool, silk, or viscose) is specially claimed. Other examples are the *p*-benzamidobenzoyl derivatives of: 2:4:8-naphthylaminedisulphonic acid \rightarrow cresidine \rightarrow *o*-anisidine (orange); metanilic acid \rightarrow Cleve acid \rightarrow *m*-toluidine (yellow-brown); sulphanilic acid \rightarrow Cleve acid \rightarrow α -naphthylamine (red-brown).

C. HOLLINS.

Orange to brown [direct] disazo dyes and their manufacture. H. JORDAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,724,660, 13.8.29. Appl., 13.11.25).—Aminobenzamidobenzoyl-J-acids are used as middle-components, the end-components being a sulphonated pyrazolone or methylindole. The dye, aniline \rightarrow 4-*p*-aminobenzamidobenzoyl-J-acid \rightarrow 1-*p*-sulphophenyl-3-methylpyrazolone (yellow-orange) is specially claimed.

C. HOLLINS.

Triarylmethane dye. H. POLIKIER and O. BOEGER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,731,637, 15.10.29. Appl., 8.7.27. Ger., 9.8.26).—See B.P. 275,609; B., 1928, 46.

Manufacture of salt-like compounds derived from dyestuff bases of the triarylmethane series. O. MEYER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,729,003, 24.9.29. Appl., 30.6.27. Ger., 5.7.26).—See B.P. 298,101; B., 1928, 888.

[Manufacture of] vat dyes. P. NAWIASKY and E. KRAUCH, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,729,006, 24.9.29. Appl., 18.6.27. Ger., 22.6.26).—See B.P. 273,247; B., 1928, 517.

Manufacture of azo dyes. H. WAGNER and E. FISCHER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,731,643, 15.10.29. Appl., 19.7.27. Ger., 29.7.26).—See B.P. 275,258; B., 1929, 239.

[Manufacture of acid] monoazo dyes. E. REBER and J. SPIELER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,731,150, 8.10.29. Appl., 22.11.27. Switz., 4.12.26).—See B.P. 281,713; B., 1929, 351.

[Manufacture of water-insoluble] azo dyes. II. GRIMMEL and H. CLINGESTEIN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,731,623, 15.10.29. Appl., 28.7.27. Ger., 19.8.26).—See B.P. 298,697; B., 1929, 48.

Dyes from thioindoxyls (B.P. 291,361).—See III. **Concentration of nitric acid** (B.P. 320,125).—See VII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Decomposition of rye straw and maize stalks by different fermentation processes. E. HORVÁTH (Papier-Fabr., 1929, 27, 664—670).—Samples of rye straw, of maize stalks, and of the rind, knots, and medullary substance of maize stalks, sterilised in different ways, were suspended in water and inoculated with pectin-dissolving cultures from hemp and flax, and with *Botrytis cinerea*. Unsterilised samples were also self-fermented. The extent of fermentation and the changes in chemical properties of the products (ash, nitrogen, protein, pentosan, lignin, etc. contents) were examined. The fermentative power of the cultures used is not evident from the results obtained, but the presence of 10% of lime in suspensions inoculated with pectin-dissolving organisms has a considerable effect on the yield and disappearance of lignin and cellulose. With rye straw in the presence of lime there is a greater loss of lignin and cellulose, but with maize stalks under the same conditions the effects are roughly reversed. In many cases there is not only considerable loss of lignin etc. (up to 50%), but also of cellulose (12—30%). Losses of ash constituents, silica, etc. are greater from the softer and more easily attacked parts of maize stalks than from the hard knots. Although further investigation is necessary to explain the results of such fermentation processes, it may be stated that the possibility of decomposition of a material with micro-organisms is to a great extent dependent on the structure or chemical composition and physical properties of the raw material.

B. P. RIDGE.

Purification of wood pulp: Japanese pulp as rayon material. H. OKADA, T. MATSUDA, and E. HAYAKAWA (J. Cellulose Inst., Tokyo, 1929, 5, 225—233).—By first bleaching sulphite wood pulp and then digesting it with alkaline liquors a perfectly white pulp of low copper number is obtained, but the α -cellulose content and the viscosity are not satisfactory. If the order of treatment be reversed, however, the α -cellulose content of the product is excellent, but the copper number and whiteness are inferior and the viscosity may be low; moreover, the bleaching operation is difficult. The following procedure, which combines the advantages of both methods, is recommended on account of ease of bleaching, economy of materials, and superiority of all the physical and chemical properties of the product. The raw, unbleached pulp is treated with 40% of the amount of chlorine necessary to give the ordinary complete bleach, then digested in the usual manner, and finally treated with the minimum amount of chlorine necessary to give a good white. Digestion of the pulp after chlorination with chlorine water is found to give the best results. Properties of viscoses prepared from cotton tissue-paper pulp, imported pulp, and Japanese pulp purified in the manner described

have been investigated, and it is found that threads produced from the purified pulp have a higher wet tenacity than those from the imported pulp.

B. P. RIDGE.

Some constituents of artificial silk. Y. KAMI (J. Cellulose Inst., Tokyo, 1929, 5, 233—237).—The moisture, ash, and fat and oil contents of 48 kinds of viscose, 7 of cuprammonium, and 1 of acetate silk have been determined. The ash content varies from 0.05 to 2.07% (average 0.25%), but higher values are given by dyed material. It also varies according to the country of origin of the artificial silk; for German material it is about 0.15%, and for French about 0.40%. This variation is due to differences in the cellulose used and in the methods of coagulation, washing, and after-treatment. The fat and oil content is 0.09—0.84%, and also varies according to the country of origin; German and Italian yarns have low, and French and Swiss very high, values. For Japanese varieties the solubility in water has a mean value of about 2% for bleached, and about 5% for unbleached, material, whilst the mean value for the sulphur content of viscoses is 0.02%. In no case is there complete absence of sulphur in viscose.

B. P. RIDGE.

Polysaccharides. XLI. Behaviour of "Lilienfeld silk" towards cellulase. P. KARRER and P. O. MANGELLI (Helv. Chim. Acta, 1929, 12, 989—990).—"Lilienfeld silk," which has the characteristics of a silk spun in a strongly acid bath, is, as expected, more readily attacked by snail cellulase than are other viscose silks (A., 1928, 276), but less readily than cuprammonium silks.

R. K. CALLOW.

Solubility of cellulose derivatives. I, II. E. W. MARDLES (Kolloid-Z., 1929, 49, 4—11, 11—16).—I. The solubility of cellulose acetate and nitrate in a number of solvents at 20° was determined by adding light petroleum (in which the cellulose derivatives are insoluble) from a burette to 5% solutions and observing when opalescence was produced. Solutions of cellulose derivatives in many solvents deposit a gelatinous mass on cooling, and the precipitation temperature has been determined for 5% solutions of cellulose acetate in a number of solvents, and the effect of altering the concentration of the solution has been examined. *cyclo*-Hexane is a useful solvent as the solution does not gelatinise until cooled to -17°. The course of the gelatinisation of a benzyl alcohol solution of cellulose acetate was followed by taking measurements of the viscosity after various time intervals, and can be expressed by the empirical formula $\eta - \eta_0 = ae^{kt}$, where k is a measure of the gelatinisation, t the time, and a is a constant. The maximum gelatinisation temperature, or the temperature at which k becomes zero, varies with the concentration. Below 12° benzyl alcohol practically ceases to be a solvent for cellulose acetate. Measurements were made of the variation of the solubility with temperature for cellulose acetate in acetone and in benzyl alcohol and for cellulose nitrate in acetone.

II. Measurements have been made of the swelling and solvent power of benzyl alcohol-anisole mixtures for some cellulose derivatives. For cellulose acetate

both the swelling and the solubility are at a maximum in a mixture containing 30% of anisole. The solubility of cellulose derivatives in mixtures of two or more liquids which are non-solvents when taken singly is discussed and a theory is advanced. The viscosity, surface tension, and density of the solutions are related to their solvent power. The degree of dispersion, the amount of swelling, and the resistance to gelatinisation on cooling are all greatest in the best solvents.

E. S. HEDGES.

Waste heat in paper mills. SMITH.—See IX.

See also A., Nov., 1234, **Swelling of cellulose acetate in binary mixtures** (SAKURADA). 1235, **Silicic acid gels** (PRASAD and HATTIANGADI).

PATENTS.

Production of artificial silk by the copper oxide-ammonia stretch-spinning process. J. P. BEMBERG A.-G. (B.P. 300,572, 29.8.28. Ger., 15.11.27).—Where a single, circulating precipitating liquid is employed, the copper and ammonia content of the used liquor may be reduced sufficiently for re-use by removing a portion, and replacing it either with fresh precipitating liquid or with used liquor which has been worked up by distilling off ammonia so that the copper is precipitated.

F. R. ENNOS.

Manufacture of viscose. L. LILIENFELD (B.P. 319,378, 22.3.28).—By carrying out at least part of the preparation of the alkali-cellulose at above 80° or even above 100°, the time required for maturing is shortened and a final product of enhanced strength is obtained.

F. R. ENNOS.

Spinning of artificial silk from viscose. J. C. HARTOGS (B.P. 319,887, 20.8.28).—Not more than 0.8% (0.3—0.5%) of finely-divided sulphur, preferably a mixture of colloidal sulphur with a protective colloid, is added at any stage in the manufacture of the xanthate solution prior to spinning.

F. R. ENNOS.

Production of artificial formations from viscose. BORVISK SYND., LTD., Assees. of B. BORZYKOWSKI (B.P. 292,627, 23.6.28. Ger., 24.6.27. Addn. to B.P. 273,647; B., 1927, 963).—A dull matte appearance is produced by the addition of organic compounds containing nitrogen, *e.g.*, casein, or of inorganic compounds, *e.g.*, barium hydroxide, to the viscose before spinning. [Stat. ref.]

F. R. ENNOS.

Preparation or treatment of liquids for use in the manufacture of artificial silk. BRYSLKA, LTD., and F. W. SCHUBERT (B.P. 319,841, 4.7.28).—In the cuprammonium stretch-spinning process, the coagulant liquor at ordinary temperature is flashed through a high vacuum to remove free oxygen, and a definite amount of oxygen is then re-introduced in accordance with the denier and number of the filaments being spun.

F. R. ENNOS.

Production of composite cooking liquor. L. BRADLEY and E. P. MCKEEFE, Assrs. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,702,587, 19.2.29. Appl., 2.3.25).—Residual liquor from the cooking of wood with a liquor containing sodium sulphite is calcined under such conditions that neither oxidation nor reduction can take place.

F. G. CROSSE.

Manufacture of substances resembling cork. I. G. FARBENIND. A.-G. (B.P. 300,207, 6.11.28. Ger., 8.11.27).—Cellulose is esterified with "estolidic acids" (inner condensation products of hydroxylated higher fatty acids, *e.g.*, triricinoleic acid) or mixtures of these with higher fatty acids. The products contain 1.5–6% of cellulose residue and are substitutes for cork. An example is the ester from linters (10 pts.), lauryl chloride (35 pts.), and triricinoleyl chloride (10 pts.) heated in chlorobenzene and pyridine at 125–135°.

C. HOLLINS.

Manufacture of wrapping material. F. K. WICKEL (B.P. 294,463, 18.4.28. Ger., 23.7.27).—Transparent films of cellulose derivatives may be rendered iridescent on one or both sides with metallic mirror-forming materials, *e.g.*, oxychlorides of antimony or bismuth; by further coating the metallic mirrors with solutions of suitable resins or other film formers, iris effects are produced.

F. R. ENNOS.

Manufacture of artificial light filters. C. HOLUB (U.S.P. 1,730,574, 8.10.29. Appl., 13.7.26. Austral., 24.7.25).—See B.P. 265,455; B., 1927, 295.

Production of cellulose from highly-lignified plants. R. RUNKEL, Assr. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,731,112, 8.10.29. Appl., 11.5.27. Ger., 20.5.26).—See B.P. 296,547; B., 1928, 810.

Non-deliquescent product from sulphite[-cellulose] waste liquor. P. ONNERTZ, H. WESCHE, and K. BRODERSEN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,731,433, 15.10.29. Appl., 12.11.26. Ger., 24.1.24).—See F.P. 592,119; B., 1926, 188.

Manufacture of artificial silk and like threads. C. W. PALMER and W. WHITEHEAD, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,731,317, 15.10.29. Appl., 29.1.23. U.K., 18.2.22).—See B.P. 198,023; B., 1923, 765 A.

Treatment of cellulose prior to esterification. H. DREYFUS (U.S.P. 1,731,299, 15.10.29. Appl., 16.1.26. U.K., 24.5.22).—See B.P. 207,562; B., 1924, 129.

Manufacture of mixed esters of cellulose. KODAK, LTD., Assces. of H. T. CLARKE and C. J. MALM (B.P. 290,570, 4.5.28. U.K., 14.5.27).—See U.S.P. 1,690,621; B., 1929, 554.

Softening of paper. K. H. MEYER, J. MÜLLER, and U. HOFFMANN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,731,679, 15.10.29. Appl., 7.1.28. Ger., 7.1.27).—See B.P. 292,430; B., 1928, 638.

Process and apparatus for degumming, washing, and drying of fibres. R. L. PRITCHARD (B.P. 319,594, 14.6.28).

Machines for spinning artificial silk. J. P. BEMBERG A.-G. (B.P. 297,417, 25.8.28. Ger., 21.9.27).

[Spinning] apparatus for manufacture of artificial silk. H. WADE. From SYNTHETA A.-G. (B.P. 320,417, 14.7.28).

Bobbins for use in preparation of artificial silk from viscose. J. C. HARTOGS (B.P. 319,888, 20.8.28).

Manufacture of resin-coated pulp-board. G. M. CLARK. From AGASOTE MILLBOARD Co. (B.P. 319,901, 31.8.28).

Chemicals from wood-pulp waste-liquor (U.S.P. 1,728,252). Mineral fibres (U.S.P. 1,730,609).—See VII. Cellulosic filament (U.S.P. 1,730,417).—See XI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Mercerisation of cellulose. F. I. BOGOYAVLENSKI (J. Appl. Chem. Moscow, 1929, 2, 167–178).—The quantity of sodium hydroxide absorbed by cellulose at 20–22° increases with concentration up to 4–5*N*; for 5–8*N* the quantity absorbed corresponds with the formula $(C_6H_{10}O_5)_4 \cdot 2NaOH$, and for 9–10*N* with $(C_6H_{10}O_5)_3 \cdot 5NaOH$. The rate of reaction is maximal at 5*N*. Absorption of potassium hydroxide increases rapidly up to 5*N*, and thereafter slowly. With 10*N*, the compound $(C_6H_{10}O_5)_4 \cdot 2KOH$ is probably formed.

CHEMICAL ABSTRACTS.

Hair-dyeing. MEYER.—See IV.

See also A., Nov., 1254, **Photographic determination of coloured solutions** (LYTHGOE and TANSLEY). 1262, **Scopometer** (EXTON). 1298, **Colour and constitution** (HODGSON).

PATENTS.

Preparation of effect threads. E. F. EHRHARDT. From CHEM. WORKS, FORMERLY SANDOZ (B.P. 318,366, 7.8.28).—The resistance of cellulose mono- and diacetates to direct cotton colours is increased by steaming under pressure and even an incompletely monoacetylated yarn can thus be rendered completely resistant. When coloured cotton is acetylated, the dye may be fixed by steaming.

C. HOLLINS.

Treatment of fabrics. [Effects on mixtures of cellulose esters and animal fibres.] BRIT. CELANESE, LTD., and G. RIVAT (B.P. 302,592, 17.12.28. U.S., 16.12.27).—Mixed fabrics such as velvets having a cellulose acetate pile on a silk or wool back are printed with mineral acids or their acid salts (sulphuric acid, sodium hydrogen sulphate); the pile is locally destroyed and may be removed by subsequent neutralisation and brushing. A solvent, softener, or swelling agent for the cellulose ester may advantageously be added to the printing paste.

C. HOLLINS.

Treatment of fibrous material. SPICERS, LTD., Assces. of H. FRIEDLAENDER (B.P. 290,625, 24.4.28. Ger., 18.5.27).—The pulp of fibrous material is impregnated with a quantity of emulsified montan wax, previously heated at 230–250° while air is blown through, which is equivalent to 1–10% or considerably less than 30% of the dry weight of the fibre; a wax solvent (carbon tetrachloride) and sizing materials (casein, resins, fats, oils, or soaps) may also be added, the whole being precipitated on the fibre by the addition of alum or aluminium sulphate.

F. R. ENNOS.

Colour printing pastes and their manufacture. E. I. DU PONT DE NEMOURS & Co. (B.P. 285,041, 9.2.28. U.S., 9.2.27).—See U.S.P. 1,705,818; B., 1929, 716.

Treatment of vegetable fibrous materials [to give wool-like effects]. G. HEBERLEIN, Assr. to HEBERLEIN PATENT CORP. (U.S.P. 1,731,245, 15.10.29. Appl., 28.1.28. Ger., 4.2.27).—See B.P. 284,686; B., 1928, 332.

Weighting fibres of cellulose derivatives. C. DREYFUS and H. PLATT, Assrs. to CELANESE CORP. OF

AMERICA (U.S.P. 1,731,298, 15.10.29. Appl., 24.10.25).—See B.P. 260,290; B., 1928, 477.

Machines for dyeing of textile fabrics. C. CALLEBAUT and J. DE BLICQUY (B.P. 320,544, 5.11.28).

Washing, bleaching, and like machines. BRIT. LAUNDERERS' RES. ASSOC., R. G. PARKER, D. N. JACKMAN, and R. E. V. HAMPSON (B.P. 320,102, 3.7.28 and 23.4.29).

Treatment of artificial silk threads and filaments. J. BRANDWOOD (B.P. 320,025, 22.3.28).

Fluid treatment of artificial silk threads and filaments. J. BRANDWOOD (B.P. 319,848, 6.7.28).

Treatment with liquid of fibres wound on permeable spools. I. G. FARBENIND. A.-G. (B.P. 294,548, 24.7.28. Ger., 25.7.27).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Desulphurisation of burnt pyrites. C. FICAI and P. PIACENTINI (Giorn. Chim. Ind. Appl., 1929, 11, 349—353).—Desulphurisation of burnt pyrites (cf. B., 1929, 21) takes place wholly in the combustion chamber, especially where the temperatures are higher. At the time of its elimination the sulphur is present entirely as sulphates, the dissociation of which is greatly retarded by their low concentration. The large excess of air maintained in the furnace helps neither the desulphurisation nor the agglomeration of the pyrites, since these are enhanced by a low concentration of the oxygen. Originally tunnel furnaces were furnished with a pipe which drew off a part of the gases before they passed to the chimney for the purpose of blowing the flame in the combustion chamber, but such arrangement appears to have been discarded in Cokerill furnaces owing to the difficulty of working the furnaces under these conditions; in view of diminished chimney losses, the authors suggest that it might be of interest to give further consideration to its use. T. H. POPE.

Determination of calcium oxide in quicklime. H. A. J. PIETERS and J. H. VAN DER HEIJDEN (Chem. Weekblad, 1929, 26, 511—512).—Direct titration gives very low results, owing to the slowness with which the oxide dissolves. The phenol and ammonia methods also give low results, though the error is less; the ammonia distillation method is more accurate still, but again gives low results. Boiling with excess of standard acid and back-titration, combined with a carbonate determination, gives quite accurate results. S. I. LEVY.

Alumina from volcanic ash as a source of aluminium. I. SEKI (Rep. Imp. Ind. Res. Inst. Osaka, 1929, 10, No. 2, 22 pp.).—Volcanic ash is dried at 130°, powdered, heated for 2 hrs. at 400—700°, and treated with sulphuric acid (*d* 1.26) for 30 min. Aluminium sulphate is best separated from calcium sulphate when the solution has *d* 1.47. Iron may be removed with ferrocyanide. The product contains Al_2O_3 99.08, H_2SiO_3 0.86, $\text{Fe}(\text{OH})_3$ 0.06%. CHEMICAL ABSTRACTS.

Grain growth in silver halide precipitates. S. E. SHEPPARD and R. H. LAMBERT (Coll. Symp. Mon., 1928, 6, 265—282).—In the first stage of digestion certain

grains grow by accretion from solution and others dissolve; growth due to recrystallisation within the aggregates produced by collision and coalescence then occurs. CHEMICAL ABSTRACTS.

Determination of water-soluble phosphate. SUCHIER.—See XVI.

See also A., Nov., 1229, Absorption of carbon dioxide by sodium hydroxide (HATTA). 1247, Production of nitric oxide in the electric arc (BRINER and RIVIER). 1250, Preparation of pure cupric sulphide (FISCHBECK and DORNER). 1251, Ultramarine (HOFFMANN). 1253, Preparation of hydrogen sulphide (GELLER and SCHAEFER). 1255, Potentiometric titration of acids and bases (KAHLENBERG and KRUEGER). Microanalysis of acids (FEIGL and KRUMHOLZ). Determination of perchlorate (FEDOROVA). 1256, Micro-determination of sulphur dioxide in air (RÖTTINGER). 1336, Colloidal lead (GANASSINI).

PATENTS.

Manufacture of sulphuric acid. H. F. MERRIAM, ASS. to GEN. CHEM. CO. (U.S.P. 1,728,213, 17.9.29. Appl., 29.4.26).—In the contact process, compressed air is used for the oxidation to sulphur dioxide of material containing sulphur. L. A. COLES.

Production of concentrated nitric acid from dilute solutions. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,125, 7.7.28).—Dilute nitric acid of not less than 50% concentration, or residual liquor from the nitration of carbohydrates, dyes, intermediates, etc., is treated in a closed vessel with liquid nitrogen peroxide at 30—60° (preferably above 40°); after the mixture has separated into layers, the nitrogen peroxide layer, which contains nitric acid in solution, is withdrawn and the nitric acid is recovered by fractional distillation or by cooling to about 5°, when the solution again settles into layers the lower of which yields almost pure nitric acid on evaporation of the dissolved peroxide. L. A. COLES.

Recovery of nitric acid [from solutions containing other volatile acids]. I. G. FARBENIND. A.-G. (B.P. 303,351, 10.12.28. Ger., 21.12.27).—Solutions containing nitric acid and, e.g., hydrochloric acid are treated with gaseous reducing agents, e.g., sulphur dioxide, preferably in quantity sufficient to reduce the nitric acid to nitric oxide which, after scrubbing with water and, e.g., milk of lime to remove hydrochloric acid etc., is oxidised to nitric acid or to nitrates and nitrites. Hydrochloric and sulphuric acids are recovered from the residual liquor. L. A. COLES.

Manufacture of alkali nitrates. I. G. FARBENIND. A.-G. (B.P. 303,355, 18.12.28. Ger., 31.12.27. Adn. to B.P. 303,351; preceding).—Alkali (potassium) sulphate or chloride either separately or in admixture is treated at 35° with nitric acid (*d* 1.20). By cooling to —5° and filtering or centrifuging, a product containing 95% of potassium nitrate is obtained, which may be further purified by purging with a saturated solution of potassium nitrate. The mother-liquor may be freed from the remaining nitric acid by the process of the prior patent, and then distilled to separate hydrochloric acid (as 20% acid). The residue, on cooling, yields

potassium bisulphate, which, suitably mixed with alkali chloride, may be re-treated with nitric acid as above.

P. E. L. FARINA.

Manufacture of ammonium salts. "MONTECATINI," SOC. GEN. PER L'IND. MINERARIA ED AGRICOLA, ASSocs. of G. FAUSER (B.P. 292,129, 13.6.28. It., 14.6.27).—Sulphuric acid is sprayed into a chamber containing ammonia, and ammonium sulphate is removed from the lower end of the chamber. Automatic means operated by the pressure in the chamber regulate the supply of acid, which is of such concentration that the water is evaporated completely by the heat of reaction, and the steam is removed subsequently from the chamber by circulation through an external cooler and rectifier, which serves also for generating the ammonia when an aqueous solution of it is used as the source of supply.

L. A. COLES.

Recovery of chemicals [sodium carbonate free from sulphide from wood-pulping waste liquor]. F. G. RAWLING (U.S.P. 1,728,252, 17.9.29. Appl., 4.2.27).—The ash obtained by concentrating and then incinerating the liquor is treated with air and steam under conditions such that a portion of the free carbon burns and thereby converts sodium sulphide into the carbonate with the liberation of hydrogen sulphide. Sodium carbonate is extracted from the product.

L. A. COLES.

Manufacture of synthetic mineral fibres. M. M. GROSSMAN (U.S.P. 1,730,609, 8.10.29. Appl., 17.10.27).—Sodium silicate is boiled for several hours under pressure with a solution of caustic soda, the resulting solution is concentrated and spun into fibres, and these are hardened by passing through a bath containing calcium chloride solution.

H. ROYAL-DAWSON.

Electrolytic revivification of potassium ferrocyanide formed in the purification of gases by oxidation of hydrogen sulphide with potassium ferricyanide. R. BRANDT (B.P. 319,147, 4.10.28).—The spent liquor, after removal of the coarser particles of sulphur by sedimentation, is subjected to anodic oxidation by being passed upwards through a cell containing upright iron electrodes with interjacent ribs to increase the surface, the potassium hydroxide formed during electrolysis being converted into the carbonate by the addition of potassium bicarbonate.

L. A. COLES.

Purification of sodium glutamate. K. OKA (B.P. 290,657, 2.4.28).—Iron compounds are precipitated by concentrating sodium glutamate solutions in the presence of sodium sulphide.

L. A. COLES.

Technically pure CrO_3 [chromium trioxide]. METALS PROTECTION CORP. (B.P. 307,061, 18.2.29. U.S., 3.3.28).—Chromium trioxide prepared by the usual method, washed, if desired, with sulphuric acid to remove sodium hydrogen sulphate, and heated after filtration to remove the greater part of the adhering mother-liquor, is mixed with the quantity of barium hydroxide, carbonate, or chromate necessary to precipitate residual sulphuric acid as barium sulphate on dissolution of the product in water.

L. A. COLES.

Manufacture of phosphorus pentasulphide. P. DUTOIT (B.P. 301,500, 22.11.28. Switz., 1.12.27).—

Sulphur vapour is made to react with ferrophosphorus (Fe_2P) at a temperature below that of fusion by heating a mixture of pyrites and ferrophosphorus (22% P) at 700–800°; the phosphorus pentasulphide evolved is then condensed.

P. E. L. FARINA.

Recovery of selenium. D. L. OGDEN and R. E. VALENTINE, ASSrs. to UNITED STATES METALS REFINING CO. (U.S.P. 1,730,681, 8.10.29. Appl., 9.5.28).—Sulphur dioxide, free from hydrochloric acid, is introduced into the hot solution containing selenium to precipitate the selenium as a black amorphous powder, which is collected, ground in a wet state, washed, and dried.

H. ROYAL-DAWSON.

Purification of gases. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, ASSocs. of SOC. CHIM. DE LA GRANDE PAROISSE, AZOTE & PROD. CHIM. (B.P. 307,039, 6.6.28. Fr., 2.3.28).—The catalyst chamber for the pretreatment of gases for use in synthetic reactions, e.g., for the reduction of oxygen and carbon monoxide in nitrogen-hydrogen mixtures for use in the synthesis of ammonia, surrounds the main reaction chamber so that the purifying process is facilitated by the heat generated in the main reaction. Means may be provided between the two chambers for removing the water etc. formed in the first chamber.

L. A. COLES.

Oxidation of potassium ferrocyanide to potassium ferricyanide. R. BRANDT (U.S.P. 1,732,117, 15.10.29. Appl., 16.10.28. Ger., 18.11.27).—See B.P. 319,147; preceding.

Decomposition of complex hydrofluoric salts. M. BUCHNER, ASSr. to A. F. MEYERHOFER (U.S.P. 1,730,915, 8.10.29. Appl., 23.3.26. Ger., 24.3.25).—See B.P. 249,860; B., 1927, 748.

Molybdenum phosphotungstate compounds. P. RABE, B. WENK, and E. HARTMANN, ASSrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,731,081, 8.10.29. Appl., 9.5.27. Ger., 14.7.26).—See B.P. 292,253; B., 1928, 603.

Extraction of hydrogen from gaseous mixtures. G. CLAUDE, ASSr. to L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (U.S.P. 1,730,805, 8.10.29. Appl., 5.3.25. Fr., 8.3.24).—See B.P. 230,413; B., 1925, 356.

Granular solids from viscous liquids (B.P. 319,893). **Acid-concentrating towers** (B.P. 319,839).—See I. **Sulphur from gas** (B.P. 297,062).—See II. **Treatment of gypsum** (B.P. 298,607).—See IX. **Fertilisers** (B.P. 305,132, 318,842, and 318,909).—See XVI.

VIII.—GLASS; CERAMICS.

Soda-zinc glasses. K. FUWA, S. SAKAI, M. SATO, and F. SUZUKI (J. Japan. Ceram. Assoc., 1928, 36, 135–153, 243–264).—The influence of chemical composition of glasses $0.5\text{--}2.0\text{Na}_2\text{O}, 0\text{--}6\text{ZnO}, 6\text{SiO}_2$ on melting, weathering, devitrification, workability, effect of reheating, coefficient of expansion, elongation, thermal endurance, and resistance to chemicals was examined. In the pull test the rate of elongation of $y\text{Na}_2\text{O}, x\text{ZnO}, 6\text{SiO}_2$ increases as y increases, the increase being diminished as x increases. The thermal endurance is not greatly influenced by variation in x if y is small,

whilst it is reduced by increase in x when y is large. The glasses are very resistant to water so long as x is large; the resistance is not much influenced by the proportion of x and y . The glasses $0.75\text{Na}_2\text{O}, 1.0-2.0\text{ZnO}, \text{SiO}_2$ are most resistant. The resistance to hydrochloric acid is greatly influenced by variation in x and y . The glass $1.0\text{Na}_2\text{O}, 1.25\text{ZnO}, 6\text{SiO}_2$ was the least soluble. The glasses are not highly resistant to sodium hydroxide solution; the solubility in sodium carbonate solution is not small.

CHEMICAL ABSTRACTS.

Coating and welding of glass. S. MURAKAMI (J. Japan. Ceram. Assoc., 1928, 36, 303—311).—For coating, the difference in coefficient of expansion should not be greater than 60×10^{-6} , the glass with the larger coefficient being used on the outside.

CHEMICAL ABSTRACTS.

Progress report on investigation of fireclay bricks and the clays used in their preparation. R. A. HEINDL and W. L. PENDERGAST (J. Amer. Ceram. Soc., 1929, 12, 640—675).—The investigation was carried out on 17 makes of fireclay bricks, 14 raw mixtures, 26 fireclays, and 3 samples of crushed quartz and silica sand. Chemical analyses and the results of a number of physical tests on clays and the bricks made therefrom are given. The thermal expansions of the fireclays at 1400° and of the firebricks after reheating them at 1400° , 1500° , and 1600° were determined, and the materials were classified according to the type of expansion. The modulus of elasticity and the transverse breaking strength were determined on the bricks at several temperatures, and also after the bricks had been subjected to different heat treatments. The thermal expansions of most bricks decreased as the firing temperature increased. The thermal expansion of a finished brick was not necessarily the average of the expansions of the clays of which it was composed. The modulus of elasticity was generally greater at 550° than at 20° , and at 1000° it was less than at either of these temperatures. The resistance to spalling decreased with increase in modulus of elasticity and increase of thermal expansion. Large variations in the number of quenchings required to cause spalling in bricks of the same brand were observed, indicating considerable differences in the physical properties. No relation was observed between the method of making the brick and its thermal expansion, but there was a direct relation between expansion and resistance to spalling. Spalling-resistance is expressed as an empirical relation, varying directly as the modulus of rupture and grog content and inversely as the modulus of elasticity and coefficient of expansion.

F. SALT.

Effect of various addition agents and treatments on the green strength of Missouri refractory clays. A. J. PAUL and M. E. HOLMES (J. Amer. Ceram. Soc., 1929, 12, 676—686).—The effects of ageing, varying the colloidal content, and developing bacterial growth on plastic and semi-flint clays and diaspore were investigated. Small additions of bentonites produced a substantial increase in the strength of bodies. The strength of a material of low plasticity was greatly increased by small additions of a highly colloidal clay. Little effect was produced by treating plastic fireclay

with steam under pressure or by adding sodium silicate. The deflocculating action of sodium carbonate and of tannic acid was moderately effective. Organic growth, induced by grape juice, had a marked effect on the strength, but many months were required to produce the same effect as bentonite. Negative results were obtained with salt and aluminium chloride. Additions of 0.5% of dextrin had a considerable effect. The "P.C.E. value" was reduced by about 1 cone per 3% addition of a bentonite.

F. SALT.

Jointing cements. EDWARDS.—See IX.

See also A., Nov., 1257, **Detection of alkali metal salts in silicates** (TANANAEV).

PATENTS.

Manufacture of glass. H. GEORGE (B.P. 299,393, 23.10.28. Fr., 25.10.27).—In a development of an electrical method of melting (cf. B.P. 279,818; B., 1928, 823) glass is placed directly in contact with, and is heated by, an electrically heated mass of molten tin. Additional tin may be added from an auxiliary chamber when melting is complete in order to facilitate fining or regulate the surface height of the glass. A. COUSEN.

Manufacture of composite or non-splintering glass. J. E. ALCOCK (B.P. 319,873, 31.7.28).—Resinous products obtained by the reactions between phthalic anhydride and glycerol, phthalic anhydride, glycerol, and succinic acid, or formaldehyde and phenols, dissolved in suitable solvents, are employed for uniting glass to celluloid etc. Nitrocellulose, dissolved in a solution of the mixed resins, assists cohesion.

A. COUSEN.

[Tunnel] kilns and the like. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 319,840, 3.7.28).—A form of kiln is described in which the heating is performed by laterally placed combustion-chamber units, constructed from highly-conductive refractory material such as silicon carbide bricks, the products of combustion then being discharged into the tunnel. Cooling is performed beyond the firing zone by passing the ware between walls of similar material, behind which are located passages for the circulation of air.

A. COUSEN.

Clouding of enamels. I. KREIDL (B.P. 297,724—5, 23. and 24.1.28. Austr., 26.9.27).—Clouding of enamels is effected by the production of finely-distributed gas bubbles which are evolved during the firing of the enamel from (A) organic substances or substances containing volatile organic compounds, or from (B) inorganic substances which are added in the enamel mill. The clouding action may be varied by altering the permeability of the enamel batch, e.g., by varying the addition of fluorides to the enamel. The clouding medium may contain a small portion of non-volatile matter which cannot of itself produce the clouding effect in the absence of gas bubbles. The clouding medium may produce its effect either directly or by interaction with other substances.

F. SALT.

Furnace [for refractories etc.]. R. MALVOS and M. CROZEMARIE (U.S.P. 1,731,901, 15.10.29. Appl., 14.11.24. Fr., 11.11.23).—See B.P. 248,036; B., 1926, 408.

Manufacture of glass-melting crucibles and similar refractory objects of clay. J. HOCHHUT (U.S.P. 1,732,088, 15.10.29. Appl., 22.12.27. Ger., 6.11.25).—See B.P. 291,305; B., 1928, 524.

Forehearth of glass-melting furnaces. BRIT. HARTFORD-FAIRMONT SYND., LTD., and T. WARDLEY (B.P. 320,334, 8.5.28).

IX.—BUILDING MATERIALS.

Waste heat in cement mills and paper mills. H. B. SMITH (Proc. Eng. Soc. W. Penn., 1929, 45, 269—296).—The use of waste-heat boilers in connexion with cement kilns in America dates from about 1910; in 1928 25% of the country's kilns were so equipped. A test in 1923 with entering gases at 660° showed a heat recovery of 68%. The steam produced may average 3.5 lb. per lb. of coal fired, and should suffice for the power requirements of the plant. The life of fans varies from 3 weeks to 3 years. Wear is promoted by inward air leakage on the train, which causes excessive dust. The draught loss through friction in the boiler must be minimised by special designing. A newer use of the waste-heat boiler is in connexion with the combustion of dehydrated black liquor from the sulphate-pulp process. This combustion is carried out primarily to recover the chemical content of the liquor. The gases deposit a clinker consisting largely of sodium salts for which provision must be made. Discussion on this paper deals principally with fan design, the risks attached to direct firing to supplement a waste-heat installation, and the possibility of sale of surplus power to a power company to be bought back when required.

C. IRWIN.

Distribution of particles of cements. K. FUJII (J. Jap. Ceram. Assoc., 1928, 36, 46—57).—Fractions of different fineness were determined. The whole cement and the fraction consisting of particles of diameter less than 18 μ contained, respectively: SiO_2 21.10, 20.42; Al_2O_3 6.01, 6.21; Fe_2O_3 2.29, 2.17; CaO 63.90, 61.60; MgO 1.03, 0.91; SO_3 1.53, 1.85; loss on ignition 3.01, 6.50%.

CHEMICAL ABSTRACTS.

Japanese cement. V.—VIII. S. NAGAI (J. Jap. Ceram. Assoc., 1928, 36, 104—113, 265—275, 440—448, 475—483).—A comparative study of recent products.

CHEMICAL ABSTRACTS.

Action of acid on cement mortar. S. NAGAI (J. Jap. Ceram. Assoc., 1928, 36, 382—394).—The effect of sulphuric and hydrochloric acids on the tensile strength of cements has been studied. CHEMICAL ABSTRACTS.

Ferruginous cements. O. REBUFFAT (Giorn. Chim. Ind. Appl., 1929, 11, 353—355).—The characters of Portland ferro-cement are described. T. H. POPE.

Jointing [refractory] cements. C. EDWARDS (Trans. Ceram. Soc., 1929, 28, 405—413).—The properties of refractory cements for use with silica bricks and firebricks are outlined. The chief raw materials used in making these cements are silica rock, silica brick, raw fireclay, and fireclay grog; and the usual bonding materials are ball-clay, lime, Portland cement, sodium silicate, and ground glass. For certain purposes carborundum is a useful constituent. To establish the

quality of a cement it should be tested for the following properties: texture and working qualities, grain size, tensile strength, refractoriness, shrinkage, adherence, and spalling. The effect of long usage on cements is discussed. F. SALT.

PATENTS.

Treatment of gypsum. C. GAMARRA (B.P. 298,607, 29.9.28. U.S., 12.10.27).—An alkaline-earth (calcium) chloride and an organic acid yielding an insoluble calcium salt, *e.g.*, tartaric acid, are added to gypsum containing carbonates so that on mixing the product with water carbon dioxide is evolved slowly and a hard, porous mass is obtained on setting. L. A. COLES.

Manufacture of cement from slag. J. G. BERGQUIST (U.S.P. 1,731,189, 8.10.29. Appl., 18.5.25).—Molten slag is transferred to a furnace and to it is added sufficient lime to form dicalcium silicate. After cooling and allowing it to disintegrate to a powder, it is mixed with powdered limestone, wetted, ground, and burned to form a clinker, which is subsequently ground.

F. G. CROSSE.

Manufacture of constructional materials. MUSAG GES. F. DEN BAU VON MÜLL- & SCHLACKEN-VERWERTUNGSANLAGEN, A.-G., and A. GROTE (B.P. 298,159, 2.10.28. Ger., 4.10.27).—Mixtures of hard-sintered refuse slags with bituminous binders are compressed into blocks.

L. A. COLES.

Heat non-conducting composition [in paste form]. R. BOWMAN & Co., LTD., and J. W. CRAGGS (B.P. 319,648, 22.8.28).—The product, which dries and hardens after application, is prepared by adding hot sodium oleate solution to hot bitumen with continued stirring and, after coagulation of the emulsion, incorporating powdered talc, Italian powder, and colouring constituents.

L. A. COLES.

Production of porous building materials from mineral binding media. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,103; 26.7.28).—Portland cement etc. is beaten up to a frothy pulp with hot or cold, dilute, aqueous solutions containing wetting agents comprising sulphonated organic compounds containing at least six carbon atoms in the molecule, *e.g.*, Turkey-red oil, or sulphonated tall oil, higher fatty acids, mineral or tar oils, alkylated aromatic hydrocarbons, and the products are cast in moulds.

L. A. COLES.

[Bituminous] lining or coating of pipes, tubes, etc. CLAYTON, SON & Co., LTD., and W. SOWDEN (B.P. 320,123, 7.7.28).—The pipes etc. are coated at 220—230° with a mixture of bituminous material, *e.g.*, "B₁ mexphalte," and about 10% of medium wool-fat pitch or fatty acid pitch blended at about 170° and subsequently incorporated with finely-divided carbonaceous material.

L. A. COLES.

Protective coatings for metal, wood, or other surfaces. J. DE B. W. GARDINER (B.P. 320,160, 23.7.28).—The surfaces are coated with an asphalt emulsion prepared with the use of clay as emulsifying agent; the emulsion, before it has set, is dusted over with a dry, powdered cement, *e.g.*, Portland cement.

L. A. COLES.

Apparatus for treating wet raw materials in the manufacture of cement. J. S. FASTING (U.S.P. 1,731,457, 15.10.29. Appl., 30.11.23. Denm., 22.12.22).—See B.P. 227,977; B., 1925, 232.

Bituminous emulsions (B.P. 319,101).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Cleaning of blast-furnace gas. The stream-line dry gas filter. R. M. COLLES (Iron & Steel Inst., Carnegie Schol. Mem., 1929, 18, 53—59).—Some experiments on the removal of dust from air by a small stream-line filter are recorded, and suggestions are made for the construction of a larger experimental filter for cleaning blast-furnace gases. Based on the results of the laboratory tests, such a filter plant covering the same ground space as a Halberg-Beth plant but being 4 ft. less in height should handle 29% more gas at 100° or the same volume of gas at 210° that the bag-plant will handle at 100°. A 6-ft. pack stream-line filter with a pressure difference between the outside and inside of 3 in. should handle 390 cub. ft./hr. or with a pressure difference of 7 in. 910 cub. ft./hr. Provided the dust is kept dry by operating the filter above 100° no clogging should occur once equilibrium has been established, and the dust should fall off the outside from time to time in the form of a cake, provided that the coarser particles were not removed as in present-day practice. A. R. POWELL.

Economics of modern dry-cleaning processes for blast-furnaces gas. M. ZILLEN (Stahl u. Eisen, 1929, 49, 1441—1444).—Detailed cost data are given for a Halberg-Beth filter plant and an electrical gas-purification plant working on the same type of blast-furnace gas. The power consumption averages 2.712 kw.-hrs./1000 m.³ of gas in the first case and 3.065 kw.-hrs. in the second case; the total cost, including overhead charges, is about 12% greater in the second than in the first-named plant. From theoretical considerations it is shown that it is not only advantageous but also economical to cool the purified gases before using them for heating purposes. A. R. POWELL.

Cast iron. IV. Influence of melting temperature on low-carbon cast iron. K. TANIMURA (Bull. Tech. Coll. Kyushu, 1928, 3, 266—276).—Iron containing 3% C and 1.19—2.5% Si was melted and slowly solidified. For high temperatures the degree of graphitisation is relatively small. The matrix of specimens melted at a high temperature was relatively heterogeneous. CHEMICAL ABSTRACTS.

Influence of structure of cast iron on changes due to high temperature. A. LE THOMAS (Compt. rend., 1929, 189, 639—641).—The differences in behaviour of a series of 10 bars of cast iron (C 3.34, Si 1.51, Mn 0.57, S 0.07, P 0.27%) of diameters varying from 13.0 to 100.5 mm., but otherwise similar, due to heat treatment have been examined. The Brinell hardness, expansion due to a cycle reaching 850°, and amplitude and temperature (this last but slightly) of the Ar transformation all steadily diminish as the thickness of the bars increases, whilst the effect of the fusion of the phosphorus eutectic is perceptible at a lower tem-

perature (960°) for a thin bar than (1020°) for a thick one. The Ac point remains unchanged throughout, and the temperature of graphitisation after rising steadily to that of the Ac point for a bar of 30 mm. diam. thereafter remains constant. Thus a more slowly cooling thick bar is more stable than a quicker cooling thin one. C. A. SILBERRAD.

Sulphur in cast iron. K. HASEGAWA (Iron and Steel, Japan, 1928, 14, 1072—1083).—In white cast iron sulphur exists chiefly as ferrous and manganous sulphides; in grey cast iron most of the sulphur is present as manganous sulphide, the remainder existing as ferrous sulphide when the manganese content exceeds 0.5%. When the iron contains less than 0.5% Mn, and the silicon content is high, part of the sulphide is insoluble in acid; the insoluble compound is probably an unstable compound formed by interaction between iron, sulphur, iron carbide, and iron silicide. CHEMICAL ABSTRACTS.

Effects of nickel and chromium on cast iron. I, II. D. HANSON (Metallurgist [Supp. Engineer], 1929, 147, 38—40, 56—60).—With increasing amounts of nickel (up to 20%) the Brinell hardness is maximal at 2% Ni, minimal at 3% Ni, subsequently rises slightly, and then declines. With silicon one maximum is observed. The joint effect is essentially the sum of the individual effects. Silicon and nickel decompose, but chromium stabilises, free carbide in grey iron. CHEMICAL ABSTRACTS.

Effect of service on endurance properties of rail steels. J. R. FREEMAN, JUN., and H. N. SOLAKIAN (Bur. Stand. J. Res., 1929, 3, 205—246).—Tests have been carried out to compare the endurance properties of steel rails before and after service. In one case after being subjected to 20,000,000 tons of traffic no change in the endurance properties was found, and it is concluded that the rails were not subjected in service to stresses exceeding the endurance limit. In another case an appreciable reduction in endurance limit was found with rails subjected to 12,000,000 tons of traffic. In this case a greater spread in the values for the endurance limit of different specimens, both before and after service, was found, and fatigue is attributed to the presence of impurities acting as fatigue nuclei. The necessity for improving the soundness of rail steels is emphasised, rather than reducing wheel loads which are below the endurance limit for sound rail steels. C. J. SMITHELLS

Low-expansion nickel steel. T. F. RUSSELL (Engineering, 1929, 128, 400—402).—The uses of a steel containing 36% Ni, known commercially as "Nilex" and generally used for clock pendulums, standard tapes, etc., have been extended to engineering parts. Although this alloy is used chiefly on account of its low coefficient of expansion ($(2-0.3) \times 10^{-6}$), the mechanical properties are comparable with those of a 0.3% carbon steel, though previous heat treatment influences both the mechanical properties and the expansion coefficient, which remains low only throughout a comparatively small range of temperature. The harder the alloy the lower is the temperature range during which there is a constant coefficient of expansion, and the lower this coefficient the more easily is it increased by heating the alloy. Alloys having a very low coefficient cannot be

heated to 100° without increasing the coefficient, though this is not liable to alter in an indefinite period if the alloys are maintained within the range of probable constancy of coefficient. C. A. KING.

Influence of the use of sponge iron on the properties of steel. W. ROHLAND (Stahl u. Eisen, 1929, 49, 1477—1485).—The mechanical and working properties of soft iron and steel made from sponge iron have been determined, using charges of 150—250 kg. melted in arc, induction, and crucible furnaces, and the results are shown in numerous tables and graphs. Direct melting of sponge iron yields a product equal to Lancashire soft iron, and mild steel made from it is free from ageing faults even when only 0.03% Mn is present. Tool steels can be made directly from sponge iron without addition of a deoxidising agent, and with only 0.02% Mn or Si they have a good surface hardness and a tough core. Owing to the high purity of the steel made in this way the pearlite can be readily transformed into granular cementite, the rate of diffusion of carbon in pure steel being very high. Alloy tool steels from sponge iron are much tougher than those made from pig iron, and less liable to develop faults during heat treatment. Steels for clock springs and cutting tools made from sponge iron are superior to those made of Swedish iron. For steel wires alloys with 0.85—0.9% C may be used in place of the usual steels with 0.7% C as they have a tensile strength 20—30% higher with an equal ductility and bending strength. Microscopically and analytically little difference can be found between steel made from pig iron and the same steel made from sponge iron.

A. R. POWELL.

Improving the toughness of high-speed tool steel by carbide annealing. R. HOHAGE and R. ROLLETT (Arch. Eisenhüttenw., 1929—30, 3, 233—239; Stahl u. Eisen, 1929, 49, 1519—1520).—The effect of hot-rolling and subsequent annealing on the toughness of a high-speed tool steel with 0.72% C, 0.15% Si, 0.30% Mn, 4.49% Cr, 19.6% W, 0.37% Mo, and 1.58% V has been investigated. In the hard-rolled state the Brinell hardness was 652 when the temperature at the end of the rolling was 400° or 900°, and 600 when the final temperature was 700°. Annealing at 850° produced the minimum hardness in all cases, but only in the case of metal rolled at 700° was the maximum bending angle obtained at this temperature; in all cases, however, this angle was small. Maximum toughness was produced by quenching the alloy at 650—750° after annealing at 800°. Micrographic examination showed this behaviour to be due to refining of the primary carbide and to the secondary carbide, originally precipitated in a disperse form, subsequently coalescing with the primary carbide and leaving a purely pearlitic ground-mass.

A. R. POWELL.

Application of spectrographic analysis in steel-works' laboratories. K. KELLERMANN (Arch. Eisenhüttenw., 1929—30, 3, 205—211; Stahl u. Eisen, 1929, 49, 1520—1521).—A review of recent work. The determination of the minor constituents of steels by the comparison of the intensities of "homologous pairs" is briefly described.

A. R. POWELL.

Rapid analysis in the metal foundry. C. TAMA (Z. Metallk., 1929, 21, 342—343).—A review. Rapid electrolytic methods with rotating electrode and spectral-colorimetric analysis save considerable time in the foundry laboratory so that copper, nickel, or iron determinations may be made while the metal is held ready for casting. A table is given showing the extinction values obtained with the Herzfeld-Hoffmann spectral-colorimeter with copper nitrate solutions of concentrations between 2.4 and 7.9 g./litre of copper.

A. R. POWELL.

Converting low-grade [copper] matte at Rio Tinto. H. R. PORRS (Bull. Inst. Min. Met., 1929, Oct., 10 pp.).—A typical Rio Tinto matte contains 44.95% Fe, 21.94% Cu, 23.96% S, 2.1% Zn, and small quantities of lead, bismuth, antimony, nickel, and slag constituents. About 3% of the iron is in the free state; about 75% of the remainder is present as ferrous sulphide, and the remainder as magnetite. The converters used are of the Great Falls type, 12 ft. in diam. with 22 tuyères of 1½ in. diam. made of solid drawn-steel tubing. The magnesite lining is 19 in. thick on the tuyère half and 15 in. on the opposite half, the bricks being set with a plain magnesite and water cement. On starting a new lining the converter is washed out with slag having a high magnetite content so as to form a protective layer for the magnesite lining. The slag produced during blowing has the following average composition: 12.64% SiO₂, 60.77% FeO, 18.61% Fe₂O₃, 3.42% Cu₂S, and 0.51% Cu₂O. In order to keep the temperature to an average of 1400° briquettes of a low-grade precipitate copper (65% Cu) are added from time to time during the blowing. The average production of a converter between one complete lining and another is 15,000—20,000 tons, but the tuyère belt is repaired after every 1200 tons. The average amount (tons) of matte used per blow is 21.4, of flux 4, of copper precipitate 3.16, and of air 3.2; the slag produced is 22 tons and the blister copper 5.04 tons.

A. R. POWELL.

Inverse segregation [in alloys]. W. FRAENKEL and W. GÖDECKE (Z. Metallk., 1929, 21, 322—324).—To study the origin of inverse segregation tests were made on copper-aluminium and copper-zinc alloys by casting in a chill mould, withdrawing the molten central portion through a hole in the bottom of the mould when a crust had formed round the sides, and analysing the crust and determining its sp. gr. In all cases the crust contained more copper than the average composition of the molten metal, hence segregation had taken place in the normal way. On the other hand the sp. gr. of the crust was about 3% less than that of the cast ingot, indicating porosity. These results substantiate the theory that inverse segregation is caused by contraction of the primary crystal skeleton causing voids between the outside of these crystals and the sides of the mould; the low pressure in these voids then sucks part of the inner molten metal through the porous crystal skeleton on the outside of which it solidifies. Thus the outside of the ingot has a lower, and the succeeding layers a higher, content of the constituent of higher m.p. than the interior.

A. R. POWELL.

X-Ray study of castings. I. Perkin's metal. G. SHINODA (Suiyokaishi, 1928, 5, 687—694).—The solubility of tin in the α -solid solution of copper and tin is 14%. The small thermal effect at 577° is caused by transformations of the β -phase. The transformation at 578° for the alloy containing 30—38.4% Sn is a peritectoid reaction, $\beta + \text{Cu}_3\text{Sn} \rightarrow \delta$; another eutectoid reaction, $\gamma \rightarrow \beta + \text{Cu}_3\text{Sn}$, occurs at 637°. The compound Cu_4Sn has no solubility on the copper side. Changes in electrical resistance with temperature were followed.

CHEMICAL ABSTRACTS.

Influence of iron, silicon, and manganese on the ageing of duralumin. K. L. MEISSNER (Z. Metallk., 1929, 21, 328—332).—Tests on duralumin made from the purest aluminium containing less than 0.03% each of silicon and iron have confirmed that silicon is not essential to ageing at the ordinary temperature, magnesium alone imparting these ageing properties to the alloy. Other than that of increasing slightly the tensile strength, addition of silicon to form Mg_2Si has no effect whatever. Iron also is without effect on the ageing of duralumin, except that it increases the absolute tensile strength to a greater extent than silicon. Addition of 0.6% Mn results in a large increase in the tensile strength, and this beneficial action is enhanced by the presence of 0.3% Si, but decreased by 0.3% Fe; the simultaneous presence of 0.3% Si and 0.3% Fe yields an alloy with the same properties as that free from these constituents. The deleterious effect of more than 2% Mg on the ageing of duralumin is ascribed to its action in depressing the solid solubility of copper in aluminium, and it is suggested that the actual function of the small magnesium content of duralumin is to accelerate the separation of CuAl_2 from the super-saturated solution, and to cause this separation to take place at lower temperatures. This theory postulates that the true age-hardening constituent in duralumin is CuAl_2 , the addition of magnesium simply intensifying its action.

A. R. POWELL.

Corrosion of aluminium alloys in superheated steam. L. GUILLET and BALLAY (Compt. rend., 1929, 189, 551—553; cf. B., 1928, 862).—The deterioration of aluminium alloys in superheated steam at 300—350° is due to formation of alumina, and occurs at the junction of the grains. It is influenced by the presence of other metals, though these do not necessarily enter into reaction, and is greatest for the purest samples of aluminium in both still and circulating steam, and least for alpac (13% Si). The rate of corrosion also appears to depend on the thermal and mechanical history of the alloy.

J. GRANT.

Solution potentials of aluminium alloys in relation to corrosion. J. D. EDWARDS and C. S. TAYLOR (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 8 pp.).—In connexion with the development of materials known as "Alclad," consisting of an aluminium alloy coated homogeneously with nearly pure aluminium, measurements have been made of the *P.D.* between pure aluminium and certain of its alloys in salt solutions. The alloys are practically always nobler than the pure metal, and are therefore protected by it from corrosion. This is particularly true for the alloy 17ST, which is a

heat-treated and naturally-aged alloy of the duralumin type. Measurements of the *P.D.* between aluminium or its alloys and brass, copper, iron, steel, zinc, and galvanised iron under similar conditions indicate that aluminium and its alloys are always baser than the other metals except zinc, galvanised iron, and, in some cases, steel. Under the conditions of experiment zinc is definitely baser than aluminium or the alloy 17ST, but there is no definite tendency for either metal to corrode at the expense of the other when in contact.

H. J. T. ELLINGHAM.

Aluminium and its alloys in aircraft. T. W. BOSSERT (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 7 pp.).—The properties of some special alloys are outlined and their particular applications in aircraft construction reviewed.

H. J. T. ELLINGHAM.

Magnesium and its alloys in aircraft. W. G. HARVEY (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 11 pp.).—The properties and applications of magnesium and its alloys are reviewed. Special reference is made to the alloy containing 7% Al and 0.4% Mn, which has a tensile strength of 25,000 lb./in.² as cast, and 33,000 lb./in.² after heat treatment. The presence of the manganese increases resistance to corrosion. Castings are now commercial products and forgings are coming into use. The lightness of magnesium alloys commends them for many aeroplane parts.

H. J. T. ELLINGHAM.

X-Ray differentiation between mechanically and electrolytically produced gold coatings. U. DEHLINGER and R. GLOCKER (Z. Metallk., 1929, 21, 325—326).—X-Ray examination by the Laue-Debye method of the coatings removed from the articles by treatment with hydrochloric acid affords an indication as to whether the article is rolled gold or annealed electro-gilt. The former gives a röntgenogram which shows a characteristic rolling structure, the (112) direction being in the direction of rolling, and the (011) plane in the plane of rolling. Annealed gilt coatings have an irregularly oriented crystal structure. If rolled-gold articles are annealed after rolling, 22-carat coatings lose the rolled structure at 500°, 14-carat at 600°, and 12-carat above 650°; hence the method cannot be applied to finished articles in all cases.

A. R. POWELL.

Desulphurisation of burnt pyrites. FIGAI and PIACENTINI. **Source of aluminium.** SEKI.—See VII. **Electrolytic corrosion.** ANGEL and BECK-FRIS.—See XI.

See also A., Nov., 1220, Producing long single crystals of metal (GOETZ and HASLER). 1229, Austenite \rightarrow martensite in hardened steel (SCHEIL). 1231, Adsorption of phosphoric acid by stannic sulphide [in bronze analysis] (CHANDELLE). 1247, Electrodeposition of chromium (ROUDNICK). 1253, Tantalum (SPITZIN and KASHTANOV). Preparation of pure uranium (BOTOLFSSEN). 1259, Electrometric determination of copper (PRING and SPENCER). Determination of aluminium (BENEDETTI-PICHLER). 1261, Continuous purification of mercury (BOTOLFSSEN).

PATENTS.

Treatment of materials for the concentration of iron contained therein. S. G. S. DICKER. From

BRADLEY-FITCH Co. (B.P. 319,309, 19.6.28).—The ore is heated at 400–450° in an atmosphere of reducing gas containing hydrogen and steam, the heat required being generated by partial combustion of the gases. The product, which contains ferrosiferrous oxide, is subjected to magnetic separation for the recovery of the iron. (Cf. B.P. 319,392; *infra*.) A. R. POWELL.

Briquettes for use in refining or treating molten metals [iron]. G. C. CASTLE, C. WOOD, and BEECROFT & PARTNERS, LTD. (B.P. 319,603, 22.6.28).—The briquettes are composed of a ferro-alloy, *e.g.*, ferro-silicon, -manganese, -phosphorus, -chromium, -vanadium, or -titanium, bonded with lime cement and sodium carbonate. A. R. POWELL.

Direct production of iron or steel. H. D. PIERSON and others (HELDING & PIERSON), and A. E. M. LEDEBOER and others (STAALSIND. DR. LEDEBOER) (B.P. 287,925, 21.3.28. Holl., 30.3.27).—Sponge iron is melted in a furnace by means of a current of hot gases produced by a surface-combustion burner, the gases being almost or completely burnt before coming into contact with the charge. A. R. POWELL.

Production of iron or steel of a low degree of cold-brittleness, blue fracture, etc. VEREIN. STAHLWERKE A.-G. (B.P. 294,246, 16.7.28. Ger., 21.7.27).—The molten metal prior to casting is treated with at least 0.1% Al, Ti, Mg, Zr, or other metal having a similar deoxidising action, and the resulting castings are rolled at temperatures between 700° and the Ar₃ point, and are then subjected to a normalising anneal. A. R. POWELL.

Heat-treatment of iron. A. F. MEEHAN, Assr. to MEEHANITE METAL CORP. (U.S.P. 1,731,346, 15.10.29. Appl., 22.7.29).—In the manufacture of cast iron a casting, resulting from molten metal to which has been added an amount of an alkaline-earth metal in excess of that which would be neutralised by any element of the molten mixture, is heat-treated. F. G. CROSSE.

Die-casting of cast iron. C. METER (U.S.P. 1,731,676, 15.10.29. Appl., 20.6.25).—To produce a free-flowing, non-scaling iron suitable for use under pressure in steel moulds, about 2% of an alloy containing not less than 50% Al, 25% Ni, and 25% Si is mixed with pig iron. F. G. CROSSE.

Cast-iron alloys. CLIMAX MOLYBDENUM Co., Assees. of J. K. SMITH (B.P. 308,751, 19.6.28. U.S., 28.3.28).—The alloy consists of iron with 2.7–3.7% C of which 0.2–0.8% is combined, 1.5–3% Si, 0.05–0.2% S, 0.1–1.25% P, 0.25–1.25% Mn, and 0.2–2.5% Mo. The preferred composition is 3.3% C (0.6% combined), 2.16% Si, 0.09% S, 0.54% P, 0.43% Mn, and 0.35–2% Mo. The last-named is added as a high molybdenum-silicon-iron alloy free from carbide. A. R. POWELL.

[Copper-iron] alloys [containing chromium]. W. P. DIGBY (B.P. 319,899, 30.8.28).—Alloys of iron with 10–25% Cu, 0–9.5% Cr, 0.2–0.8% Mn, and 0.8% C are claimed. The materials are melted in an electric furnace the electrodes of which supply the carbon, and the manganese is added about 5 min. before casting. A. R. POWELL.

Production of molybdenum-bearing iron. CLIMAX MOLYBDENUM Co., Assees. of A. KISSOCK (B.P. 299,800, 19.6.28. U.S., 1.11.27).—Pig iron containing a predetermined quantity of molybdenum is obtained by adding the requisite quantity of molybdenum oxide, calcium molybdate, or molybdenum sulphide to the usual blast-furnace charge. A. R. POWELL.

Air-toughened alloy [manganese] steel. J. H. HALL and J. S. COMERFORD, Assrs. to TAYLOR-WHARTON IRON & STEEL Co. (U.S.P. 1,732,202, 15.10.29. Appl., 16.5.27).—A steel containing less than 1% C, an effective amount up to 5% Ni, less than 0.5% Si, and 10–15% Mn. F. G. CROSSE.

Electrodeposition of metal [iron]. F. K. BEZZENBERGER, Assr. to ALUMINUM Co. OF AMERICA (U.S.P. 1,729,607, 1.10.29. Appl., 14.7.21. Renewed 7.6.24).—As electrolyte a solution of ferrous sulphate containing precipitated iron hydroxide in suspension and having a mud-like consistency is claimed. F. G. CROSSE.

Froth-flotation processes. F. KRUPP GRUSONWERK A.-G. (B.P. 310,834, 4.8.28. Ger., 1.5.28).—Pulp for flotation treatment is fed centrally into a conditioning vessel through which it passes alternately towards and away from the periphery along a series of superimposed transverse surfaces in a tortuous path to a central discharge outlet in such a way that settling and thickening are avoided. A. R. POWELL.

Roasting of fines ore. C. L. LEVERMORE, Assr. to GEN. CHEM. Co. (U.S.P. 1,730,738, 8.10.29. Appl., 23.2.28).—In the desulphurisation of finely-divided sulphide ores having a moisture content of less than 0.3%, the ores are roasted while in gaseous suspension. F. G. CROSSE.

Extraction of mercury from its ores and from residues of their roasting. I. CAVALLI (U.S.P. 1,731,669, 15.10.29. Appl., 22.9.27. It., 24.9.26).—The ore or waste material is concentrated hydraulically, and subjected to the action of an alkali hypochlorite. F. G. CROSSE.

Working-up of materials containing lead, tin, copper, and antimony. HÜTTENWERK NEDERSCHÖNEWEIDE A.-G. (B.P. 294,487, 23.7.28. Ger., 21.7.27).—Scrap bearing metal etc. is heated in a shaft furnace by means of oil burners operating at the top so that the heat travels downwards by conduction. The lead-tin alloy which melts out of the mass gradually flows downwards, alternately solidifying and re-melting, and is finally tapped from the bottom of the shaft. A. R. POWELL.

Production of [non-corrosive] aluminium alloy. D. R. TULLIS (B.P. 319,967, 31.10.28).—Aluminium is alloyed with 0.5–5% Mg, 0.2–3% Cr, 0.1–2% Ca, and, if desired, up to 0.2% Si. The alloy is refined by the process described in B.P. 272,326 (B., 1927, 606). F. G. CROSSE.

Production of pliable tungsten. M. N. RICH, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,731,269, 15.10.29. Appl., 23.1.25).—Tungsten which has become brittle by the absorption of hydrogen is rendered pliable by arranging it as the anode in an electrolyte, so that when a current is passed through it nascent oxygen is evolved at the surface of the metal and is isolated from the action of cathodic hydrogen. F. G. CROSSE.

Resistance alloy. M. N. RICH, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,731,267, 15.10.29. Appl., 1.3.27).—An alloy containing at least 60% Cr and at least 40% W. F. G. CROSSE.

[Tungsten-tantalum] alloy. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,731,255, 15.10.29. Appl., 22.10.26).—A mixture of powdered tungsten with tantalum oxide is compressed, sintered *in vacuo* to consolidate it and reduce the oxide, and heat-treated at about 2000° to cause diffusion of the metals.

F. G. CROSSE.

Recovery of manganese values from solutions containing manganese. S. G. S. DICKER. From BRADLEY-FITCH Co. (B.P. 319,392, 19.6.28).—In the extraction of manganese from ores by the process described in B.P. 284,098 (B., 1928, 235) the solution of manganese sulphate obtained by leaching the reduced ore with ammonium sulphate solution is heated first with a small quantity of ammonia and agitated with air to remove iron, then with a larger quantity of ammonia and air to precipitate the manganese, the ammonia being derived from the previous leaching process. The insoluble residues from this process are magnetically separated and the non-magnetic product is returned to the reduction furnace, preferably after treatment on concentrating tables for the removal of silica.

A. R. POWELL.

Treatment of ores and the like containing manganese. S. G. S. DICKER. From BRADLEY-FITCH Co. (B.P. 319,605, 19.6.28).—Manganese ore reduced in coal gas at 400–450° (cf. B.P. 284,098; B., 1928, 235) is leached in two stages with hot ammonium sulphate solution under reduced pressure, the ammonia being removed as rapidly as it forms and more than 2 mols. of ammonium sulphate being present for every mol. of manganous oxide. (Cf. B.P. 319,392, preceding.)

A. R. POWELL.

Gold alloys. V. D. DAVIGNON, Assr. to GEN. PLATE Co. (U.S.P. 1,731,210–3, 8.10.29. Appl., [A, B] 10.6.26, [C, D] 23.6.28).—Homogeneous, malleable, and ductile alloys are claimed containing (A) Au 58, Cu 8, Mg 3, Zn 5, Ag (by diff.) 26%; (B) Au 50, Si 1–3, Mn 1%, and the remainder copper; (C) Au 25–85%, substantial amounts up to 3% of metal of the group consisting of silicon and titanium, small but substantial amounts of manganese, and the remainder chiefly silver; and (D) Au 25–85%, Ti up to 3%, and the remainder chiefly copper.

F. G. CROSSE.

Case-hardening of metals. P. W. and E. B. SHIMER (B.P. 292,557, 21.6.28. U.S., 22.6.27).—A case-hardening bath comprises a fused mixture of calcium and sodium chlorides to which is added at hourly intervals 0.25–1% of calcium cyanide and/or cyanamide.

A. R. POWELL.

Treatment of iron pyrites. S. I. LEVY (U.S.P. 1,730,514, 8.10.29. Appl., 25.2.28. U.K., 10.2.28).—See B.P. 307,188; B., 1929, 399.

Purifying pig iron. F. WÜST (U.S.P. 1,730,960, 8.10.29. Appl., 30.6.27. Ger., 17.7.26).—See B.P. 274,438; B., 1928, 862.

Hardening of high-speed steel. W. STAUFFER, Assr. to A.-G. DER MASCHINENFABR. ESCHER WYSS & CIE. (U.S.P. 1,731,549, 15.10.29. Appl., 7.4.28. Switz., 23.4.27).—See B.P. 289,082; B., 1929, 250.

Bearing-metal alloy. K. MÜLLER and W. SANDER (U.S.P. 1,731,021, 8.10.29. Appl., 21.7.28. Ger., 1.10.20).—See B.P. 174,610; B., 1923, 316 A.

[Cast-steel] annealing cases. ACIÉRIES RÉUNIES DE BURBACH-EICH-DUDELANGE SOC. ANON. (B.P. 310,489, 25.6.28. Luxembourg, 27.4.28).

Casting and rolling copper. UNITED STATES METALS REFINING Co., Assees. of W. F. EPPENSTEINER and H. M. GREEN (B.P. 305,998, 12.2.29. U.S., 13.2.28).

Furnaces for heat-treating metals etc. (B.P. 319,300).—See I. Electric furnace for metals (B.P. 297,826).—See XI.

XI.—ELECTROTECHNICS.

Electrolytic corrosion due to current leakages and its prevention. G. ANGEL and C. BECK-FRIIS (Chem.-Ztg., 1929, 53, 553–554, 574–575).—Some examples of electrolytic corrosion due to leakage of current in a generating station and in the neighbourhood of underground conductors are briefly described, together with methods for measuring the resistance of the local cells set up and the loss of energy due to leakage. Several methods of preventing corrosion of electric tramway rails are mentioned.

A. R. POWELL.

Oxidation of toluene. MITCHELL.—See III.

See also A., Nov., 1247, **Electrodeposition of chromium** (ROUDNICK). **Production of nitric oxide** (BRINER and RIVIER). **Preparation of phenylhydrazine** (McCLURE). 1251, **Behaviour of alkali fluoroborates in tungsten-filament lamps** (DE BOER). 1259, **Determination of copper** (PRING and SPENCER). **Cæsium-magnesium photo-cell** (ZWORYKIN and WILSON). 1276, **Electrolytic reduction of acetone** (HAGGERTY). 1291, **Electrolytic preparation of 2:4-diaminophenol** (BRADT and BROWN).

PATENTS.

Electric resistance furnace for the thermal treatment of metals etc. "INFRA," Assees. of V. SORREL and L. A. LAFONT (B.P. 297,826, 17.9.28. Fr., 29.9.27).—The temperature of an electric furnace is automatically regulated by a balance, the equilibrium of which is controlled by the variations with temperature of the state of magnetisation of a magnetic body.

J. S. G. THOMAS.

Electron-discharge device. F. ROTHER (B.P. 300,547, 24.10.28. Ger., 14.11.27).—A cathode in an evacuated vessel is bombarded with α - or β -particles or with γ -rays emitted from a radioactive anode freed from gas, the active area of the cathode being smaller than the area of the anode.

J. S. G. THOMAS.

Electron-discharge device. H. C. RENTSCHLER and J. W. MARDEN, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,731,268, 15.10.29. Appl., 28.3.25).—The envelope contains a cathode of the thoriated type and an anode composed of copper containing occluded hydrogen.

F. G. CROSSE.

Insulating [cellulosic] filament. H. A. GARDNER (U.S.P. 1,730,417, 8.10.29. Appl., 10.1.28).—A cellulosic filament contains an electrically non-conducting resin and finely-divided, non-conducting, micaceous material.

J. S. G. THOMAS.

Electrolytic preparation of antiseptic and cicatrising solutions. F. CHEVRIE and M. L. GRILLE (B.P. 320,096, 4.4.28).—An antiseptic solution is electrolysed between substantially horizontal electrodes, the anodic current density being so high that considerable ozone is produced.

J. S. G. THOMAS.

Photo-electric cell. R. HART (U.S.P. 1,730,505, 8.10.29. Appl., 30.1.28).—A selenium cell containing iodine is claimed.

H. ROYAL-DAWSON.

Manufacture of nickel flakes for use as admixture in the active material of alkaline accumulators. E. BREUNING and O. SCHNEIDER, Assrs. to ELECTRIC STORAGE BATTERY CO. (U.S.P. 1,732,179, 15.10.29. Appl., 6.7.27. Ger., 27.7.26).—See B.P. 275,221; B., 1928, 198.

[Liquid replenisher for] electrical accumulators and storage batteries. D. E. WASHINGTON (B.P. 320,026, 31.3.28).

[Vulcanite frames for electrodes of] electric accumulators. C. J. WARD and S. E. HILLMAN (B.P. 319,634, 21.6.28).

Manufacturing [spraying of emissive substances on to] cathodes of thermionic valves. S. LOEWE (B.P. 293,694, 9.7.28. Ger., 9.7.27).

[Medical] apparatus for X-ray investigation. SIEMENS-REINIGER-VEIFA GES. F. MEDIZ. TECHN. M.B.H. (B.P. 301,377, 11.10.28. Ger., 28.11.27).

Charging gases with vapours (B.P. 305,089). Apparatus for evaporating, distilling, etc. (B.P. 319,849).—See I. Indicator for inflammable gases (B.P. 319,530). Converting carbon into liquid hydrocarbons (U.S.P. 1,730,997).—See II. Revivification of potassium ferrocyanide (B.P. 319,147).—See VII. Electrodeposition of iron (U.S.P. 1,729,607). Pliable tungsten (U.S.P. 1,731,269). Resistance alloy (U.S.P. 1,731,267).—See X. Dehydration of rubber (B.P. 299,713).—See XIV. Preservation of liquids (B.P. 294,903).—See XIX.

XII.—FATS; OILS; WAXES.

Fats. A. VAN RAALTE (Rec. trav. chim., 1929, 48, 1058—1060).—Fats are separated into solid and liquid phases by extraction with a mixture of alcohol and acetone at ordinary temperature. The proportion of liquid varies with the source (e.g., lard > beef fat). The refractive indices, iodine and Crismer values of the original fat and solid and liquid phases all vary considerably. The different solid (or liquid) fractions of the fats examined also show varying differences.

H. BURTON.

Fat of the seed of the murumuru (*Astrocaryum murumuru*). M. SARAIVA (Mem. Inst. Chim. Rio de Janeiro, 1929, No. 2, 5—19).—A botanical description of the murumuru is given. The dried seed contains 39.7% of fatty material extractable by ether, m.p. 32°, iodine value (Hübl) 11.0, acid value 14.5, saponif.

value 242.0, Reichert-Meissl value 2.8, n_D^{20} 1.4540 (cf. André and Guichard, A., 1925, i, 1124; B., 1925, 728). The composition, determined by separating the lead salts of the fatty acids, bromination of the unsaturated fraction, and distillation of the methyl esters of the saturated fraction, is as follows: glyceryl radical 5.89%; acid radicals: octoyle 1.03%, deconoyle 1.47%, lauryl 39.92%, myristyl 34.55%, palmityl 4.26%, stearyl 2.01%, linoleyl 0.38%, oleyl 10.13%, unsaponifiable matter 0.26%. The fat is edible and tasteless and suitable for the manufacture of margarine or pomades.

R. K. CALLOW.

Microdetermination of glycerol in fats by Zeisel and Fanto's method. B. FLASCHENTRÄGER (Mikrochem., 1929, Pregl Fest., 89—90).—The glycerol in 20 mg. of fat can be satisfactorily determined by Willstätter's method, using concentrated hydriodic acid. The isopropyl iodide formed is decomposed and the silver iodide weighed.

H. F. HARWOOD.

Injurious action of heavy metals on soaps during the washing process. E. L. LEDERER (Deut. Parfümerieztg., 1928, 14, 487—488; Chem. Zentr., 1929, i, 2714).—A discussion.

A. A. ELDRIDGE.

Chemical and physical characteristics of cod oil [industrial cod-liver oil]. A. D. HOLMES, W. Z. CROUGH, and R. J. OWEN (Oil and Fat Ind., 1929, 6, [10], 15—18).—The characteristics of a number of industrial cod-liver oils of various qualities (pure to tannery cod oils) were determined; these show wide variations and the need for standards of these oils is emphasised.

E. LEWKOWITSCH.

Oil seeds. ANON. (Bull. Imp. Inst., 1929, 27, 277—289).—The oils prepared from two varieties of perilla (*P. ocymoides*) seed from Assam and one (*P. nankinensis*) from Manchuria had, respectively, d_{4}^{15} 0.9350, 0.9344, 0.9348; n_D^{20} 1.475, 1.475, 1.474; acid value 1.2, 2.3, 2.6; saponif. value 192.6, 192.6, 192.9; iodine value (Hübl, 17 hrs.) 198, 196.8, 192.6; unsaponif. matter 0.9, 1.1, 0.9%. The characteristics are thus similar to those recorded for *P. ocymoides* and commercial perilla oil, and meet the requirements of the U.S. specifications for this oil.

Oiticica nuts (*Icacia rigida*, Benth) from Rio Grande and Brazil yielded 74% of kernels which contained 4.7% of moisture and 61% of oil. The extracted oil was partly liquid and partly solid; a commercially expressed oil was a soft fat. The extracted (2 samples) and expressed fats had, respectively, d_{4}^{15} 0.9674, 0.9679; n_D^{20} 1.507, 1.504; acid value 1.8—5.6, 4.4; saponif. value 189.5—186.1, 190.8; iodine value (Wijs, 3 hrs.) 140.5—144.8, 139; unsaponif. matter 0.5—0.9, 0.9%; solidifying point of fatty acids 47.4—45.4, 44.8°. On heating the oil to 190° and above gas bubbles are formed; on heating at 250—270° (30 min.) the oil does not polymerise, but on slowly raising the temperature to 300° the oil forms a transparent jelly.

Mlenda seed (*Sesamum angustifolium*, Engl.) from Tanganyika contained 7.5% of moisture and by extraction yielded 28.9% of a pale green limpid oil which gave a very slight deposit on keeping. The oil had d_{4}^{15} 0.9365, acid value 16.8, saponif. value 181.6, iodine value (Hübl, 17 hrs.) 117.7, n_D^{20} 1.4708, and gave a positive Baudouin

reaction. The meal contained (corresponding figures for sesame cake in parentheses): moisture 8 (8.12)%, crude proteins 23.8 (39.6)%, fat 0.3 (13.01)%, carbohydrates etc. (by diff.) 18.5 (22.0)%, crude fibre 42.8 (4.36)%, ash 6.6 (12.9)%, nutrient ratio 1:0.8 (1:1.31), food units 79 (154). The seeds thus resemble sesame, but are of lower value on account of the low yield of oil and the poor cake containing a very large amount of fibre.

The kernels (64.2%) from kullan nuts (*Balanites orbicularis*, Sprague) from British Somaliland contained 6.3% of moisture and 37.2% of a clear yellow oil (extracted) which had: d_{4}^{15} 0.9184, n_D^{20} 1.4623, acid value 0.3, saponif. value 192.7, iodine value (Hübl, 17 hrs.) 75.9, unsaponif. matter 0.5%, solidifying point of fatty acids 38.6°. The residual meal contained saponin but no cyanogenetic glucosides, gave positive reactions when tested for alkaloids, and had moisture 9.6%, crude proteins 30.5%, oil 1.4%, carbohydrates etc. (by diff.) 50.4%, crude fibre 3.7%, ash 4.4%, nutrient ratio 1:1.8, food units 130. The oil is non-drying and suitable for soap-making, and possibly for edible purposes.

Babassu (*Orbignia martiana*, Barb. Rodr.) fruits and kernels, from the States of Para and Amazonas, on extraction with light petroleum yielded solid fats and buff-coloured tasteless meals. The characteristics of the oils agreed closely with those already recorded for babassu fat (cf. Bray and Elliott, B., 1916, 1121). The kernels contained, respectively, 5.1 (4.9)% of moisture, and 67.3 (67)% of oil, which had d_{4}^{20} 0.8672, 0.8672; m.p. 24.1°, 25.4°; n_D^{20} 1.450, 1.450; acid value 3.5, 1.8; saponif. value 251.0, 254.7; iodine value (Wijs, 3 hrs.) 13.4, 9.7; unsaponif. matter 0.7, 0.8%; solidifying point of fatty acids 21.4°, 21.1°; volatile acids (sol.) 6.0, 6.4%; (insol.) 13.3, 13.0%. The composition (%) of the meals (expressed on meal containing 7% of fat) was: moisture 10.8 (10.5), crude proteins 22.7 (20.5), fat 7.0 (7.0), carbohydrates etc. (by diff.) 42.3 (47.8), crude fibre 12.7 (8.7), ash 4.5 (5.5); their nutrient ratio was 1:2.6 (1:3.1), food units 117 (117).

E. LEWKOWITSCH.

Oil from seeds of the anda-assú (*Johanesia princeps*, Vell). L. GURGEL and F. RAMOS (Mem. Inst. Chim. Rio de Janeiro, 1929, No. 2, 21—29).—A botanical description of the anda-assú is given. The seed contains 51.8% of oil extractable by ether, acid value 24.8, saponif. value 207.3, iodine value (Hübl) 122.5, n_D^{25} 1.4742. The composition, determined by separating the lead salts of the fatty acids, bromination of the unsaturated fraction, and distillation of the methyl esters of the saturated fraction, is as follows: glyceryl radical 4.23%; acid radicals: oleyl 42.5%, linoleyl 43.10%, myristyl 2.20%, palmityl 5.03%; unsaponifiable matter 0.97% (a substance, m.p. 131°). The oil is used as purgative, particularly in veterinary medicine. It is suitable for use in soap-making.

R. K. CALLOW.

Oil from [seeds of] the páo marfim (ivory wood) (*Agonandra brasiliensis*, Miers). L. GURGEL and T. F. DE AMORIM (Mem. Inst. Chim. Rio de Janeiro, 1929, No. 2, 31—38).—A botanical description of the plant is given. Extraction of the seeds with ether yields

35% of brown, viscous oil, d_{4}^{15} 0.9602, n_D^{20} 1.4925, acid value 66.8, saponif. value 207.3, iodine value (Hübl) 112.3, completely soluble in cold alcohol, soluble in 11 pts. of light petroleum. The composition, determined by separating the lead salts of the fatty acids, bromination of the unsaturated fraction, and distillation of the methyl esters of the saturated fraction, is as follows: glyceryl radical 4.90%; acid radicals: ricinoleyl 44.85%, linoleyl 34.65%, oleyl 11.45%, myristyl 2.21%, palmityl 1.32%, unsaponifiable matter 0.60%. The oil is easily oxidised, and may be sulphonated to yield a product resembling Turkey-red oil. Prolonged action of sulphuric acid and partial hydration yields a spongy substance which might be used in the preparation of rubber substitutes.

R. K. CALLOW.

Oil from seeds of *Sapindas trifoliatus*, Linn. [soapnut tree]. D. R. PARANJPE and P. R. AYYAR (J. Indian Inst. Sci., 1929, 12A, 179—184).—The soapnut fruits gave 65.7% of pericarp and 34.3% of seeds (one third kernels). The kernels yielded on extraction 44.7% of a dark yellow oil which deposited a small amount of stearine on keeping and had d_{4}^{20} 0.8540, n_D^{20} 1.4764, saponif. value 194.1, iodine value 58.5, Reichert-Meißl value 1.5, acetyl value 0.0, unsaponif. matter 1.2%, Hehner value 93.5 (cf. Menon, J.S.C.I., 1910, 29, 1431). The mixed fatty acids had saponif. value 195.4, iodine value 61.3, equiv. wt. 287, titre 47°; saturated acids 38.5%, n_D^{20} 1.4400, equiv. wt. 292.5; unsaturated acids 61.5%, iodine value 86.2, equiv. wt. 283, n_D^{20} 1.4450. From bromination experiments and fractionation of the methyl esters of the saturated acids it is deduced that the oil consists of the glycerides of palmitic (5.6%), stearic (8.5%), *n*-eicosic (21.9%), lignoceric (2.5%), and oleic (61.5%) acids. The unsaponifiable matter yielded 30% of (mixed) sterol, m.p. 115—120°. The oil is thus a rich source of *n*-eicosic acid.

E. LEWKOWITSCH.

Pecan [*Hicoria pecan*] oil. G. S. JAMIESON and S. I. GERTLER (Oil and Fat Ind., 1929, 6, [10], 23—24).—The nut fragments from the preparation of the edible pecan nuts or "meats" can be utilised for the production of an excellent salad oil of a mild agreeable flavour. The (expressed) oil had: d_{4}^{25} 0.9141, n_D^{25} 1.4692, saponif. value 190, iodine value (Hanus) 100, unsaponif. matter 0.35%, acid value 7.0, Reichert-Meißl value 0.05, Polenske value 0.30, acetyl value 7.4, saturated acids 5.09% (corr.), unsaturated acids 89.54% (corr.; iodine value 105.5). The saturated acids were separated by the lead salt-ether method, esterified (methyl alcohol), and fractionally distilled; the composition of the unsaturated acids was calculated from the iodine value. The oil contained the glycerides of oleic 77.8, linoleic 15.8, myristic trace, palmitic 3.3, stearic 1.9, and arachidic acids 0.1%, and unsaponif. matter 0.35%.

E. LEWKOWITSCH.

Vegetable oils of the Union of S.S.R. III. Nature of fatty oils of *Anacardiaceæ*, in connexion with the climate of district of origin. S. IVANOV and A. J. MAGNITOVA (Chem. Umschau, 1929, 36, 322—324; cf. B., 1929, 923).—Characteristics of the oils from various members of the *Anacardiaceæ* (pistacia, sumach, etc.) are collated: the preponderating unsaturated acid of the oils of these tropical and sub-tropical

plants is oleic acid. In passing from sub-tropical to tropical plants, the linoleic acid content of the oils decreases progressively and the amount of saturated acids increases; the iodine value changes correspondingly within the limits 120 (e.g., *Pistacia nutica*) and 25—30 (e.g., *Mangifera indica*). E. LEWKOWITSCH.

Formula for calculating the organically combined sulphur trioxide in sulphonated oils. R. HART (Chem. Umschau, 1929, 36, 321—322).—It is shown that the second formula, $\% \text{SO}_3 = 8(F - B)/56 \cdot 1$, given by Nishizawa and Winokuti (cf. B., 1929, 402) is a special case of the general formula, $\% \text{SO}_3 = 8(F + A)/56 \cdot 1$, given by Bauer and the American Leather Chemists' Association (cf. B., 1919, 871 A), which is applicable to all cases if A (mg. KOH/g. of oil) is taken as positive when it indicates the amount of acid needed to neutralise the soap, and as negative when it indicates the alkali required to neutralise an acid oil. E. LEWKOWITSCH.

Reclamation of tung oil. STEINHOFF.—See XIII.

See also A., Nov., **Determination of organic peroxides** (MARKS and MORRELL). 1270, **Preparation of fatty acids from their higher homologues** (ROCHUSSEN). 1271, **Mechanism of degradation of fatty acids by mould fungi** (STENT and others). **Separation of glycerides** (SUZUKI and others). 1292, **Irradiated sterols** (MONTIGNIE). 1306, ***Embelia Ribes*** (KAUL and others). 1330, **Halibut tumour oil** (KAMM).

PATENTS.

Rendering and/or digesting subjects containing fats and separating the products. M. GEARIN & SONS, LTD., and J. R. CULLEN (B.P. 297,728, 2.7.28. Austral., 26.9.27).—The material is treated in a steam-jacketed digester with open steam introduced at a temperature and pressure lower than those employed for the jacket steam (e.g., 5—20, and 30—50 lb./in.², respectively) to ensure the dryness of the former; the digester is designed to facilitate the collection of the oil from the charge. E. LEWKOWITSCH.

Detergents. H. and J. COPLAND (B.P. 320,088, 4.7.28).—A jellified detergent for carpets etc., containing soap, sodium carbonate, colloidal clay, and a large proportion of water, with, if desired, sodium borate, is claimed. E. LEWKOWITSCH.

Manufacture of toilet soaps, and of medicated or disinfectant soaps. P. VILLAIN (B.P. 319,804 and 319,832, 26.4.28).—The rosin in soap is replaced by an aromatic resinous composition produced by emulsifying as required terpenes (or sesqui- or poly-terpenes), synthetic or natural resins, and residues from the solvent-extraction of flowers ("floral wax") with glycerin, a suitable solvent (e.g., methyl alcohol, diacetone), and an emulsifier (e.g., 5% ammonium sulphoricinoleate). Medicinal and disinfectant substances (e.g., iodine, formalin, birch tar) are added as required. E. LEWKOWITSCH.

[Medicated] soap. P. H. TODD (U.S.P. 1,731,551, 15.10.29. Appl. 25.5.27).—A soap composition contains 1—15% of cresol and 0.25—3% of mercuric iodide.

F. G. CROSSE.

Production of [non-caking] soap threads. A. WELTER (B.P. 319,807, 26.5.28).—An alkali salt, or

mixture of such salts, excepting sodium carbonate, e.g., bicarbonates, sulphates, borates, per-salts, β -naphthalenesulphonates, up to the amount producing salting-out, is added to the soap paste or curd soap (preferably in the liquid state), with or without the addition of fat solvents; the solid mixture is pressed through fine nozzles (max. diam. 1.5 mm.).

E. LEWKOWITSCH.

Apparatus for spraying and applying viscous oils or fats. C. RICHTER (B.P. 295,362, 10.8.28. Ger., 10.8.27).

Substances resembling cork (B.P. 300,207).—See V.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oxidation and weathering of linseed oil paints. II. J. D'ANS (Z. angew. Chem., 1929, 42, 997—999; cf. B., 1928, 935).—It has been noted that the oxidation products of linseed oil include a large number of fatty acids. To test the effect of these on the durability of paint, films of linseed varnish either alone or with different pigments were prepared and drops of the acids in question allowed to fall upon them. With the varnish alone the effect varied inversely with the volatility of the acid, that of formic and acetic acid being negligible. If basic pigments, e.g., white lead, are present, the lower acids dissolve the pigment without affecting the film, producing transparent spots. The higher acids, however, render the film brittle and cause blisters, particularly with zinc white. Hexoic acid in a moist atmosphere at 34° renders all films soft and pasty in a short time, and the effect persists after removal of the acid. In this case the linoxyn molecule has apparently suffered hydrolysis; the products of this hydrolysis are known to be very complicated. In appearance the various films closely resembled films naturally damaged by weather. The steps in the weathering of paint are therefore (1) oxidation, (2) hydrolysis of the oxidation products and interaction of the compounds so formed with the pigment.

C. IRWIN.

Testing and evaluation of the durability of paints. H. WOLFF (Farben-Ztg., 1929, 35, 182—185).—A dissertation on weathering tests, normal and accelerated, and the conclusions to be drawn therefrom. Stress is laid on common fallacies in the interpretation of such tests, e.g., a "chalking" paint may be more durable than one that develops porosity without chalking. Two types of rusting are considered: (a) the "classic" type, in which the combined action of water and air causes disintegration of the iron surface under the paint; and (b) the type in which dissolved iron permeates the paint film and is oxidised as a surface layer, leaving the iron surface in good condition under the paint, as can be demonstrated by removing the film. Forecasts of the durability of paints can but be guesses in view of variations in conditions of application, drying, etc., and guarantees of a definite life for a paint are considered to be unreliable and misleading.

S. S. WOOLF.

Action of pigments on tung oil. H. WAGNER (Farben-Ztg., 1929, 35, 179—182).—The consistency of

pigment-tung oil pastes stored in tubes for one year showed a general tendency towards thickening, leading in some cases to the production of hard, crumbly masses, attributable to the separation of β -elæostearin. Freshly ground pastes examined microscopically at ages of from 1 to 14 days show differences as to the extent and crystalline form of glyceride separated. Iron oxide pigments, chromium hydroxide-green, cadmium-orange, some types of ultramarine-blue, barytes, china clay, calcium carbonate, etc. ground in tung oil show little divergence from the behaviour of a tung oil film in regard to separation of β -elæostearin, such variations as may occur being attributable to mechanical influence on crystallisation. Catalytic influence on the $\alpha \rightarrow \beta$ rearrangement is, however, considered to be evinced by the relatively great divergence from tung oil of pastes containing zinc sulphide, lithopone, and most ultramarine-blues (positive catalysis), and zinc oxide, calcium carbonate (negative catalysis). Photomicrographs of the various pastes are given. It is suggested that the phenomenon is capable of being linked with the drying and polymerisation of tung oil. S. S. WOOLF.

Pigment and oil. E. KLUMPF and H. MEIER (Farben-Ztg., 1929, 35, 127—128).—The view that oil absorption corresponds to pore-volume only, virtually regardless of the size and nature of the pigmentary particles, is discussed (cf. B., 1929, 862). The relations between primary and secondary particles account for all possible changes in oil absorption on grinding pigments. Baldwin's experiments (cf. B., 1929, 443) are criticised on the grounds that he worked with indefinite secondary particles. By the use of peas, glass beads, seeds, etc. it is demonstrated that the oil absorption-composition curve for binary mixtures of primary pigments of different sizes should show a minimum. This is confirmed, using mixtures of barytes (diam. of particle 10—15 μ) with white lead (2 μ), "high-dispersion" red lead (1.5—2 μ), and zinc yellow (1.5 μ), respectively. As the difference in particle size diminishes, so does the curve flatten, and at equal particle size the minimum is absent, the mixture of two primary pigments behaving as a single one. S. S. WOOLF.

Glycerin used to reclaim china-wood [tung] oil [varnishes]. E. STEINHOFF (Oil and Fat Ind., 1929, 6, [10], 25, 43).—Glycerin may be used for the purpose, whether the batch is cold and solid or has just begun to congeal. *E.g.*, a long-oil varnish containing 60 gals. of tung oil is heated with 1 gal. of glycerin, 5 lb. of litharge being sprinkled on to reduce the froth; the mixture is cooled and thinned as usual. The glycerin, forming an ester with the rosin and tung oil, hardens the product and makes it more waterproof, whilst the litharge serves to accelerate the drying.

E. LEWKOWITZCH.

Oxidation of turpentine in the open air. K. N. KOROTKOV (Zapiski Belarus. Dzyarzh. Akad. Selsk. Gasp., 1928, 7, 142—153).—In the absorption of oxygen by turpentine carbon dioxide is formed; 30% of the oxygen absorbed is "active" and available. The turpentine from *Pinus sylvestris* can absorb 34 litres of oxygen per litre.

CHEMICAL ABSTRACTS.

See also A., Nov., 1251, Ultramarine (HOFFMANN). 1306, Resin of *Euphorbia palustris* (MÜLLER).

PATENTS.

Manufacture of paint [from celluloid etc.]. R. J. CARRUTHERS (B.P. 319,978, 7.11.28).—Zinc oxide is ground in a mixture of a methyl-alcoholic solution of shellac and a suitable cellulose acetate solution, and added to a solution of preferably non-inflammable celluloid cuttings in a mixture of acetone, benzol, and absolute alcohol. S. S. WOOLF.

Preparation of synthetic resin varnishes. G. E. LANDT and W. H. ADAMS, JUN., ASSRS. to CONTINENTAL DIAMOND FIBRE Co. (U.S.P. 1,731,071—2, 8.10.29. Appl., 31.8.27).—(A) The initial condensation product of a synthetic resin is dissolved in a solution prepared by causing ammonia and formaldehyde to interact to produce hexamethylenetetramine in an organic solvent. (B) The hexamethylenetetramine is produced in the solution of the synthetic resin in the organic solvent.

S. S. WOOLF.

Liquid coating composition. G. H. MAINS, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,730,857, 8.10.29. Appl., 6.8.27).—The condensation products of formaldehyde, a drying oil, and a coal-tar acid containing a substantial amount of xyleneol are claimed.

S. S. WOOLF.

Coating of articles. E. I. DU PONT DE NEMOURS & Co. (B.P. 287,940, 30.3.28. U.S., 30.3.27).—An "anchoring" [priming] coat comprising a mixed ester of a polyhydric alcohol with a polybasic acid and a drying oil or the acids derived therefrom, a solvent for the ester, a drier, and pigment is applied to a metal or other surface, allowed to dry, and a coating of different composition is applied. The latter may be a "binding" coat of the drying-oil type, to be followed after drying by a "top coat" of a cellulose ester lacquer.

S. S. WOOLF.

Synthetic resins and compositions prepared therefrom. E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 320,041, 27.3.28).—A polyhydric alcohol other than pentaerythritol is heated in a non-reactive atmosphere to 180—265° with substantially the theoretically required amounts of a polybasic acid and a drying-oil acid, with or without other acidic materials, *e.g.*, natural resins having an acid value of at least 50. Combinations of the synthetic resins thus obtained with cellulose esters or ethers are also claimed.

S. S. WOOLF.

Manufacture of artificial [furfuraldehyde-amine condensation] products. H. KAPPELER (B.P. 293,872, 13.7.28. Switz., 14.7.27).—Furfuraldehyde, alone or mixed with other aldehydes, is condensed with urea and/or other amino-compounds, part of the reaction at least being conducted in the presence of an acid or a salt having an acid reaction. Solid, black, artificial resins, insoluble in the usual resin solvents, stable towards alkali lyes and dilute acids, and having the appearance of ebonite are obtained. S. S. WOOLF.

Cutting plates and sheets from blocks of artificial resins. K. W. and K. W. RASCHIG (F. RASCHIG) (B.P. 319,806, 30.4.28).

Non-splintering glass (B.P. 319,873).—See VIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Natural coagulation of *Hevea* latex. A. S. CORBET (Rubber Res. Inst. Malaya, Bull. No. 1, 1929, 17 pp.).—Latex in process of spontaneous coagulation is swarming with bacteria; isolation of the predominating organism has now been effected. This organism, designated *Bacillus pandora*, is a facultative anaerobe, able to decompose latex substances with formation of acid, and consequently to cause separation of the rubber. It subsequently attacks the proteins with production of ammonia and so is responsible for the acid serum and alkaline surface layer observed during the natural coagulation of latex. The effect of sodium sulphite or ammonia as anticoagulants is attributed to their removal of free lactic acid; assuming that a molecule of methylinositol can yield five carboxyl groups, the proportion of this substance in latex calls for 0.35 g. of ammonia per 100 g. of latex to prevent development of acidity.

D. F. TWISS.

Oil of páo marfim. GURGEL and DE AMORIM.—See XII.

See also A., Nov., 1307, Isoprene and caoutchouc (STAUDINGER and BONDY).

PATENTS.

Concentration of natural [rubber] latices. K.D.P., LTD., Assees. of METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 293,771, 11.7.28. Ger., 11.7.27).—In the production of reversible concentration products from natural latices, the addition of protective colloids such as soap can be dispensed with if alkali fluorides and/or salicylates are introduced prior to concentration.

D. F. TWISS.

Preparation of concentrated india-rubber latex. DUNLOP RUBBER Co., LTD., II. J. ALCOCK, ANODE RUBBER Co. (ENGLAND), LTD., KOOPMAN & Co., and AKTIEBOLAGET SEPARATOR (B.P. 319,410, 26.4.28).—Fresh latex is treated with 0.1–0.3% of ammonia and is then concentrated by centrifuging, e.g., in an Alfa Laval machine (cf. B.P. 219,635; B., 1925, 139). The ammonia content of the concentrate is then adjusted to approx. 0.3–0.4%. The rubber in the "skim" is recovered by coagulation with acid.

D. F. TWISS.

Manufacture of rubber or rubber-like material. GOODYEAR TIRE & RUBBER Co., Assees. of A. M. CLIFFORD (B.P. 302,147, 4.9.28. U.S., 10.12.27).—Substituted aryl hydroxy-compounds, without substantial accelerating effect on vulcanisation, e.g., α -amino- or α -chloronaphthol, are applied as anti-oxidants or age-retarders for rubber or rubber-like materials. D. F. TWISS.

Manufacture of rubber-like materials. J. C. WICHMANN, Assr. to CACTUS RUBBER Co. OF AMERICA (U.S.P. 1,703,702, 8.10.29. Appl., 20.9.27).—The concentrated juice from the fleshy parts of the cactus family is boiled with sodium tungstate, sodium molybdate, boiled linseed oil, and rubber dissolved in turpentine, and the resulting mass is dried and oxidised.

H. ROYAL-DAWSON.

Apparatus for filtering rubber dispersions and the like. ANODE RUBBER Co., LTD., Assees. of A. SZEGVARI and C. M. SPENCER (B.P. 294,215, 20.7.28.

U.S., 20.7.27).—A filtering device for latex, with a minimum of coagulant effect, permits continuous uniform flow or circulation of the liquid, such as is desirable, e.g., in the manufacture of articles by electrodeposition. A continuous strip of filtering material, maintained in a moist condition, passes round a drum or cage into the interior of which flows the liquid to be filtered; the filtered liquid passes into the vessel in which the drum is contained and leaves by an aperture provided.

D. F. TWISS.

Production of deposits or coatings from aqueous suspensions of rubber. ANODE RUBBER Co. (ENGLAND), LTD., Assees. of COMP. GÉN. D'ELECTRICITÉ (B.P. 291,339, 29.5.28. Ger., 27.5.27).—Gel-forming substances (gelatin, agar-agar, white of egg) are added to aqueous suspensions of rubber which are then subjected to temperature changes through the surface of moulds brought into contact with the latter so that congealed layers incorporating rubber globuloids are formed at the mould surfaces. The formation of deposits may be aided by the addition of favourable substances (e.g., sulphates, acetates, in the case of gelatin) and of preservatives, and also by the diffusive action of coagulants from the moulds. Substances which retard gelation or which tend to hydrolyse certain gel-forming substances, e.g., alkalis, should be removed from the suspensions by known means, e.g., dialysis. If the presence of the gel-forming substances as such in the deposits is undesirable they may be removed or rendered insoluble by treatment with suitable agents.

D. F. TWISS.

Treatment of aqueous dispersions of organic materials such as rubber or the like. ANODE RUBBER Co. (ENGLAND), LTD. From F. GABOR (B.P. 319,801, 29.3.28).—Coagulation or gelling of latex is effected by heating, e.g., for a short time at 80–90°, after the incorporation of a substance such as white of egg which normally has a neutral or stabilising action on the latex, but is coagulable by heat. This method of coagulation or gelling is applicable to latex in various forms, e.g., in bulk, in layers (in or on moulds), or as filaments or tubing issuing from nozzles.

D. F. TWISS.

Dehydrating rubber deposited from aqueous dispersions. ANODE RUBBER Co., LTD., Assees. of C. L. DARBY (B.P. 299,713, 29.10.28. U.S., 28.10.27).—Layers or articles of rubber or similar substances formed from aqueous dispersions can be dried efficiently and rapidly by electroendosmosis, as much as 90% of the water in the deposit being removable in a few minutes with a sufficiently high voltage; e.g., the deposit formed on an electrically conductive material is immersed in a conducting fluid such as mercury or an electrolyte, or is brought into contact with metal plates, the conducting fluid or metal plates being connected with the negative pole and the electrically conductive material which supports the rubber, with the positive pole of a source of electric current.

D. F. TWISS.

Manufacture of rubber compositions. M. C. TEAGUE, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,730,485, 8.10.29. Appl., 31.3.25).—The union between rubber and pulverulent materials is improved by evacuating the latter, treating with ammonia gas in the presence of

moisture, and then introducing them into the rubber, whereby fine dispersion is effected. D. F. TWISS.

Curing [vulcanising] of sheet rubber and the like. C. MACINTOSH & Co., LTD., and H. C. YOUNG (B.P. 319,448, 27.7.28).—In vulcanising lengths of sheet rubber by passing the sheet between an endless band and a heated rotatable drum, the weight of the drum is sustained by the endless band; the pressure thus exerted may be augmented or stabilised by other means, e.g., by the application of hydraulic pressure.

D. F. TWISS.

Manufacture of synthetic rubber. I. G. FARBER-IND. A.-G. (B.P. 308,755, 6.7.28. Ger., 31.3.28. Addn. to B.P. 283,841; B., 1929, 485).—In the synthesis of rubber by polymerisation of an appropriate hydrocarbon in an atmosphere of hydrogen, nitrogen, or carbon dioxide, under the influence of an alkali metal, the presence of ethyl or phenyl ether or an olefine oxide or peroxide is especially valuable.

D. F. TWISS.

Production of [hollow] rubber articles. W. W. TRIGGS. From VEREIN. GUMMIWAREN-FABR. WIMPASING VORM. MENIER-J. N. REITHOFFER (B.P. 320,451, 10.8.28).

Emulsions of diolefines (B.P. 318,296).—See III.
Dyes from rubber derivatives (U.S.P. 1,724,270).—See IV.

XV.—LEATHER; GLUE.

(A) **Appearance and (B) bacteriology of red stains on salted raw hides.** F. STATHER and E. LIEBSCHER (Collegium, 1929, 427—437, 437—450).—(A) The flesh side of salted calfskins is sometimes covered with pink, orange-red, or red stains. In the last case the flesh side is slimy, the hair is easily removed after soaking, the fatty tissue in the adipose tissue is much decomposed, the surrounding fibres are attacked, and some damage is done to the grain. There are bacterial colonies in the epidermis, which is often destroyed. The extent of the bacterial growth and the damage to the hair roots vary directly as the extent of bright red stains on the flesh side. Increased loss of hide substance was observed in the soak liquors and lime liquors. (B) *Sarcina lutea* and *auriantica*, *Micrococcus tetragenus* and *roseus*, *Bacillus subtilis*, *Proteus*, and *Actinomyces* have been isolated from stained skins. These include bacteria which form red and yellow dyestuffs, respectively. *Actinomyces* which split up fats are known, and there is evidence that the *M. tetragenus* possess fat-splitting properties. The growth of these microorganisms was stimulated by the presence of sodium chloride in the case of *S. lutea* and *M. roseus*. As much as 16% of sodium chloride was required to prevent the growth of *S. lutea* and *auriantica* and *M. roseus*, and only 8% for *Proteus*, *Actinomyces*, and *B. subtilis*; 16% of sodium chloride considerably repressed the growth of *M. tetragenus*. The growth of these organisms was prevented at p_H 11.0, which requires an addition of 3% of calcined sodium carbonate to the sodium chloride used in salting.

D. WOODROFFE.

Sedimentation method of determining the insoluble matter in tannin analysis. V. KUBELKA and

V. NĚMEC (Collegium, 1929, 421—427).—The tan liquor of analytical strength is placed in a glass cylinder (diam. 10 cm.) through the upper part of the wall of which passes a tube fitted with a stopcock. The inside end of the tube bends down and terminates at a point called the zero mark. The liquor fills the cylinder to a mark 15 mm. above this. After keeping for 24 hrs. at 18—20°, particles larger than 1 μ have settled out of the tan liquor above the zero mark; the liquor can be withdrawn through the tube and used for the determination of the total soluble matter. Determinations of insoluble matter should be made on tan liquors of different strengths by this method. The results obtained at analytical strength are much less than those given by the filter-paper or filter-candle methods. A chestnut extract showed a maximum amount of sediment at a concentration of 22.5 g./litre, whilst the sediment from a quebracho extract increased continuously with increased concentration.

D. WOODROFFE.

Glue and gelatin. O. GERNGROSS (Z. angew. Chem., 1929, 42, 968—972).—The properties desired in both these substances are due to the gluten content. This can be judged by the definition of interference rings in the X-ray spectrum, but the method is too tedious for practical use. The water content is best determined by dissolving in a small quantity of water and evaporating so that a thin, easily dried film is obtained. The p_H value of gelatin can be determined potentiometrically, but glue must be titrated with nitrophenol indicators by Michaelis' method. The gluten content of gelatin can be roughly judged by measuring the bulk of precipitate formed by adding 2*N*-sulphosalicylic acid to its aqueous solution. A recent method has also been developed by which the gluten content is determined from the rate of gelatinisation under given conditions. Methods for the determination of viscosity, m.p., bending strength, etc. are described.

C. IRWIN.

See also A., Nov., 1235, **Behaviour of swollen gelatin in water vapour** (SCHREINEMAKERS). 1335, **Swelling of cod-fish skins** (KERNOT and KNAGGS).

XVI.—AGRICULTURE.

Determination of the maximum molecular moisture-holding capacity of soils by centrifuging, and the characterisation of the mechanical properties of soils by this determination. A. F. LEBEDEV (Pochvovedenie, 1928, 23, No. 1—2, 49—69).—The force necessary to remove gravitational water from various types of soil has been determined. A classification of soils based on the molecular moisture-holding capacity checks well with the classification based on mechanical analyses.

CHEMICAL ABSTRACTS.

Field trials and Neubauer experiments [for determining fertiliser requirements of soils]. F. SCHEFFER (Z. Pflanz. Düng., 1929, 8B, 501—515).—Relative practical values of the results of field trials and those of Neubauer's seedling method are compared and discussed. The work of Gerlach (B., 1929, 143) is considered in the light of the requisite limiting values. The differences in the nature of the factors measured by the two methods are discussed. A. G. POLLARD.

Fixation of phosphates in soils. C. O. WILLIAMS (Proc. Dept. Conf. Div. Chem., S. Africa, Series No. 100, 1929, 426—438).—The literature concerning soil acidity and phosphate fixation is reviewed and the general inference is drawn that the superiority of water-soluble phosphates over mineral phosphate fertilisers is largely discounted in the acid soils of Natal.

A. G. POLLARD.

[Simplification of] the citrate test [for the determination of water-soluble phosphate in super-phosphate]. A. SUCHIER (Z. angew. Chem., 1929, 42, 990—992).—The precipitate of magnesium ammonium phosphate in as crystalline a form as possible is prepared as usual, rapidly collected and washed on a glass filter, decomposed with a known excess of 0.5*N*-hydrochloric acid, and the excess titrated with methyl-orange and standard alkali. If the prescribed quantities are strictly adhered to the results do not vary more than 0.2% from those given by the gravimetric method.

C. IRWIN.

Decomposition of citric acid by soil. L. A. and A. L. DEAN (Soil Sci., 1929, 28, 281—287).—Certain Hawaiian soils are able to decompose citric acid with the production of acetone and carbon dioxide. The determination of citric-soluble phosphates is seriously affected thereby.

A. G. POLLARD.

Relation between the adsorbed and the exchangeable calcium and magnesium content of a soil four years after additions. W. H. MACINTYRE and K. B. SANDERS (Soil Sci., 1929, 28, 289—304).—Soils and subsoils were treated with lime, limestone, and dolomite and examined by means of lysimeters for their "fixation" of calcium and magnesium, and by Hissink's method for exchangeable bases. The amounts of calcium and magnesium "fixed" were consistently greater than those accounted for as exchangeable bases, the difference being more pronounced in surface soils than in subsoils. The ratio of exchangeable calcium to exchangeable magnesium was greater than the ratio of these bases in the rain-water percolates. It is suggested that a portion of the added lime etc. is combined with soil components other than the absorbing complex.

A. G. POLLARD.

Micro-organisms of and reactions occurring in farmyard manure made in different ways, with special reference to H. Krantz' method of preparing synthetic farmyard manure. W. GOETERS (Landw. Versuchsstat., 1929, 108, 1—60; Bied. Zentr., 1929, 58, 456—458).—The total number of micro-organisms in farmyard manure is greatly diminished both at high and at low temperatures, *i.e.*, 60—80° or 0°, whilst the greatest activity takes place at 20—40°. Higher temperatures kill most of the vegetative forms, only spores surviving. At 60° thermophilic organisms are active. Properly handled "synthetic" farmyard manure is poor in vegetative organisms, and mesophilic organisms are much reduced in numbers by heat in the first day after composting. Urea-decomposing bacteria develop heat-resisting forms and play an important part in dung ripening. The development of cellulose-decomposing organisms varies according as the conditions are anaerobic or aerobic, the anaerobic ones being less able to

withstand high temperatures. The number of heat-resistant cellulose-decomposing bacteria in synthetic farmyard manure is very low. The aerobic bacteria thrive best at 38—40°. The high bacterial content of the usual stall manure is reflected in its high loss of dry matter and plant foods, which is not the case with the synthetic material. The degree of nitrification of the nitrogen compounds in soil containing 1% of organic manure is 3 times as great in the internal layers as in the surface, and in the inside layers of synthetic manure 2—3 times as high as in the outside layers of farmyard manure. Plant yield increases in pots manured with hot-fermented manure (the Krantz method) were 2—4 times as large as when ordinary farmyard manure was used. In further pot experiments the "nitrogen efficiency" of the hot-fermented straw manure was 2—3 times, and of the hot-fermented peat manure 4 times, that of the ordinary material. E. HOLMES.

Manurial trials with town sewage in 1928. W. ZIELSTORFF, A. KELLER, and W. BRUTLER (Z. Pflanz. Düng., 1929, 8B, 481—501).—Treatment of field soils with sewage effluent resulted in increased crops of wheat, oats, potatoes, and pasture grasses. The fertiliser value of the effluent lies mainly in its nitrogen content. Considerable amounts of phosphate are also present, but the potash content is small. The digestibility of pastures was not greatly affected by treatment with sewage effluent, but the total crop per unit area was more than doubled. In pot experiments, clarification of the effluent before use had but little influence on the results.

A. G. POLLARD.

Oily plants. N. M. TULAIKOV (Zhur. Opit. Agron. Yugo Vostoka, 1928, 6, No. 2, 105—138).—Seasonal variations in the dry matter content of sunflower and flax were determined. The ash is highest in the leaves. The nitrogen content is high in the early period of growth, thereafter diminishing. The accumulation of ash and nitrogen in flax continues up to harvesting.

CHEMICAL ABSTRACTS.

Relation of calcium to the nodulation of soya beans on acid and neutral soils. W. A. ALBRECHT and F. L. DAVIS (Soil Sci., 1929, 28, 261—279).—The improved inoculation of soya-bean roots with *B. radicola*, brought about by the liming of acid soils, is due to the addition of calcium salts as well as the reduction of soil acidity. The natural fertility and previous cultural history of the soil are also important factors. No apparent relationship exists between the increased nodulation and the hydrogen-ion concentration or the amount of electro-dialysable calcium of the soil. Soya-bean organisms in clay suspensions are carried down with the coagulum during flocculation with calcium chloride solution, but only to an insignificant extent when potassium chloride is used. A. G. POLLARD.

Müntz nitrification process. A. P. FORJAZ (Compt. rend., 1929, 189, 585—586).—The roots of *Cytisus proliferus* (var. *palmensis*) were separated from the nodules accompanying them, each portion was incinerated separately, and the flame spectra of the hydrochloric acid extracts were examined. Lines not common to both preparations were due to molybdenum, nickel, and

cobalt (?), and these are considered specific elements in the biocatalytic fixation of atmospheric nitrogen.

J. GRANT.

Sulphur dioxide content of air and its influence on the plant. G. KÖCK, P. RECKENDORFER, and F. BERAN (Forts. Landw., 1929, 4, [6], 170; Bied. Zentr., 1929, 58, 467—470).—Systematic experiments were carried out in 1928 to test the effects of sulphur dioxide-air mixtures, varying from 1 pt. in 200,000 to 1 pt. in 20,000 pts. of air, on the growth and composition of red clover. In all, 32 plots, of approx. 2 m. \times 1.5 m., and covered with glass, were employed, 16 as controls, and 16 under different treatments. Sulphur dioxide in concentrations of 1 pt. per 200,000 and per 100,000 kept in contact with the plants for 1—4 hrs. had no significant effect on the amount of SO_2 in the dry matter of the plants when examined either 1 hr. or 2 days after fumigation, nor on the total digestible or indigestible protein of the clover. A significant increase in SO_2 in the plants was found when the concentration of sulphur dioxide was increased to 1 pt. per 50,000 and per 20,000 of air, but there was no variation in the protein content. Under the conditions of the experiment no deleterious effect on the fodder value of the clover was observable.

E. HOLMES.

Mechanism of plant nutrient intake. W. W. BUKEWITSCH (Landw. Jahrb., 1929, 69, 521—541; Bied. Zentr., 1929, 58, 460—462).—A general discussion of the mechanism of the diffusion of nutrient salts through colloidal membranes and the bearing of the results on plant nutrient intake problems. From experiments with oats and buckwheat, it is found that increase in the acidity of the nutrient medium increases the nitrogen-hunger of the plant when the source of nitrogen is ammoniacal. With deficiency of phosphoric acid the plant shows minimal development in alkaline solutions. In the case of potash deficiency yield of oats is higher when the nutrient solution is alkaline. E. HOLMES.

Potash manuring of sugar beet. E. OHLY and T. REMY (Landw. Jahrb., 1929, 69, 401—427; Bied. Zentr., 1929, 58, 452—454).—It has been shown at Poppelsdorf that 1 kg. of nitrogen gives a 170-kg., and 1 kg. of potash a 70-kg., increase in yield of sugar beet. The increase due to nitrogen is obtained at the expense of quality, whilst with potash quality is maintained. Potash bound in soil complexes is more available to old than to young beet plants. After June either kainit or potash manurial salts gave quicker growth and brighter foliage of beet than potassium sulphate, and no soil acidification was apparent from the sulphate dressings. The proportion of leaf to root is increased more by kainit than by 40% potash salts; a tentative explanation is advanced. The original paper evidently discusses the potash requirements of the young plant at length.

E. HOLMES.

Effect of manuring on the quality of potatoes. E. GLASER and KLEBERGER (Landw. Jahrb., 1929, 69, 543—554; Bied. Zentr., 1929, 58, 454—456).—From experiments carried out during three years on a light, sandy loess-loam rich in humus, and on a heavy, sticky loam, both with average amounts of lime, under varying weather conditions, it is concluded that lack of phosphoric

acid and potash has important effects on total yield, and particularly on the yield of tubers, and the addition of these materials is often more important than soil type in potato cultivation. Increase in the application of phosphoric acid and potash gave as great increases in yield as increase in the complete dressings, including nitrogen. Heavy dressings of farmyard manure had neither as good an effect on the total yield nor on the yield of tubers as the complete mineral fertiliser, but additional dressings of phosphoric acid as super-phosphate increased yields, and gave tubers of better quality. It is concluded that for potatoes cultivated for industrial and fodder purposes, increased amounts of phosphoric acid and potash must be used, whilst for potatoes for ordinary consumption increased amounts of a complete fertiliser are advisable. The normal dressing used contained 15 kg. N, 10 kg. P_2O_5 , and 20 kg. K_2O per hectare.

E. HOLMES.

See also A., Nov., 1256, **Determination of traces of iodine in organic substances** (SCHWAIBOLD). **Colorimetric determination of phosphorus** (VÁSÁRHELYI). **Determination of arsenic by the Gutzeit method** (NELLER).

PATENTS.

Manufacture of fertilisers. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, and E. URBAIN (B.P. 305,132, 18.1.29. Fr., 31.1.28).—A mixture of potassium and ammonium chlorides obtained as described in B.P. 210,399 (B., 1924, 530) is heated at 220—260° with phosphoric acid (d 1.6) sufficient to form the compounds $\text{M}'\text{H}_5\text{P}_2\text{O}_8$. When all the hydrochloric acid has been expelled, water is added until the temperature falls to 150° and gaseous ammonia is then passed into the vessel with vigorous stirring until a dry powdery mass of crystals of ammonium and potassium dihydrogen phosphates is obtained. Further quantities of ammonia may be added, after cooling to 50°, to form the monohydrogen phosphates.

A. R. POWELL.

Manufacture of fertilisers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,842 and 318,909, 10.3.28).—The products claimed in B.P. 236,494 (B., 1925, 732) are modified by replacing, completely or partly in every case, (A) the urea by ammonium sulphate or mixtures of it with sodium nitrate, and, if desired, the potassium nitrate and/or potassium sulphate by potassium chloride to yield a product with $\text{N}:\text{P}_2\text{O}_5:\text{K}_2\text{O}::1:0.9:1.2$, or (B) the urea by sodium nitrate, and, if desired, the potassium nitrate and/or potassium sulphate by potassium chloride to yield a product with $\text{N}:\text{P}_2\text{O}_5:\text{K}_2\text{O}::1:0.6:1.5$.

L. A. COLES.

Fertilisers (B.P. 318,404).—See II.

XVII.—SUGARS; STARCHES; GUMS.

Manuring of sugar beet. OHLY and REMY.—See XVI.

See also A., Nov., 1248, **Effect of X-rays on crystalline and dissolved sucrose** (REINHARD and TUCKER). 1273, **Acids from pectin of sugar beet** (EHRlich and SCHUBERT). 1281, **Solubility of starch**

in water below the swelling temperature (SAMEC and TOMAZO). 1337, Invertase from honey (PAPADAKIS).

PATENTS.

Treatment of molasses for yeast growth. DISTILLERS Co., LTD., W. G. BENNETT, and A. M. PEAKE (B.P. 319,641, 25.6.28).—For the removal of substances inhibiting yeast growth and of colouring matters, an adsorbent which is either ground-nut cake or calcium carbonate is added to neutral or slightly acid molasses. At the same time, or subsequently, a protein precipitant, such as tannin, is added together with a protein, *e.g.*, horse-blood serum, if the molasses is deficient in these.

C. RANKEN.

Cultivation of micro-organisms (B.P. 318,649).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Electrometric titration [of worts]. F. EMSLANDER (Woch. Brau., 1929, 46, 407—409).—The titration vessel consists of a U-tube, of which the two arms are connected by a wide tube immediately above the curved part. Just above this, one arm is widened to allow of the addition of the titration reagent without marked increase in depth of liquid. The wide arm carries the bridge and a spiral rotating stirrer, by which the contents of the vessel are caused to mix by circulating through the bend and connecting tube. The narrow arm is closed by a rubber stopper fitted with an inlet tube for hydrogen, and with a gold electrode plated with iridium and coated with platinum-black. This just dips into the liquid. The excess of hydrogen escapes through the connecting tube to the wide, open arm, to which the titration reagent is added. Equilibrium is very rapidly attained. The author titrates to p_H 4.5, 6.8, 8.3, and 9.18.

F. E. DAY.

Titration [of worts and beers] by stages. L. HEINTZ (Woch. Brau., 1929, 46, 393—396).—Since many beers have a reaction below p_H 4.27, the limit proposed by Kolbach (B., 1929, 373) for the acid titration, it is considered that the acid titration should be carried to p_H 4.00, especially as buffering is strong at this reaction. For the next stage p_H 5.50 is preferred to p_H 5.67 as being more nearly intermediate between p_H 4.00 and 7.07, and sufficiently removed from the original p_H of most worts (about 5.7—5.8) to give a check on the buffering. In the formol titration the additional alkali needed to reattain the final end-point at p_H 9.18 is independent of treatment or otherwise of the wort or beer with baryta and of the reaction at which the formalin is added. With 12.5% worts about 55% of the original titration value between p_H 7.07 and 9.18 remains after treatment with baryta, and as this is certainly not due to amino-acids capable of reacting with formalin, the inclusion of this with the formol titration as total amino-nitrogen is incorrect. The increased alkali used after addition of formalin is quite well-defined, and is best expressed as "formol nitrogen." The titration value after precipitation with baryta may be employed to give a further insight into the nature of the buffering substances. Details of

procedure are given, and the permanent standards of Kolthoff (B., 1922, 235 A) are found satisfactory and convenient, a single standard sufficing for methyl-orange at p_H 4.00 and for methyl-red at p_H 5.62.

F. E. DAY.

Decomposition of rye straw and maize stalks. HORVÁTH.—See V.

See also A., Nov., 1261, **Apparatus for distillation of sensitive solutions in a vacuum** (KRAUT and others). 1262, **Scopometer** (EXTON). 1266, **Determination of ethyl alcohol by chromic oxidation** (SEMICHON and FLANZY). 1339, **Physiological selection of enzymes by alcohol** (SEMICHON). **Determination of gas in fermentations** (RAYMOND). **Relation of acetyl group to fermentation** (HIGASHI). **Effect of various preparations on baker's and brewer's yeasts** (WILLIAMS and others). **Separation of nitrogen during yeast fermentations** (IVANOV and KRUPKINA). **Sulphite fermentation** (POLAK). 1340, **Behaviour of sodium glucosesulphite with yeast** (NEUBERG).

PATENTS.

Influencing the speed of enzymic reactions. F. F. NORD (B.P. 291,443, 18.5.28. Ger., 3.6.27).—Enzymes in the form of colloidal solutions or contained in cells are activated and rendered more resistant by exposure to an unsaturated hydrocarbon such as acetylene, ethylene, etc. An adsorption layer is formed on the surface of the enzyme and, in the case of enzymes contained in cells, the permeability of the cells is increased.

C. RANKEN.

[Solutions for the] cultivation of micro-organisms. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 318,649, 8.3., 23.6., and 10.9.28).—Peat is hydrolysed by heating with dilute acids, and the liquor, after filtration, is purified by the addition of, *e.g.*, calcium, barium, or zinc oxide in quantity sufficient to neutralise the acid and to precipitate humus substances, but not to react with the sugars present, yielding a solution which, after filtration and preferably after the addition of potassium and magnesium salts etc., is suitable for the cultivation of yeast etc. Removal of the humus substances is facilitated by aeration of the solution and by the precipitation of phosphates in it, *e.g.*, by adding phosphoric acid or soluble phosphates before the addition of calcium oxide etc. Stronger solutions are obtained by using the same acid liquor for the successive extraction of two or more batches of peat.

L. A. COLES.

Production of butyl alcohol and acetone by fermentation. DISTILLERS Co., LTD., and H. B. HUTCHINSON (B.P. 319,642, 25.6.28).—Starchy material, such as manioc, is fermented with added ammonium acetate to increase the yield of acetone, and with added ammonium lactate to increase the yield of butyl alcohol. The addition of such nitrogenous nutrients decreases the yield of ethyl alcohol.

C. RANKEN.

Manufacture of yeast. W. H. F. BÜHRIG, Assr. to FLEISCHMANN Co. (U.S.P. 1,730,876, 8.10.29. Appl. 14.5.25).—See B.P. 252,193; B., 1927, 857.

Molasses for yeast growth (B.P. 319,641).—See XVII.

XIX.—FOODS.

Detection of milk pasteurised by the holding method. R. HOCK (Milch. Zentr., 1929, 58, 309—311).—Milk which has been pasteurised by heating at 60–65° for $\frac{1}{2}$ hr. can be detected by the volume of serum which separates when the milk is treated with rennet. A 50-c.c. sample is brought to a Soxhlet–Henkel acidity of 5 by addition of 0.25*N*-caustic soda. To the milk warmed to 35–40° is added $\frac{1}{2}$ c.c. of dilute rennet essence (1:10), and, after shaking, the mixture is incubated in Petri dishes at 37° for 14 hrs. With unpasteurised milk the volume of serum is more than 40 c.c., but if the milk has been heated to 60–65° the volume is less than 40 c.c. The maximum found for pasteurised milk was 36 c.c., and the minimum for fresh milk 41 c.c. The nature of the precipitated casein affords further evidence of the heat-treatment the milk has undergone. Milk which has been heated to 70° or over gives a voluminous white curd with slight bubble formation due to aerogenic bacteria, whereas fresh milk or milk which has not been heated above 65° gives a thin, dense, fissured coagulum which adheres firmly to the bottom of the dish. The curds from milk heated to 65° or over can be readily poured off with the serum.

H. J. DOWDEN.

Baking quality of wheat varieties. F. SCHNELLE (Wiss. Arch. Landw., Abt. A, Pflanz. I, 1929, 471—555; Bied. Zentr., 1929, 58, 462—464).—The baking quality of various American, English, and European varieties of wheats is discussed. Differences in baking quality due to season are best reflected in the 1000-corn weight and litre weight, differences between varieties by the sp. gr. In general, rising power of a dough and the nitrogen content of the grain can be correlated with baking quality, but seasonal variations may alter the latter conclusion.

E. HOLMES.

Determination of vitamins-A, -B, and -C in collards and turnip greens. G. W. BURTON (J. Home Econ., 1928, 20, 35—42).—Cooking turnip greens or collards in water caused a loss of 10% or 40–50%, respectively, in the vitamin-B content; the vitamin-A was unaffected. Biological determinations of vitamins-A, -B, and -C are recorded.

CHEMICAL ABSTRACTS.

Vitamin-A content of avocados. L. S. WEATHERBY, J. E. YONTZ, and R. V. WATSON (J. Home Econ., 1929, 21, 360—364).—The avocado is rich in vitamin-A; biological tests are recorded. CHEMICAL ABSTRACTS.

Determination of free ammonia in decomposing meat. MUCHLINSKY (Z. Fleischn. Milch-Hyg., 1929, 39, 189—190; Chem. Zentr., 1929, i, 2712).—The minced material (10 g.) is distilled for 1.5–2 hrs. with 20 pts. of carbon tetrachloride, in a flask provided with a ground-in adapter, delivery tube, and pipette-condenser, into 0.01*N*-hydrochloric acid. A. A. ELDRIDGE.

Packaging and quick-freezing perishable flesh products. III. Sanitary measures in fish-dressing plant. C. BIRDSEYE (Ind. Eng. Chem., 1929, 21,

854—857; cf. B., 1929, 697, 621).—When caught the fish is immediately gutted and boxed in ice for placing in cold store on reaching land. In the processing of haddock fillets, the fish is scaled under sea water, washed, filleted, skinned if not already scaled, brined, wrapped, and frozen very rapidly. To mitigate bacterial infection, which is high and increases during the processing, the plant is supplied with chlorinated sea water containing originally 7 p.p.m. and 1–3 p.p.m. by the time the wash-tank is reached. Sea water is preferable to fresh water since the former increases the salt content of the fish and, by partially coagulating the surface protein, lessens the absorption of protein by the water. The free chlorine content of the brine is maintained at 1–3 p.p.m. by hourly additions of sodium hypochlorite, and by this treatment the bacterial count of the fillets is reduced by 50%. Monel metal and nickel-chromium or chromium-plated equipment is used extensively, and the plant and buildings are cleansed with chlorinated sea water or hypochlorite solution.

H. J. DOWDEN.

Fats. VAN RAALTE. **Murumurú fat.** SARAIVA. **Pecan oil.** JAMIESON and GERTLER.—See XII.

PATENTS.

Preserving liquid nutriment such as milk and the like. F. SEIDAL and A. GIANI (B.P. 294,903, 30.7.28. Austr., 28.7.27).—The milk is sterilised, without affecting the flavour etc., by preheating to 63° and then passing through an evacuated vessel wherein it is subjected to a magnetic field and also to an electric current at 440 volts passing through the milk and thence from its surface through the rarified air to another electrode. Flow is continuous, the milk remaining in the vessel for about 15 min. Increased vitamin content is claimed.

E. B. HUGHES.

Preservation of the yolk and white of eggs, and of a mixture of the two. M. D. C. MICHAUD, and COMP. OLIVIER (B.P. 293,794, 2.7.28. Fr., 12.7.27).—Egg yolk or white or whole egg is mixed with sucrose (15–50%) or sodium chloride (4–17%), or mixtures of the two, with or without lactic acid (0.2–1%), and the product is dehydrated *in vacuo* to 70–90% of total solids and then packed in cans in an atmosphere of carbon dioxide and/or nitrogen.

E. B. HUGHES.

Sterilising and drying of fish materials and fish waste. C. DOWNS and R. A. BELLWOOD (B.P. 320,107, 5.7.28).—The vapours from the fish-drying plant are passed through a heat exchanger whereby the air used for drying is preheated.

E. B. HUGHES.

Production of egg products. A. K. EPSTEIN, Assr. to EMULSOL CORP. (U.S.P. 1,730,879, 8.10.29. Appl., 24.12.26).—See B.P. 305,844; B., 1929, 301.

Treatment [separation of leaf and stalk] of tea. C. S. BATEMAN (B.P. 319,843, 4.7.28).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Preserving chloroform. N. SCHOORL (Pharm. Weekblad., 1929, 66, 880).—The addition of anhydrous citric or oxalic acid proposed by Oberhard (B., 1929, 338) is

found not to prevent oxidation of chloroform on keeping, even in sealed tubes.

S. I. LEVY.

Evaluation of vitamin-D preparations. A. SCHEUNERT and M. SCHIEBLICH (Klin. Woch., 1929, 8, 699; Chem. Zentr., 1929, i, 2800—2801).—A biological method is described, and comparative values for commercial preparations are recorded. A. A. ELDRIDGE.

Degree of hygroscopicity of tobacco. G. O. GABEL and G. KIPRIANOV (Ukraine Chem. J., 1929, 4, [Tech.], 117—134).—The degree of hygroscopicity of various grades of tobacco was determined at temperatures of 20°, 25°, and 30°, and humidities of 60, 70, 80, and 90%. With a humidity of 60% and temperatures of 25° and 30°, most tobaccos gradually lose moisture, the degree of hygroscopicity falling from 8.35—14.11% to 6.5—7.5%. Temperature changes of 5° and variations in humidity of 5% had no appreciable effect. At 20° and 25° and with 70% humidity most tobaccos with an initial degree of hygroscopicity of 7—8% tend to absorb moisture very slowly. With 80% humidity moisture is absorbed markedly at 20°, 25°, and 30°. Variations in temperature of 10° influence the rate of absorption, but not its final value. The rate is very high during the first day, but decreases rapidly, the lower grades of tobacco reaching their final values earlier than the higher ones. The experimental results were much less accurate than with the other sets of conditions. The hygroscopicity with a humidity of 90% was very high (20—33.2%), the lower grades having the lower value. As in the case of 80% humidity, the experimental results did not agree closely, and experiments showed that no apparent biochemical change was taking place in the leaves, the discrepancies being probably due to variations in the physical condition of individual leaves.

M. ZVEGINTZOV.

Extraction of nicotine from tobacco dust by kerosene. G. O. GABEL and G. I. KIPRIANOV (Ukraine Chem. J., 1929, 4, [Tech.], 109—116).—The tobacco dust was treated with calcium hydroxide solution, and the aqueous suspension shaken with kerosene. It was found that by using 700 c.c. of kerosene per 100 g. of tobacco (nicotine content 0.38—1.38%), and extracting at 80°, about 96% of the nicotine could be removed. The efficiency of the extraction decreased when the concentration of nicotine in the kerosene layer reached 2.83 g. On shaking the kerosene solution with sulphuric acid in equimolecular proportions 85% only of the nicotine could be extracted, the remainder requiring acid of 50% concentration. As nicotine sulphate is insoluble in kerosene, it was suspected that another basic substance was present. On treating the first acid extract with alkali a colourless liquid showing feebly basic properties and giving a picrate, m.p. 207—209°, was obtained. Its nature, however, could not be identified.

M. ZVEGINTZOV.

Use of kerosene for determination of the nicotine content in tobacco. V. D. KUTZENOS (Ukraine Chem. J., 1929, 4, [Tech.], 135—139; cf. preceding abstract).—A résumé of the existing methods of determining nicotine in tobacco, with a description of an experimental comparison of Tote's and Keller's methods. It was found

that extraction with light petroleum could in both methods be successfully replaced by extraction with kerosene.

M. ZVEGINTZOV.

Examination of samples of opium of varying composition. E. W. ANSINGH (Pharm. Weekblad, 1929, 66, 865—872).—The difficulties introduced into the analysis by the presence of considerable quantities of various gums may be overcome by a new method involving extraction of the morphine with 96% alcohol containing tartaric acid. An examination of results obtained by using varying quantities of quicklime showed that very low results may be recorded if insufficient lime is used, e.g., if the lime is partly slaked or contains magnesia.

S. I. LEVY.

Analysis of Peruvian bark, and the liquid extracts prepared therefrom. J. A. C. VAN PINXTEREN (Pharm. Weekblad, 1929, 66, 929—961).—The method of Commelin (Med. Kinaproefstat., 1912) for determinations of total alkaloids in the bark, and of quinine and cinchonidine, is discussed, and the objections raised by various authors are considered. In the crystallisation of quinine and cinchonidine tartrates, a double tartrate containing 1 mol. of quinine to 2 mols. of cinchonidine may be formed; this loses all its water of crystallisation in 3—4 hrs. at 110°. In the preparation of the liquid extract changes occur in the composition of the alkaloid mixture, though no specific action on any alkaloid group by hydrochloric acid could be detected. In the method of preparing the extract laid down by the Dutch pharmacopoeia, the hydrogen-ion concentration changes irregularly, on account of the strong buffer action of constituents of the bark, and effective decomposition of the tannates occurs only after these have been removed in the early stages of the percolation. A good extraction can be obtained with the use of only 70% of the quantity of hydrochloric acid specified by the pharmacopoeia.

S. I. LEVY.

Essential oils of the huahuan (*Laurelia serrata*). G. FESTER and J. SALGADO (Z. angew. Chem., 1929, 42, 987—988).—The bark of this tree, which is a native of Southern Chile, yielded on distillation 2.7% and the leaves 3.8% of essential oil calculated on dry weight. The bark oil has d_{4}^{15} 1.0426, n_D^{20} 1.52182, acid value 0, ester value 6.9, and consists chiefly of safrole. The leaf oil has d_{4}^{15} 0.9469, n_D^{20} 1.49632, α —12.5°, acid value 1.4, ester value 9.3, and contains a small quantity of sulphur. The commercial extraction of the oil in competition with camphor and sassafras oil is considered possible.

C. IRWIN.

Pyrethrum flowers. I. Determination of active principles. C. B. GNADINGER and C. S. CORL (J. Amer. Chem. Soc., 1929, 51, 3054—3064).—Using a modification of the method described by Staudinger and Ruzicka (A., 1924, i, 700), pyrethrins I and II have been isolated from the flowers of *Chrysanthemum cinerariæfolium*, Bocc., and the toxicity of these towards cockroaches has been determined; the results agree with those of Staudinger and Ruzicka. Alcoholic solutions of pyrethrins I and II, like pyrethrolone (loc. cit.), reduce alkaline copper solutions; the reducing powers are considerably less than those of dextrose.

A modified Folin method (A., 1926, 648) is described which is applicable to the determination of the pyrethrins in the flowers. This involves extraction of the active principles with light petroleum, treatment of the alcoholic solution of the residue from the extract with lead acetate, and subsequent reduction of an alkaline copper sulphate solution with the filtrate. The reduced solution is compared colorimetrically with a similar solution using dextrose. For 16 samples of flowers examined the amount of pyrethrins I and II varied between 0.4 and 1.21%; the stems contain 0—0.04%. Daisy flowers (*Chrysanthemum leucanthemum*) contain no pyrethrins. Japanese and Dalmatian pyrethrum flowers contain the same active principles.

H. BURTON.

Preparation of some perfumes and flavouring extracts from furfuraldehyde and its derivatives. Esters of β -furylacrylic acid. H. GILMAN and G. F. WRIGHT (Iowa State Coll. J. Sci., 1929, 3, 109—112).—A study of the odour and taste of methyl, ethyl, propyl, isopropyl, butyl, amyl, benzyl, and furfuryl β -furylacrylate, the preparation of which is described.

CHEMICAL ABSTRACTS.

Anda-assú oil. GURGEL and RAMOS.—See XII.
Vitamins in collards and turnip greens. BURTON.
Vitamin-A in avocados. WEATHERBY and others.—See XIX.

See also A., Nov., 1213, **Spectrographic studies of hormones** (GRAUBNER). **Adsorption spectra of hormones and allied substances** (MARCHLEWSKI and SKARIZYŃSKI). 1255, **Limits of applicability of indicators in p_H determinations** (EISENBRAND). 1295, **Phthalide derivatives** (MAYER and others). 1306, **Cyclamin** (BUREŠ and BERGAUER). 1308, **Crystalline compounds from essential oils** (WIENHAUS and SCHOLZ). 1318, **Lupin alkaloids** (CLEMO and RAPER). **Identification of atropine** (FULTON). 1319, **Microchemical reactions of sparteine** (WAGENAAR). **Identity of pectinin with carnegin** (SPÄTH and KUFFNER). **Angostura alkaloids** (SPÄTH and PIKL). 1320, **Potentiometric determination of morphine** (MARICQ). **Strychnos alkaloids** (LEUCHS and others). 1321, **Benzthiazole arsenicals** (BOGERT and HESS). 1323, **Simultaneous determination of nitrogen and mercury in organic mercury compounds** (HERNLER).

PATENTS.

Manufacture of physiologically active substances [from internal secretive organs]. SCHERING-KAHLBAUM A.-G. (B.P. 291,006, Addn. B.P. 294,651, and 320,278, [A] 16.5.28, [B] 25.7.28, [C] 10.12.28. Ger., [A] 23.5.27, [B] 29.7.27).—(A) Extracts from, e.g., placenta or testicles are stirred with an alkaline-earth hydroxide and, if desired, a non-reacting adsorbent, e.g., kaolin, silica gel, in the presence of a mixture of water with not more than 50% of an organic solvent, e.g., methyl alcohol, acetone, and at a temperature not above 80°. After filtration, the solution is concentrated to expel the organic solvent and the residue is extracted with ether to recover the desired product. (B) The finely-divided organs are themselves treated, instead of

the extracts, as described in (A). In (C) the dried, finely-divided internal organs are treated as in (B).

L. A. COLES.

Manufacture of germ-gland hormones from vegetable organisms. SCHERING-KAHLBAUM A.-G. (B.P. 294,650, 25.7.28. Ger., 29.7.27).—Vegetable organisms, e.g., yeast or finely-ground wheat, or hormones previously extracted from them, are treated in suspension in water or in a mixture of an organic solvent and water with an alkaline-earth hydroxide and, if desired, a non-reacting adsorbent, e.g., kaolin or silica gel. After filtration, the solution is, e.g., evaporated to dryness or extracted with ether to recover the hormone (cf. B.P. 271,492; B., 1928, 769).

L. A. COLES.

Preparation of therapeutic substances from tubercle bacilli. I. SAISHO (B.P. 320,048, 2.5.28).—Tubercle bacillus preparations, after drying by suction on a filter, are ground for 5—10 min. with 20—37% hydrochloric acid or with 60—94% sulphuric acid and the acid, diluted if desired with physiological salt solution, is removed by centrifuging followed by repeated washing with physiological salt solution. The product, which is free from waxes, is diluted with physiological salt solution containing 0.5% of phenol to the strength required for injection.

L. A. COLES.

Manufacture of o-hydroxybenzylaminearsinic acids and their aroyl derivatives. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 318,315, 5.6.28).—*N*-Hydroxymethyl derivatives of aromatic amides (benzamide, phthalimide) are condensed with a *p*-nitrophenol, which may contain an *o*-substituent, to give the corresponding acylated nitrohydroxybenzylamine, which is reduced and converted by Bart's reaction into the hydroxybenzylaminearsinic acid, the acyl group being subsequently removed by hydrolysis if desired. Instead of a *p*-nitrophenol a *p*-hydroxyphenylarsinic acid may be used. The products are therapeutically valuable and have no neurotoxic properties. Examples are: 2-hydroxybenzylamine-5-arsinic acid, m.p. 290° (benzoyl derivative, m.p. 220—221°; phthaloyl derivative, m.p. 276°); 3-aminomethyl-*o*-cresol-5-arsinic acid, m.p. above 290° (phthaloyl derivative, m.p. 273°); phthaloyl derivative, m.p. 263—264°, of 6-chloro-2-aminomethylphenol-4-arsinic acid. The phthaloyl derivatives, m.p. 225—226° and 95—97°, respectively, of 5-nitro- and 5-amino-2-hydroxybenzylamines are described.

C. HOLLINS.

Manufacture of ointments, particularly water ointments. F. MERZ (U.S.P. 1,732,105, 15.10.29. Appl., 28.9.26. Ger., 30.3.26).—See B.P. 268,719; B., 1928, 769.

Separation of particles suspended in liquids (B.P. 319,629).—See I. **Antiseptic solutions** (B.P. 320,096).—See XI. **Medicated soaps** (B.P. 319,832 and U.S.P. 1,731,551).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES

Grain growth in silver halide precipitates. SHEPPARD and LAMBERT.—See VII.

See also A., Nov., 1248, **Colour selectivity of photo-chloride** (WEIGERT and ELVEGARD). **Colour selectivity in dye systems** (WEIGERT and NAKASHIMA). 1249, **Daylight photometry** (ATKINS and POOLE).

PATENTS.

Films for colour photography and cinematography. SOC. CIVILE POUR L'ÉTUDE DE LA PHOTOGRAPHIE ET DE LA CINÉMATOGRAPHIE EN COULEURS (B.P. 293,306, 29.6.28. Fr., 2.7.27).—Halation is eliminated on lenticular films, *e.g.*, those described in B.P. 10,611 (B., 1910, 720), by introducing into the sensitive emulsion insoluble or soluble coloured material capable of rendering non-actinic the rays reflected by the surface of the emulsion in contact with the support, *e.g.*, silver chromate, indulines, tartrazine.

L. A. COLES.

Production of gelatin relief plates or films for production of dye imbibition prints. W. C. FAIRWEATHER. From JOS-PE FARBENPHOTO-GES.M.B.H. (B.P. 319,077, 20.6.28).—Unripe gelatin-silver bromide emulsions of 5–8° Scheiner are used for the production of relief by tanning and subsequent removal of the untanned gelatin, the emulsions containing also a hardening agent, *e.g.*, alum, substances to ensure dissolution of the untanned gelatin, *e.g.*, ammonium carbonate, material to break up the homogeneity of the layer, *e.g.*, resin, casein, salicylic acid, and, preferably, potassium iodide.

L. A. COLES.

Selector screens for colour photography and cinematography. SOC. FRANÇ. CINÉCHROMATIQUE (PROC. R. BERTHON), Assees. of SOC. CIVILE POUR L'ÉTUDE DE LA PHOTOGRAPHIE ET CINÉMATOGRAPHIE EN COULEURS (B.P. 294,493, 23.7.28. Fr., 22.7.27).

XXII.—EXPLOSIVES; MATCHES.

Pentaerythritol tetranitrate and its mixtures with nitroglycerin ("Penthrinite") as filling materials for projectiles. A. STETTBACHER (Chem.-Ztg., 1929, 53, 533–534, 554–556).—Pentaerythritol tetranitrate ("Penthrite") is soluble in nitroglycerin to the extent of 2% at 20° and 7–8% at 75–80°, and is therefore thoroughly wetted by the ester even in a finely-powdered form. The mixture of 80% of "Penthrite" and 20% of nitroglycerin ("Penthrinite") has a detonating velocity of 8600 m./sec. compared with 6700 for trinitrotoluene (T.N.T.) and 7800 for glycol dinitrate, and forms a plastic mass of maximum sp. gr. under 500–600 kg./cm.² compared with a pressure of 3000 kg./cm.² for T.N.T. The respective heats of explosion are 1449.5 and 950 kg.-cal./kg. and the volumes of gas formed at N.T.P. 762 and 690 litres/kg. Hence "Penthrinite" has a much greater shattering power and a higher safety factor than any of the nitro-aromatic compounds usually employed in high explosives. (Cf. B., 1928, 626.)

A. R. POWELL.

PATENTS.

Explosive. G. A. RUPP, Assr. to TROJAN POWDER Co. (U.S.P. 1,728,307, 17.9.29. Appl., 29.2.28).—An

aqueous suspension of a carbohydrate nitrate is stirred with a liquid immiscible with water until this liquid is absorbed by the ester.

L. A. COLES.

Flash-reducing agent for smokeless powders. W. T. INGRAHAM (U.S.P. 1,731,378, 15.10.29. Appl., 8.11.23).—A nitroglycerin-free propellant includes nitro-cellulose and rosin.

F. G. CROSSE.

Blasting cartridges. D. HODGE (B.P. 320,226, 5.10.28 and 1.1.29).

Manufacture of matches. H. A. GILL. From S. E. RAHE (B.P. 320,249, 22.10.28).

Acid-concentrating towers (B.P. 319,839).—See I. **Detection of explosive gases** (B.P. 319,231). **Eudiometry of explosive gases** (B.P. 300,281).—See II. **Concentration of nitric acid** (B.P. 320,125).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Methods of chemical analysis as applied to sewage and sewage effluents (Rep. Comm. Min. Health, 1929, 71 pp.).—In order to secure uniformity in the methods employed in obtaining the data by which the character of a sewage or sewage effluent is judged, the methods in use have been reviewed. The methods now recommended for general use are largely based on those included in the Fourth Report (pt. V) of the Royal Commission on Sewage Disposal. The Committee recommend *inter alia* the substitution of 0.0125*N*- for 0.125*N*-potassium permanganate in the "oxygen-absorption" test at 26.7° and the use of the Gooch crucible method for "suspended solids," and emphasise the importance of ammonia-free distilled water as a diluent when determining "free and saline ammonia."

C. JEPSON.

Phenols in effluent from lignite carbonisation. ROSIN and JUST.—See II. **Manurial trials with sewage.** ZIELSTORFF and others.—See XVI.

PATENTS.

Disposal of sewage or other waste organic matter. C. G. WIGLEY, Assr. to C. POTTS (U.S.P. 1,730,489, 8.10.29. Appl., 7.4.27).—The sewage matter, after being freed from excess moisture, is spread on the floor of a closed but aerated chamber where it is converted into a dry, granular, innocuous material by means of a fungoid growth, which may be propagated therein if the material is maintained at a suitable temperature, under aerobic conditions, and in the absence of sunlight.

C. JEPSON.

Refuse destructor installations. WOODALL-DUCKHAM (1920), LTD., and A. SCOTT (B.P. 320,140, 11.7.28).—The water-jacket surrounding the shaft of the furnace is kept at a temperature corresponding with the steam pressure in the boiler, either by causing the water from the boiler to flow into the jacket and back into the water-space of the boiler, or by connecting the upper part of the jacket with one end, and the lower part with the other end, of a closed duct which extends through a water-space of the boiler.

C. JEPSON.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY



DEC. 20, 1929.

I.—GENERAL; PLANT; MACHINERY.

Dissociation of water in steel tubes at high temperatures and pressures. C. H. FELLOWS (J. Amer. Water Works' Assoc., 1929, 21, 1373—1387).—In high-pressure boilers, particularly those in which distilled water is used and which are otherwise unprotected against corrosion, the corrosive action of pure water is of primary importance. Small-scale experiments having indicated that superheated steam contains more free hydrogen than saturated steam, a stream of water vapour was passed under varying conditions of temperature and pressure through tubes of either low-carbon steel (0.10% C) or chrome steel (16.35% Cr) and the amount of hydrogen determined before and after. It was found that a low-carbon steel was more liable to cause dissociation than chrome steel, that the rate of decomposition increases with rise of temperature and decreases as the thickness of oxide film formed increases, whilst within the limits used variation in steam pressure was without effect. The velocity of the steam through the tube was very considerably lower in the experiment than in actual practice, and since later investigations indicate that at still higher velocities no hydrogen whatever can be detected, it would seem that there is a definite relation between steam velocities (which are an important factor in determining the temperature of the metal) and oxidation of the latter due to the dissociation of steam.

C. JEPSON.

Determining thermal conductivity of heat-insulating materials. H. STILES (Chem. Met. Eng., 1929, 36, 625—626).—For the rapid determination of the thermal conductivity of such materials as wall-boards, two similar lagged brass cylindrical vessels are adapted to fit one above the other with the wall-board between the bottom of the upper and the cover of the lower, which serves as a boiler. Inside the upper vessel, which is an ice container, a small bakelite cylinder is soldered through the medium of a brass ring to the base; this cylinder has a tightly-fitting cover through which a glass tube with two bulbs and index marks is fitted. The ice container and central cylinder are filled with ice (weighted) and water, and when steady conditions have been attained, as indicated by the level in the glass tube, the vessel is put on the boiler. As the ice in the bakelite cylinder melts, the time taken by the water-level in falling between the index marks is observed. Repetition experiments may be conducted quickly by refilling the bakelite cylinder to above the upper index mark and continuing observations on the contraction.

C. A. KING.

Sampling of gas over mercury at a constant

rate. S. PEXTON and W. K. HUTCHISON (J.S.C.I., 1929, 48, 242—244 T).—A gas sampling device is described which is portable and self-contained, with mercury as the confining liquid. It will sample continuously for long periods at a rate which is constant to within 1%. The theory is described, and a full mathematical analysis is given.

Gas analysis. E. OTT (Gas- u. Wasserfach, 1929, 72, 862—863).—The author's portable apparatus has been improved, particularly in the means for determination of carbon monoxide, which is converted into carbon dioxide by the action of iodine pentoxide. For measuring the inert constituents of a gas mixture, the combustible material is eliminated in passing over copper oxide heated in a small electric furnace.

R. H. GRIFFITH.

Determinations of inert gas content of gas mixtures by means of calcium as an absorbent. M. LEATHERMAN and E. P. BARTLETT (Ind. Eng. Chem. [Anal.], 1929, 1, 223—225).—An apparatus is described which permits the analysis of various gas mixtures for the noble gases by a method involving absorption of all common gases by hot metallic calcium and the measurement of change of pressure at constant volume. Samples of air, nitrogen, and hydrogen have been analysed. Results are reproducible to within 0.002—0.003%. Sources of error and possible improvements in the apparatus are discussed.

H. S. GARLICK.

Liquorice root. HOUSEMAN and LACEY.—See XX,

PATENTS.

Furnaces. G. and D. M. NAISMITH (U.S.P. [A] 1,725,879, 27.8.29. Appl., 24.12.27, and (B) 1,727,898, 10.9.29. Appl., 12.12.27).—The furnaces are suitable for reheating ingots etc., that described in (A) being recuperative, and (B) regenerative. In (A) a horizontal mixing and combustion chamber comes between the recuperators and goods chamber; in the latter the hot gases pass downwards, but in the recuperators the exhaust gases and air pass to and fro horizontally, gases downwards, air upwards. In (B) the air-heating chambers are above the gas-heating chambers, and the air and gas mix before they enter the goods chamber and pass horizontally among the goods.

B. M. VENABLES.

Furnace for sintering, burning, and roasting of substances. E. C. LOESCHE (B.P. 298,651, 13.10.28. Ger., 14.10.27).—The material is fed upwards through the centre of a hearth, and is heated by a downward flame or electric arc immediately above the same point; the feed opening is therefore always covered with material, which may be allowed to form a cone from the

base of which the new material is pushed upwards to the apex and, after contact with the heat, rolls down the sides.

B. M. VENABLES.

[Reverberatory] furnaces. H. S. KNOWLES and W. BURKINSHAW (B.P. 318,748, 12.9.28).—The combustion chamber is divided into a number of compartments by transverse walls extending to the roof, but provided with ports for the burning gases. In front of each row of ports is a bridge wall so that the gases are deflected upwards on entering each compartment; at these points additional air is blown in vertically from below.

B. M. VENABLES.

Rotary [annular] furnaces. TROCKNUNGS-, VER-SCHWELUNGS-, U. VERGASUNGS-GES.M.B.H. (B.P. 301,904, 26.11.28. Ger., 9.12.27).—A form of furnace is described in which several annular hearths for the material, heated by fluid in the hollow hearths, are supported with a certain degree of flexibility above a common annular carriage. The furnace is suitable for the continuous heat-treatment of pulverulent material in very thin layers (of the order of 1 mm.), one use being the low-temperature distillation of coal.

B. M. VENABLES.

Boilers and like apparatus for evaporation or heating of liquids. A. LAMB (B.P. 320,482, 6.9.28).—The hot gases from a fire or waste-heat flue pass downwards through a bank of horizontal, superposed tubes through which the liquid rises in zig-zag fashion by thermo-siphon action, aided in the case of evaporation by the vapour bubbles. When an integral combustion chamber is provided, the upper pair of tubes only may extend through the inlet port at the top of the main heating chamber into the upper part of the combustion chamber.

B. M. VENABLES.

Steam accumulators. J. D. TROUP (B.P. 319,108, 31.7.28).—A storage vessel is provided with an external circulation pipe in which is inserted an injector device operated by the incoming steam.

B. M. VENABLES.

Apparatus for effecting contact between gases and divided solids. E. J. BRADY, ASSR. to UNITED GAS IMPROVEMENT CO. (U.S.P. 1,731,223, 8.10.29. Appl., 21.7.28).—The comminuted solid is allowed to fall between two confronting, perforated walls having relative movement, and the gas is caused to pass across the stream several times in a zig-zag manner generally countercurrent to the solid, i.e., upwards.

B. M. VENABLES.

Apparatus for heating by circulation of hot fluids. J. A. REAVELL (B.P. 318,652, 8.5.28).—A heat-transmitting fluid is passed in thin streams over electric heating elements provided with ribs or projections to ensure that the flow of liquid is turbulent.

B. M. VENABLES.

Thermostats. A. B. and T. F. C. POTTERTON, and T. POTTERTON, LTD. (B.P. 320,270, 22.11.28).—The multiplying lever of a known type of temperature-sensitive device operates a bell-crank lever or other device attached to a gas valve by means of a pin and slot, so that the gas is turned suddenly off or on; the thermostat is therefore suitable to control Bunsen burners.

B. M. VENABLES.

Thermometer, pyrometers, and the like [for hot-air furnace gases etc.]. W. GILBERT (B.P.

320,194, 30.8.28 and 12.1.29).—To prevent errors due to radiation to or from the walls of the conduit conveying the gases, the thermometer bulb or hot-junction is enclosed in a number of spaced concentric tubes or plates, which come up to the temperature of the gases.

B. M. VENABLES.

Grinding and crushing. E. BARTHELMLESS (B.P. 291,460, 2.6.28. Ger., 4.6.27).—The material is charged into a helical passage and propelled along it by a gaseous or liquid stream blown in with sufficient velocity to produce disintegration of the material merely by friction and centrifugal impact. Forms of construction are described in which the helical passages are built of a number of superposed similar units. (Cf. B.P. 251,665; B., 1926, 775.)

B. M. VENABLES.

Grinding mills. FULLER LEHIGH CO., ASSEES. OF R. M. HARDGROVE (B.P. 311,688, 7.7.28. U.S., 14.5.28).—The mill is similar in principle to that of a cup-and-cone ball-bearing, the balls not being positively driven. The angle between the faces of the upper ring (cup) and lower table (cone) is between 20° and 70°, and both surfaces slope outwardly downwards. The ring is usually stationary and the table rotated, relative adjustment is provided, and, to avoid grooving, balls of different sizes may be used simultaneously.

B. M. VENABLES.

Construction of grinding mills. A. E. G. MACCALLUM (B.P. 318,873, 11.5.28).—A form of construction of roller and block mill (as used for paints) is described, ensuring accurate alinement of roller and block. The base and bearing supports are in one piece, and the bearing for the rollers can be machined while practically the whole machine is assembled.

B. M. VENABLES.

Separation of mixed materials. T. FRASER (B.P. 299,777, 24.10.28. U.S., 31.10.27).—In a process for the separation of mixed dry materials by floating or sinking in a fluid mass of dry particles, kept fluid by upward currents of air, the fluid mass is subjected also to segregating or transitional movements, which increase the separating effect so that lighter and cheaper material such as sand may be used instead of expensive heavier material such as magnetite for the fluid mass. The segregating movements may be horizontal or vertical and unidirectional or pulsating. Suitable apparatus is described.

B. M. VENABLES.

Apparatus for [air] separation of solids. W. A. DUERR, ASSR. to CABOT CO. (U.S.P. 1,728,046, 10.9.29. Appl., 21.7.27).—To one end of a worm-conveyer a blast of air is supplied, at the other end is a feed hopper for the material, and intermediately (near the hopper) is an air-separating device.

B. M. VENABLES.

Centrifugal separator. ATELIERS T. ROBATEL J. BUFFAUD & CIE. (B.P. 306,124, 15.1.29. Fr., 16.2.28).—A centrifuge with horizontal shaft and adapted either to filtration or decantation is described. The casing is easily renewable independently of the supporting framework, and the discharging scraper can be easily withdrawn and when in operation will cease travelling should the resistance become too great.

B. M. VENABLES.

Centrifugal separators. SOC. GÉN. D'ÉVAPORATION PROC. PRACHE & BOUILLON (B.P. 313,097, 6.6.29. Fr.,

6.6.28).—A centrifuge for the separation of solid matters in suspension in a liquid is described, having continuously acting, gear-driven scrapers for cleaning the outlets for solids.

B. M. VENABLES.

Centrifugal cleaning of liquids. AKTIEBOLAGET SEPARATOR (B.P. 307,933, 15.3.29. Swed., 17.3.28).—To a centrifugal separator having continuous discharge of separated solid matter is added a pulp rich in solid matter with the object of preventing too great an outflow of liquid through the discharge ports for solids, while permitting the ports to be of reasonable size. The additional or circulating solid matter may be of the same nature as the impurities to be separated, the quantity may be regulated less or more according as there is more or less solid in the new feed, and it is preferably supplied to points near the discharge openings.

B. M. VENABLES.

Centrifugal machines, especially for purification or separation of liquids. KOEFOED, HAUBERG, MARSTRAND, & HELWEG A./S. TITAN, and S. HAGERUP (B.P. 320,300, 26.1.29).—A form of construction of a centrifuge having a horizontally divided bowl directly above an electric motor is described.

B. M. VENABLES.

[Pressure] filtering apparatus. A. R. JAHN (B.P. 318,687, 18.6.28).—The filter comprises a fixed head carrying a number of filter units which are enclosed in a detachable pressure-tight cover forming the reservoir for prefilter. The filter walls of each leaf or unit are kept apart by a corrugated or ribbed plate which prevents choking of the passages for filtrate. Brushes and/or compressed air in reverse flow may be used to remove deposited solids.

B. M. VENABLES.

Apparatus for separating impurities from liquid stored in tanks and the like. LIQUID MEASUREMENTS, LTD., and F. HAMMOND (B.P. 320,239, 15.10. and 7.12.28).—Some of the liquid is withdrawn from the point where the impurities have accumulated (usually the bottom of the tank) by means of a pump, and is delivered to a separating vessel, where the impurities are retained by settling and/or filtration; the excess clean liquid runs back into the tank. The whole apparatus may be self-contained and made airtight with the tank (and is then suitable for petrol), and the separating vessel may have a glass wall.

B. M. VENABLES.

Breaking a foam into liquid particles and vapours and gases. J. F. HARKOM (B.P. 320,191, 27.8.28).—A centrifugal rotor is situated in the vessel where the foam is produced, and the gases and vapours are exhausted through the rotor and its hollow shaft against centrifugal force, the liquid being flung off the rotor.

B. M. VENABLES.

Gas scrubber. J. P. FLIPPEN (U.S.P. 1,732,086, 15.10.29. Appl., 6.1.26).—A gas scrubber is divided into two compartments and a different liquid used in each. The scrubbing surfaces are provided by co-operating stationary and rotary grids.

B. M. VENABLES.

Removal of dust from gases. INTERNAT. COMBUSTION, LTD., and P. W. MCGUIRE (B.P. 320,068, 3.4.28).—In the process of removing dust from hot products of combustion by scrubbing with water, there is injected

first into the gases water in such quantities that it entirely evaporates and leaves the gases substantially saturated. The gases are then scrubbed by a counter-current rain of water.

B. M. VENABLES.

Separation and recovery of gases and vapours by solid absorbents. A. GODEL, and SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIFÈRES (B.P. 317,629, 3.8.28. Addn. to B.P. 267,369; B., 1927, 689).—In the apparatus described in the prior patent, the cooling and drying of the absorbent after regeneration is effected by a gas following a closed circuit, cooled at one point and heated at another, thereby preventing loss of adsorbed solvent. (Cf. also B.P. 295,504; B., 1928, 773.)

B. M. VENABLES.

Physical or chemical treatment of gases with solid materials. J. S. MORGAN, and M. NEWBOULD & PARTNERS, LTD. (B.P. 320,450, 9.8.28).—In heterogeneous physical or chemical reactions between a gas and a finely-divided solid, the temperature is controlled by the addition of solid materials of high heat conductivity or capacity which may be inert to the reaction. *E.g.*, in the removal of a constituent of a gas by absorbent carbon, the reaction being exothermic, small metallic balls are mixed with the absorbent, and, after the reaction, are separated from the carbon; the carbon is re-activated and the balls are cooled for re-use.

B. M. VENABLES.

Utilising the heat of gases. C. G. HAWLEY, ASSR. to CENTRIFIX CORP. (U.S.P. 1,726,828, 3.9.29. Appl., 11.3.26).—In an apparatus suitable for the heating of liquids by contact with hot gases, the gas enters a casing in a whirling manner outwardly from a central, rising inlet conduit, and leaves through the top of the casing where are also situated water-sprays. In addition to a sludge outlet at the bottom, there is a circumferential port at the top through which liquid and dust are thrown out by the whirling motion.

B. M. VENABLES.

Viscosimeter. C. D. MILLER (U.S.P. 1,727,836, 10.9.29. Appl., 12.6.22).—The liquid is admitted between two surfaces which are closely adjacent over a large area and have relative motion. The flow of the liquid is checked at some point remote from the inlet and is allowed to pass out at some intermediate point. The pressure of the liquid at the check is a measure of the viscosity.

B. M. VENABLES.

Pulverising mill. E. C. LOESCHE (U.S.P. 1,733,417, 29.10.29. Appl., 10.2.26. Ger., 12.2.25).—See B.P. 247,562; B., 1926, 695.

Heat exchanger. J. P. RATHBUN, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. Co. (Re-issue 17,480, 5.11.29, of U.S.P. 1,708,031, 9.4.29).—See B., 1929, 457.

Extraction of substances from fluids. A. H. PEHRSON (U.S.P. 1,732,819, 22.10.29. Appl., 17.11.27. Swed., 7.10.26).—See B.P. 278,722; B., 1929, 192.

Apparatus for detection and determination of impurities and dissolved matter in water and other liquids. C. E. PERRY, ASSR. to EVERSHED & VIGNOLES, LTD. (U.S.P. 1,734,342, 5.11.29. Appl., 8.4.27. U.K., 18.6.26).—See B.P. 268,597; B., 1927, 400.

Mixing of gases and liquids. E. PLAYER, Assr. to PARC ENGINEERING Co., LTD. (U.S.P. 1,733,101, 22.10.29. Appl., 21.6.27. U.K., 23.12.26).—See B.P. 286,812; B., 1928, 352.

Refrigerating machines. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. L. HEISLER (B.P. 301,833, 5.12.28. U.S., 6.12.27).

Absorption refrigerating apparatus containing an inert gas. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 298,575, 11.10.28. Swed., 11.10.27).

[Device for producing gyratory movement of flue gases in] furnaces. G. HIRSCH (B.P. 320,768, 19.10.28).

Pressure gauge for high vacua. MASCHINENFABR. OERLIKON (B.P. 301,454, 26.10.28. Switz., 30.11.27).

Energy from water etc. (B.P. 319,604).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Transformation of the fatty acids during geological periods. III. G. STADNIKOV and A. WEIZMANN (Brennstoff-Chem., 1929, 10, 401—403; cf. B., 1929, 158).—Further examination of the Suchokujaski boghead coal shows that the residue after extraction with organic solvents and with alcoholic potash consists of the polymerisation products of unsaturated fatty acids. These products possess carboxyl groups and can form salts and esters. A comparison of coorongite with the Siberian boghead coals shows these to have been produced from similar parent substances, viz., the fats of the algæ, but in the coorongite, of which a high proportion is soluble in organic solvents, the polymerisation processes are not so far advanced as in the boghead coals. A. B. MANNING.

Examination of coal in small and large gas-works. W. BERTELSMANN (Gas- u. Wasserfach, 1929, 72, 965—968).—The equipment of a gas-works laboratory is discussed, and a description is given of the laboratory under construction at the Berlin Gas Works. Provision is there made for the proximate analysis of 80—100 samples of coal per day. Any sample showing a low volatile matter content can be further examined in a high-temperature assay apparatus consisting of a wrought-iron retort holding a charge of 700 g. of coal; this can be heated in an electrical furnace, the usual carbonising temperature being 1100°. If desired, a full-scale carbonisation test can be carried out in an experimental retort setting attached to the laboratory. A. B. MANNING.

Determination of the oxidisability of bituminous coals by dilute potassium permanganate solution. D. J. W. KREULEN (Brennstoff-Chem., 1929, 10, 397—400).—Samples weighing 0.0025, 0.005, and 0.0075 g. of the finely-ground coal were heated with 20 c.c. of 0.01N-potassium permanganate at the b.p. for 10 min., 20 c.c. of 0.01N-oxalic acid were then added, and the excess was titrated back with permanganate. The values so obtained for the oxidisability of coal, expressed as c.c. of oxygen taken up per g. of ash-free, dry coal, were extrapolated back to 0.0 g. in order to eliminate variations due to differences in the final concentration of

the permanganate solution. The final values vary from 66 for an anthracite to 886 for a coal of high volatile matter content (38.7%). For a number of coals a rough proportionality exists between the oxygen taken up and the volatile matter content, but there are coals of the same volatile matter content which yet differ widely in oxidisability. The results give a measure of the tendency of the coals to oxidise on storage, although for this determination a method based on oxidation by air is preferable. Durain and vitrain possess approximately the same oxidisability, 990 and 969 respectively; fusain possesses a very much lower value, about 80, which is of the same order as that of wood charcoal. Pre-oxidation of coal in the air at 100—170° has little effect on its oxidisability by potassium permanganate. A. B. MANNING.

Comparison of methods for testing the caking properties of coal. L. SLATER (Dept. Sci. Ind. Res., Fuel Res. Bd. Rept., 1928—9, 110—119).—The caking indices of 20 British coking coals determined by the methods due to Gray and Campredon, Meurice, Barash, and Burdekin, respectively, exhibited no close agreement in the order of the values given by each method, nor was it possible to correlate these results with the shatter indices of the cokes prepared from the same coals in full-scale by-product coke ovens. None of the laboratory methods examined therefore appears capable of assessing the value of a wide range of coals in their application to the production of by-product cokes. Within the limits of the range of carbon content examined, the shatter index increases fairly regularly with increasing carbon content, whilst the caking value rises sharply to a maximum with coals of approx. 86% C and then falls rapidly. A knowledge of the caking index is of value for coals which are to be modified by blending prior to carbonisation. A. B. MANNING.

Separation of the constituents of coke-oven gas by washing under pressure. F. FISCHER, K. PETERS, and W. TER-NEDDEN (Brennstoff-Chem., 1929, 10, 348—349).—Coke-oven gas has been treated with various solvents under a pressure of 20 atm., and the composition of the dissolved and residual gases has been determined. Using petroleum as solvent, the methane content of the gas could be raised from about 23% to 48% (in the dissolved gas), the hydrogen content at the same time falling from about 32% to 17%. Water, a saturated solution of ferrous sulphate, or a soap solution acts as preferential solvent for carbon dioxide. It is sometimes of advantage to use two non-miscible solvents simultaneously; thus, by using water together with petroleum, the gas dissolved in the latter has its methane content raised, and at the same time its carbon dioxide content lowered. A. B. MANNING.

Determination of the sulphur content of gases from boiler furnaces. E. TAYLOR and H. F. JOHNSTONE (Ind. Eng. Chem. [Anal.], 1929, 1, 197—199).—Apparatus and methods are described for sampling the gases after they have passed the boiler tubes and from the furnace. These are designed to reduce the possibility of catalytic action between the points of sampling and analysis. At the lower furnace temperatures an iron pipe with glass lining is used, and at furnace tempera-

tures a copper, glass-lined, water-cooled sampler. A suction pump draws the gases at a constant rate of 0.1 cub. ft. per mm. through the apparatus which absorbs the sulphur gases, after which they pass through a flowmeter. The absorption apparatus for the determination of sulphur dioxide and trioxide consists of one bottle with a fine-grained dry alundum thimble which retains the droplets of sulphuric acid, and a second with a coarser alundum thimble immersed in a sodium hydroxide solution containing hydrogen peroxide, which absorbs the sulphur dioxide. Water is run through the glass tubing into the first thimble and bottle, and the sulphur trioxide is determined by titrating with sodium hydroxide. The second bottle is titrated with standard acid to determine the sulphur dioxide. Results checked against known mixtures show a high degree of accuracy. When only the total sulphur is required, the first bottle is omitted. If the gases contain soot and tar, which would clog the alundum thimble, the impinger principle is used, there being two impinger bottles in series. The composition of the gases is also determined by an Orsat apparatus, and from these data and the coal analysis, the ratio of sulphur in the gases to that in the coal can be computed. H. S. GARLICK.

Deposition of carbon in reaction between carbon dioxide and hydrogen. M. RANDALL and W. H. SHIFFLER (Ind. Eng. Chem., 1929, 21, 941).—A contribution to the discussion of Randall and Gerard (B., 1929, 82) in which the equation $\text{CO}_2 + 2\text{H}_2 = \text{C} + 2\text{H}_2\text{O}$ is offered as a possible explanation of the experimental results obtained by these investigators.

H. S. GARLICK.

Carbon deposits from lubricating oils. Experiments with heavy-duty engines. C. J. LIVINGSTONE and W. A. GRUSE (Ind. Eng. Chem., 1929, 21, 904—908; cf. B., 1926, 571).—Three very heavy lubricating oils of different characteristics have been used in experiments made with 18 motor coaches having sleeve-valve engines. These coaches travelled 180,000 miles during the tests. The same oils were tested in a single-cylinder laboratory engine with poppet valves. It was found in the road tests that whereas on the average the use of a naphthenic oil made cleaning of the ports of the engines necessary once in about 9300 miles, the corresponding mileages for low- and high-carbon paraffinic oils, respectively, were 2400 and 1900. Although the carbon-residue test gives a fairly reliable index of carbon deposition in a poppet-valve engine, some other test seems desirable for predicting the behaviour of a lubricating oil in a sleeve-valve engine. A distillation test which appears to place the oils employed in the order of their suitability for this type of engine is described. H. INGLESON.

Synthetic benzene from water-gas. H. TROPSCH and H. KOCH (Brennstoff-Chem., 1929, 10, 337—346; cf. B., 1928, 324).—The benzene fraction of the synthetic petroleum obtained by the catalytic reduction of carbon monoxide under ordinary pressures consisted of a mixture of olefines (65%) and paraffins, containing only small amounts of aromatic hydrocarbons (benzene 0.1%, toluene 0.4—0.5%), and no naphthenes. The benzene was fractionated and the olefines and paraffins in

suitably-chosen fractions were separated by means of a modified mercuric acetate method. The following hydrocarbons were isolated and identified: Δ^{α} - and Δ^{β} -pentenes, Δ^{α} - and Δ^{β} -hexenes, $\gamma\gamma$ -dimethyl- Δ^{α} -pentene, heptene, (?) Δ^{β} -octene, nonene, *n*-pentane, *n*-hexane, *n*-heptane, *n*-octane, γ -methyloctane, *n*-nonane. A small quantity of a solid crystalline substance (m.p. 106°) of unknown constitution, but possessing some of the properties of a γ -pyrone derivative, was isolated from the fractions of b.p. 100—120°. A. B. MANNING.

Determination of total moisture in carbon blacks.

C. M. CARSON (Ind. Eng. Chem. [Anal.], 1929, 1, 225).—A 5-g. sample of carbon black is placed in a 500-c.c. round-bottom flask with 25—35 c.c. of dry xylene and 200 c.c. of dry mineral oil. A short air-condenser leads to the bottom of a 25-c.c. distilling flask which, in turn, is connected to two or more calcium chloride tubes. The flask containing the sample is heated to 150—170° in an oil-bath, and a stream of dry nitrogen passed through the apparatus. The water and xylene distil into the small distilling flask and thence by warming in a water-bath into the calcium chloride tubes. The amount of water thus determined is considerably higher than the loss by heating in an oven at 105° for 5 hrs.

H. S. GARLICK.

Low-temperature tar from bituminous coal and its utilisation in the artificial resin, the varnish, and disinfectant industries. K. EHLMANN (Brennstoff-Chem., 1929, 10, 405—406).—The commercial utilisation of some of the constituents of the low-temperature tar produced in the K.S.G. plant at Karnap is briefly described. The refined light fractions, preferably mixed with an equal volume of benzol, are used as motor spirit. The tar acids, after removal of the sulphur compounds, are fractionated; the fraction of b.p. 180—230° is used in the artificial resin and the varnish industries; the fraction of b.p. 230—280° possesses marked bactericidal properties and is used, in the form of an emulsion, as a disinfectant; the fraction of b.p. 280—360° finds use as a fungicide. A. B. MANNING.

Effect on concrete of acid water from stored bituminous coal. E. F. WOLF (Ind. Eng. Chem., 1929, 21, 908—910).—The effects of the acid solution formed by the percolation of rain through stored bituminous coal on concrete containing walls are not serious, since the initial rapid reaction which occurs produces an insoluble protective layer which causes further attack to be very slow. H. INGLESON.

Ceramic coatings—an outcome of corrosion difficulties in oil cracking. J. C. MORRELL and W. F. FARAGHER (Chem. Met. Eng., 1929, 36, 596—599).—Those metals and alloys which show the greatest resistance to the destructive corrosion of derived hydrogen sulphide in oil-cracking systems are either inherently unsuitable or too expensive for building the larger elements of a cracking plant. A mixture which has proved suitable for linings consists of furnace cement 60 lb., white silica foundry-sand (90% to pass 50-mesh) 30 lb., short-fibre asbestos 1½ lb., sodium silicate solution (d 1.38—1.42) 1 gal., water 1½ pints. This mixture is sprayed on to the cleaned vessel to a depth of $\frac{1}{8}$ — $\frac{1}{4}$ in., and is then brushed over with a mixture of furnace

cement 6 lb., and sodium silicate solution 1 gal., made to suitable consistency with water. When dried and carefully heated such a lining has proved resistant to corrosion and is of sufficient mechanical strength and tenacity.

C. A. KING.

Compression of refinery and casinghead gases. W. J. MURRAY (Ind. Eng. Chem., 1929, 21, 917—919).—A chart and series of equations are given for calculating the composition and quantity of both the liquid and gaseous phases produced in the compression of refinery and casinghead gases.

H. S. GARLICK.

Stabilising Grozni casinghead gasoline. A. N. SACHANOV and A. I. DOLADUGIN (Nef. Choz., 1928, 15, 464—471).—Suitable apparatus is described.

CHEMICAL ABSTRACTS.

Adsorption of gasoline and of benzene vapour by acidic clay. H. ISOBE, Y. ENDO, and I. KUNISE (Bull. Inst. Phys. Chem. Res. Tokyo, 1929, 8, 805—816).—The adsorption coefficients of acid clays from various Japanese localities have been determined for gasoline and for benzene vapour.

H. F. GILLBE.

Specific heats of petroleum vapours. W. H. BAHLKE and W. B. KAY (Ind. Eng. Chem., 1929, 21, 942—945).—The sp. heats of five petroleum distillates from mid-continent crude having $d_{40} 0.9$ — 0.68 have been determined at substantially atmospheric pressure over temperatures ranging from just above that of complete vaporisation to 350° . The constant-flow method was used in which the superheated vapours of a given distillate are passed at a constant rate through a calorimeter containing a heater to which a known amount of electrical energy was imparted. An equation was found to fit the experimental data with an average deviation of 1.3%.

H. S. GARLICK.

Action of sulphuric acid on mercaptans. S. F. BIRCH and W. S. NORRIS (Ind. Eng. Chem., 1929, 21, 1087—1090).—During the refining of certain light petroleum distillates by sulphuric acid a small amount of a substance soluble in petroleum, insoluble in concentrated sulphuric acid, and corrosive to copper at the b.p. of naphtha is formed. This is shown to occur only when mercaptans are present in the initial petroleum and to be due to the formation of a polysulphide (probably a trisulphide). Diethyl trisulphide, b.p. $78^{\circ}/5$ mm. (cf. A., 1908, i, 308), possesses all the properties of the corrosive substance. The formation of traces of corrosive polysulphides by the action of sulphur on lead mercaptides in naphtha solution is also described. *Di-n-heptyl disulphide*, b.p. $164^{\circ}/6$ mm., $d_{40}^{25} 0.9082$, has no corrosive action on copper. *Di-n-butyl disulphide*, b.p. $88^{\circ}/4$ mm., from *n*-butyl mercaptan could not be converted into the disulphoxide by concentrated sulphuric acid in light petroleum solution. D. W. HILL.

Determination of mercaptans in naphtha. P. BORGSTROM and E. E. REID (Ind. Eng. Chem. [Anal.], 1929, 1, 186—187).—By thoroughly shaking the sample of naphtha with excess of 0.5*N*-silver nitrate, adding excess of 0.03—0.05*N*-thiocyanate, and back-titrating with the silver nitrate solution in the presence of iron alum as indicator, results can be obtained with an average absolute error of 0.002%. Alternatively, the mer-

captans can be removed by shaking with silver nitrate, collecting the precipitated silver mercaptides on a filter, and determining the residual sulphur by the lamp method. Shaking the mercaptan solutions with mercury, as in the method for the removal of elementary sulphur, does not remove any of the mercaptans studied, neither does acidified cadmium chloride, which is recommended for the removal of hydrogen sulphide. Glacial acetic acid and zinc used for the reduction of disulphides cause a partial decomposition of the mercaptans; hence the titration method cannot be used. The percentage of sulphur should be determined by the lamp method, applied before the reduction and after the removal of the resulting mercaptans and other substances formed from the disulphides.

H. S. GARLICK.

Sulphuric acid test of motor benzol. E. KAUDELA (Brennstoff-Chem., 1929, 10, 404—405).—The treatment of motor benzol with concentrated sulphuric acid, which is necessary if the benzol is to satisfy the acid test, destroys some of the valuable constituents. To obtain a satisfactory refined benzol only those fractions which would otherwise form gummy deposits in the cylinder or on the valves etc. of the engine need be acid-washed. It is suggested that the acid test be replaced by an evaporation test, a suitable form of which is being developed.

A. B. MANNING.

Comparison of Russian and American petrolatums. A. S. VELIKOVSKI and S. S. NIFONTOVA (Nef. Choz., 1928, 15, 477—489).—The following values for American, Grozni, and Baku petrolatums, respectively, are recorded: $d_{60}^{60} 0.820$ — 0.875 , 0.880 , 0.869 ; viscosity (E_{60}^{60}) 10.34 — 14.68 , 3.94 , 10.3 ; colour (Duboscq) 18 — 157 mm., 21 mm., 9 mm.; acidity (% SO_3) 0.0008 — 0.006 , 0.020 , 0.051 ; ash 0.0002 — 0.0162 , 0.371 , 0.130 ; asphaltene none, none, 0.34% ; iodine value 9 — 15 , 8.12 , 3.1 ; oxygen value 0.73 — 4.30 , 2.67 , 4.45 ; paraffin wax 32.88 — 61.5 , 19.01 , 19.52% . The paraffin content of Grozni petrolatum can be increased by dissolving in naphtha (initial b.p. 150°), cooling to -10° , decanting the upper layer, and distilling the solvent from the lower layer. CHEMICAL ABSTRACTS.

Cracking mazout containing paraffin wax under high hydrogen pressures. V. IPATIEV, N. ORLOV, and M. BIELOPOLSKI (Brennstoff-Chem., 1929, 10, 346—347).—Heating a Grozni mazout with hydrogen under 55 — 265 atm. and at 440 — 460° for periods of 1.1 — 2.5 hrs. yielded 25 — 34% of benzine (boiling to 150°) and 29 — 35% of kerosene (b.p. range 150 — 300°). The benzine was clear and colourless, and unaffected by fuming sulphuric acid. The kerosene darkened on keeping, but could be refined by treatment with concentrated sulphuric acid or powdered silica gel. The yields were unaffected by the addition of such catalysts as iron or copper oxide. In only one experiment was any coke formation observed.

A. B. MANNING.

Composition of paraffin wax. S. W. FERRIS, H. C. COWLES, JUN., and L. M. HENDERSON (Ind. Eng. Chem., 1929, 21, 1090—1092).—Previous work on paraffin wax indicates the presence of straight-chain hydrocarbons only (cf. B., 1922, 809 A.; 1923, 173 A.; A., 1925, ii, 367; 1926, 43). Evidence is here adduced

for the presence of other types of hydrocarbons, probably branched-chain rather than cyclic or unsaturated. Paraffin wax from American mid-continent petroleum crudes, fractionally distilled at less than 1 mm. and selected fractions crystallised from ethylene dichloride, gave six final fractions, each crystallised 8–14 times. Relative purity of the fractions was indicated by small separation on further crystallisation and by examination of their cooling curves. The m.p. varied from 59.9° to 29.4°, and the highest-melting fractions exhibited the lowest mol. wts. The maximum difference in mol. wt. corresponded to slightly less than two carbon atoms. The last fraction was almost 700 times as soluble in ethylene dichloride at 14° as the first. The close agreement between the observed and calculated molecular refractivities was taken to indicate that the first five and possibly the last fractions are completely saturated.

D. W. HILL.

Acids of montan wax. H. TROPSCH (Brennstoff-Chem., 1929, 10, 403–404; cf. Holde, Bleyberg, and Vohrer, B., 1929, 667).—Holde's criticism of Tropsch's results is briefly discussed. Fractional distillation of the methyl esters *in vacuo* and fractional precipitation of the acids by magnesium acetate leads to a more efficient separation of the acids of montan wax than do the corresponding processes using the ethyl esters and lithium acetate, respectively.

A. B. MANNING.

Sampling of gas over mercury. PEXTON and HUTCHISON. **Gas analysis.** OTT. **Determination of inert gas content of gas mixtures.** LEATHERMAN and BARTLETT.—See I. **Removal of mercaptans.** GREER. **Amyl alcohol from pentanes.** AYRES. **Determination of phenol in presence of salicylates.** HAMILTON and SMITH.—See III. **Utilisation of seaweed.** DILLON and LAVELLE.—See VII. **Carbon black.** CARSON and SEBRELL.—See XIV.

PATENTS.

Plants for washing coal and other minerals. A. FRANCE (B.P. 296,775, 31.5.28. Belg., 9.9.27).—In plants for washing coal etc., in which the material is conveyed in a stream along a suitably inclined launder, the walls of the ports in the bottom of the launder, through which the particles of higher density are discharged, are formed of curved surfaces of adjustable shape and position. The discharge pocket may comprise two superposed chambers separated by a partition to which a flap is pivoted, or by an oblique partition below which a rotary drum is arranged; a shutter rotatable about a horizontal axis serves to adjust the dimensions of the discharge port. The inclination of the shutter may be automatically adjustable according to the height of the liquid stream in the launder.

A. B. MANNING.

Combustion of fuel in furnaces. J. S. CROSSLEY and G. G. TURRI (B.P. 293,461, 3.7.28. Austral., 7.7.27).—The fuel is fed well into the interior of a hollow rotary grate, preferably by means of an invertible trough sliding within a tube lying along the axis of the grate. The fuel may be dried and preheated by being retained for a time within the grate before dropping it. The grate is shaped like the frustum of a stepped cone with its axis horizontal, being made up of circular grate

bars which increase in diameter as they are further from the feed end. Passages between the grate bars serve to admit air and to allow the ash to escape. The inner surfaces of the grate bars are provided with ribs which partition the air passages, and are so designed as to promote longitudinal movement of the fuel.

A. B. MANNING.

Carbonisation and distillation of wood and coal. CARBONISATION SOC. GÉN. D'EXPLOIT. DES CARBONES, Assees. of G. JAKOVA-MERTURI (B.P. 293,321, 2.7.28. Fr., 30.6.27).—The hot combustion gases from a furnace are treated with charcoal to remove any residual oxygen, and are then passed through horizontal cylindrical retorts containing the wood or coal to be carbonised. Steam may be introduced periodically into the retort in order to regulate the progress of the carbonisation.

A. B. MANNING.

Wood distillation. F. S. CLARK (U.S.P. 1,731,242, 15.10.29. Appl., 19.4.27).—The retort containing the wood is heated by the furnace fire, to warm up the walls to prevent condensation of vapours, and also during the final carbonisation stage, but distillation of volatile products, without decomposition, is effected by closed steam-pipes. Steam is also injected into the retort during the distillation and carbonisation stages.

F. G. CLARKE.

[Heat] treatment [and carbonisation] of solid, and especially bituminous, substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,147, 13.7.28).—A retort for the low-temperature carbonisation of bituminous fuels consists of a number of inclined cylindrical pipes connected in series and arranged in zig-zag formation. The pipes are mounted at such an angle that the charge travels automatically down the retort under gravity. Baffles are provided to prevent the charge from completely filling the pipes. The carbonisation gases are drawn off at one or more of the angles of the retort without having to pass through the charge. The retort is heated externally by hot combustion gases which are recirculated through the heating flues and the surplus of which is discharged as required.

A. B. MANNING.

Manufacture of activated charcoal. SOC. ANON. DES CHARBONS ACTIFS E. URBAIN (B.P. 294,214, 19.7.28. Fr., 20.7.27).—Vegetable material, preferably such as yields a dense charcoal, is carbonised at below 600° and then treated with a mixture of phosphorus vapour and hydrochloric acid, carried over the material in a current of superheated steam at 500–700°. The product is washed with water until substantially free from phosphoric acid.

A. B. MANNING.

Gas-purification process and apparatus. L. MELLERSH-JACKSON. From KOPPERS Co. (B.P. 290,660, 27.4.28).—Fuel gases are washed with an ammoniacal liquid to free them from hydrogen sulphide and other acidic impurities. The absorbed impurities are oxidised by means of air in the presence of a suitable catalyst, e.g., a compound of nickel, cobalt, or iron, which is added to the liquid in amount sufficient to promote the oxidation of the sulphides to thiosulphates and to prevent the liberation of hydrogen sulphide, but insufficient to cause the liberation of free sulphur. A part of the

liquid is removed from time to time, and is heated and aerated in order to convert the ammonium thiosulphate into sulphate. The air used for oxidation is subsequently washed for the recovery of the ammonia therein.

A. B. MANNING.

Elimination of sulphur compounds from gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,190, 25.8.28. Addn. to B.P. 310,053; B., 1929, 506).—In a modification of the process of the prior patent, catalysts consisting of highly porous adsorbents of an inorganic nature, *e.g.*, active silica or alumina, are used at temperatures above 250°, and preferably above 400°.

A. B. MANNING.

Distillation of tar. BARRETT CO., ASSEES. OF S. P. MILLER (B.P. 288,270, 26.3.28. U.S., 6.4.27).—Modifications are introduced into the processes described in prior patents, particularly B.P. 284,703 (B., 1929, 803). The tar to be distilled is brought into contact in the common collector main with the hot gases from a battery of coke ovens, and the enriched gases are cleaned by electrical precipitation while still at a high temperature. Means, *e.g.*, a rotary shaft with paddles, may be provided for spraying tar or pitch from the bottom of the main into the gases passing through. The temperatures in the collector main and in the electrical precipitator may be so regulated that different pitch products are obtained therein; these may be collected separately or, if desired, may be combined. The oils in the enriched clean gases are fractionally condensed.

A. B. MANNING.

Separation of the constituents of low-temperature tar and its distillates. G. T. MORGAN and D. D. PRATT (B.P. 318,116, 28.4.28).—The crude phenols obtained from low-temperature tar as described in B.P. 307,382 (B., 1929, 385) are treated with petroleum of low b.p. whereby the crystallisable phenols are extracted. The viscous black residue is separated into two fractions, resinols-C and -D, by treatment with ether, benzene, or low-temperature spirit: the resinols-C are left as an insoluble residue, whilst the resinols-D are precipitated on the addition of petroleum of low b.p. to the solution. These resinols may be used as a basis for lacquers or varnishes, or they may be condensed with formaldehyde to give synthetic resins.

A. B. MANNING.

Distillation of petroleum or the like. P. J. WARD, J. MITCHELL, and STEEL BROS. & CO., LTD. (B.P. 320,135, 14.6.28).—An apparatus is described wherein the latent heat of steam is used for the separation of the more volatile constituents (up to 260°) of the oil, and the latent heat of mercury for that of the less volatile constituents (up to 400°). Distillation is effected in a battery of evaporators connected in series. The incoming oil is preheated in a series arrangement of heat exchangers by condensates or vapours.

W. S. E. CLARKE.

Apparatus for condensing hydrocarbon vapours. J. E. BELL, ASSR. TO SINCLAIR REFINING CO. (U.S.P. 1,730,350, 8.10.29. Appl., 22.1.25).—The vapours pass from a still to a condenser; the condensate and uncondensed gases and vapours are further cooled in separate coolers, the condensates from the latter being passed to a common collector. A liquid-seal device maintains full of liquid the cooler for the condensate from the condenser.

F. G. CLARKE.

Conversion of hydrocarbons of high b.p. range into others of low b.p. range. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,421, 7.7.28. Cf. B.P. 315,991; B., 1929, 843).—Mineral oils, tars, and hydrogenation products of these substances and of coal are distilled at 410–600°, under pressure, in the presence of amides, amines, and/or aldehydes or ketones which contain more than 3 atoms of carbon in the molecule; cracking is facilitated by the admixture of metals and their compounds capable of combining with aldehydes and ketones. In an example, a tar fraction, b.p. 200°, which was cracked at 600° with 2% of formamide, in a quartz column filled with pumice, yielded 31% of products boiling below 200°.

C. B. MARSON.

Manufacture of hydrocarbons, particularly those of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,473, 31.8. and 22.11.28).—Hydrogenation of coal, tar, mineral oils, etc. is carried out by fractional treatment so that constituents not affected in the first stage are removed and treated separately under more intensive conditions, *e.g.*, under higher temperatures and pressures; in the final stage it is preferable to employ the materials in the form of a vapour, the reaction being carried out at a relatively high temperature. *E.g.*, a heavy American oil, b.p. 350°, which was treated with hydrogen at 200 atm. and 450° in the presence of a molybdenum-zinc catalyst, yielded 60% of benzene and middle oil, which was removed continuously; the residue of high b.p., rich in asphalt, which was then treated with hydrogen at 1000 atm. and 460° in the presence of a molybdenum-chromium-manganese catalyst in a second reaction vessel, yielded 90% of hydrocarbons boiling below 350°.

C. B. MARSON.

Production of hydrocarbons of low b.p. from oils or from solid fuels. R. E. GOLDSBROUGH and H. TEVIS (B.P. 320,619, 13.7. and 25.8.28).—Highly-heated oil or pulverised solid fuel together with superheated steam is injected into a chamber lined with a catalyst containing, *e.g.*, silica 14 pts., sodium silicate 9 pts., alumina 4 pts., zinc oxide 2 pts., and traces of magnesite and lime. The mixture is thereby gasified and is subsequently passed through a conduit also lined with the catalyst where the temperature is reduced gradually to below the gasifying point. Vapours are cooled and condensed out of contact with the catalyst.

W. S. E. CLARKE.

Purification of the gaseous cloud formed by heat transformation of heavy oils. C. CHILOWSKY (B.P. 302,292, 13.12.28. Fr., 13.12.27).—Condensing surfaces on which tar-containing vapours are condensed become inoperative owing to the formation of a covering of heavy tar. In the apparatus described the vapours pass upwards over surfaces that are cooled by water at 40–60°. The tendency for tar to adhere to the cooling surface is thereby minimised and the lower portions are also washed by the condensed light liquids from the higher portions of the condenser. A fan may be used to circulate the hot gases in contact with the cooling surface.

T. A. SMITH.

Decolorisation of hydrocarbon oils. F. GARDNER (U.S.P. 1,732,465, 22.10.29. Appl., 10.1.27).—An apparatus is described in which two separate solids are simultaneously fed into a liquid contained in a mixing

tank, the liquid and solids being fed on to a rotary mixer in the upper portion of the tank. The lower portion of the tank is fitted with an agitator. W. S. E. CLARKE.

Removal of amorphous wax from petroleum oils. P. M. TRAVIS, Assr. to TRAVIS PROCESS CORP. (U.S.P. 1,732,143—4, 15.10.29. Appl., 16.7.27).—(A) The oil is chilled in the presence of a finely-divided solid material, and centrifuged. The resulting layer of wax-free oil is continuously discharged towards the axis of rotation from one point of the centrifuge, and the solids are removed from another point in the same direction. (B) The wax and a solution of the wax-free oil are similarly obtained by conducting the process in the presence of a low-boiling diluent. F. G. CLARKE.

Manufacture of purified montan wax. A. RIEBECK'SCHE MONTANWERKE A.-G. (B.P. 297,102, 24.7.28. Ger., 16.9.27).—Solutions of montan wax in oxygenated solvents such as mono- or poly-hydric alcohols, esters, or ketones are decolorised by boiling with activated carbon ("carboraffin") or fuller's earth. The resinous impurities, though not very soluble in the solvents, pass into solution in the presence of the wax. The bulk of the resin is removed by extracting crude wax with the cold solvent. The purified wax is nearly white and has properties comparable with those of carnauba wax.

T. A. SMITH.

Lubricating oil. D. STRYKER (U.S.P. 1,732,780, 22.10.29. Appl., 23.3.25).—A hydrocarbon oil is mixed with an anti-chattering natural wax, e.g., carnauba, bayberry, japan, spermaceti, or bees-wax.

W. S. E. CLARKE.

Manufacture of lubricants. H. VIMMIG, Assr. to TEXAS CO. (U.S.P. 1,729,823, 1.10.29. Appl., 4.10.21).—A mineral lubricating oil is compounded with lead oleate which is completely and permanently soluble in the oil; at low temperatures the product has a viscosity lower than that of the mineral oil.

H. ROYAL-DAWSON.

Gas-purification process and apparatus. F. W. SEBERR, JUN., Assr. to KOPPERS CO. (U.S.P. 1,733,321, 29.10.29. Appl., 18.11.25).—See B.P. 261,755; B., 1928, 357.

Gas purification and regenerating sulphided alkaline solutions. R. A. MORGEN, W. S. YARD, and L. ROSENSTEIN, Assrs. to KOPPERS CO. (U.S.P. 1,732,905, 22.10.29. Appl., 11.10.24).—See B.P. 241,221; B., 1926, 5.

Apparatus for cracking oils. S. SEELIG (U.S.P. 1,732,664, 22.10.29. Appl., 29.12.26. Ger., 25.3.26).—See B.P. 268,323; B., 1927, 805.

Splitting of hydrocarbons. O. SCHMIDT, O. GROSSKINSKY, and G. NIEMANN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,732,381, 22.10.29. Appl., 31.5.27. Ger., 19.6.26).—See B.P. 297,398; B., 1928, 884.

Production of non-combustible chlorinated hydrocarbon. F. S. VIVAS, Assr. to INTERNAT. FIRE-PROOF PRODUCTS CORP. (U.S.P. 1,733,843, 29.10.29. Appl., 11.3.27).—See B.P. 286,726; B., 1929, 349.

[Screening] apparatus for dry-milling of coal and its by-products. H. M. SUTTON, W. L. and E. G. STEELE (B.P. 320,621, 16.7.28).

Automatic regulators for coke-oven plants. W. M. SHALLCROSS (B.P. 321,084, 1.12.28).

Apparatus for quenching coke. SOUTH METROPOLITAN GAS CO., and A. H. ANDREWS (B.P. 320,651, 18.7.28).

Gas burners. L. H. V. REYNOLDS and W. B. ENSIGN (B.P. 320,827, 16.1.29).

Rotary furnaces (B.P. 301,904). **Thermostats** (B.P. 320,270). **Condenser** (U.S.P. 1,727,403). **Purifying liquid stored in tanks** (B.P. 320,239). **Treatment of gases** (B.P. 320,450).—See I. **Hydrogen from methane** (B.P. 319,957).—See VII. **Asphalt-like materials** (B.P. 291,749).—See IX.

III.—ORGANIC INTERMEDIATES.

Preparation of acetic anhydride. E. FRITZMAN (J. Appl. Chem., Russia, 1928, 1, 27—36).—Chlorine (320 g.) is passed during 1.25 hrs. into a cooled, agitated mixture of sodium acetate (985 g.), sulphur (48 g.), and acetic anhydride (1200 g.), and the mixture is then kept for 1 hr. at 90°. The acetic anhydride (90% yield) is distilled off at 100°/50—100 mm. Cast iron or steel apparatus can be used; lead and tin, but not aluminium, are quickly corroded. Alternatively, equimolecular quantities of sodium acetate and toluene-*p*-sulphonyl chloride afford acetic anhydride in 80—90% yield. CHEMICAL ABSTRACTS.

Evaluation of acetic anhydride. E. TERLINCK (Chem.-Ztg., 1929, 53, 814—815, 850—851).—The various methods proposed for the evaluation of acetic anhydride are discussed, and Richmond's statement that the direct acidimetric titration of the sample with 0.5*N*-alkali leads to low results is confirmed. A new procedure is recommended in which a sample (1 g.) is treated with 50 c.c. of 0.5*N*-sodium hydroxide, and, after keeping overnight, the excess alkali is titrated with 0.5*N*-sulphuric acid. A second portion (1 g.) is treated with 15 c.c. of a 20% solution of 2:4-dichloroaniline in chloroform; after keeping overnight, 50 c.c. of 0.5*N*-sodium hydroxide are added, the whole is vigorously shaken, and the excess of alkali titrated as before. The anhydride content of the sample may then be calculated from the decreased acidity in the second case. An improved method is described for the preparation of the dichloroaniline, and for its recovery from the analysed samples. H. F. HARWOOD.

Manufacture of anhydrous ethyl alcohol. D. B. KEYES (Ind. Eng. Chem., 1929, 21, 998—1001).—A résumé of patents to establish American priority in the development of the commercial production of absolute from 95% alcohol by distillation of the latter with a third liquid (benzene, ethyl acetate, carbon tetrachloride, etc.). D. W. HILL.

Detection of *n*-propyl alcohol in commercial allyl alcohol. O. HÜTHIG (Ber. Schimmel, 1929, 163—165).—The presence of *n*-propyl alcohol in commercial allyl alcohol is proved by treatment of the fraction, b.p. 96—97°, with bromine in ether solution and distillation under reduced pressure. The fraction, b.p. 39°/60 mm., gives a *phenylurethane*, m.p. 50—51°, and an

α -naphthylurethane, m.p. 79–80°, identical with those obtained from *n*-propyl alcohol.

J. W. BAKER.

Amyl alcohols from the pentanes. E. E. AYRES (Ind. Eng. Chem., 1929, 21, 899–904).—The method employed in preparing the various amyl alcohols from *n*- and *iso*-pentanes consists in chlorination of the hydrocarbons followed by hydrolysis. The *n*- and *iso*-pentanes employed are derived, by fractionation, from natural-gas gasoline. Chlorination at temperatures below 100° either in the vapour or liquid phase requires the influence of light or of a catalyst, and produces low yields of primary chloropentanes. At temperatures above 200° chlorination of the vapour without the influence of catalysts or of light gives the highest yields of primary chlorides. The presence of anhydrous chlorides of multivalent metals reduces the yield of primary chlorides. The use of iodine, sulphur chloride, or red phosphorus with liquid pentanes causes excessive formation of polychlorides. Insoluble, non-volatile chlorides in contact with the liquids or vapours give high yields of monochlorides, but cause the transposition of the chlorine from primary to secondary or tertiary positions. In the large-scale process a stream of chlorine obtained by the electrolysis of brine is passed into a very rapid stream of the heated vapour of the *n*- or *iso*-pentane, and the hydrogen chloride evolved is absorbed in water; the hydrolysis of the amyl chloride is carried out by hot, aqueous caustic soda produced with the chlorine. The salt solution resulting is returned to the electrolytic cell. The products of hydrolysis together with unchanged pentane are fractionated and the desired portions are recirculated. Equal amounts of primary and secondary monochlorides are produced in the chlorination of *n*-pentane. The secondary fraction consists largely of γ -chloropentane together with some β -chloropentane. *iso*-Pentane on chlorination yields 85% of primary *iso*-amyl chlorides and 15% of *tert.*-amyl chlorides, but no secondary *iso*-amyl chloride. Of the primary chlorides, one third is δ -chloro- and two thirds α -chloro-3-methylbutane. It has been found impossible to hydrolyse or esterify the amyl chlorides completely, but since constant-boiling mixtures are formed with water small amounts present in the alcohols can be removed by fractionation in presence of water. In the process outlined 100,000 gals. of pentanes are chlorinated per day. H. INGLESON.

Removal of mercaptans from solution by adsorption on metallic sulphides. E. J. GREER (Ind. Eng. Chem., 1929, 21, 1033).—Ethyl mercaptan is easily removed from solution in hydrocarbons by shaking with amorphous cupric, lead, stannic, cadmium, and arsenious sulphides. Cupric sulphide also removes *sec.*-amyl mercaptan, which is difficult to remove by other means.

C. W. GIBBY.

Determination of phenol in presence of salicylates. E. H. HAMILTON and C. M. SMITH (Ind. Eng. Chem. [Anal.], 1929, 1, 232).—In mixtures (e.g., coal-tar insecticides) of phenol, oil of birch, *p*-cresol, and kerosene in varying proportions, the phenol can be accurately determined by the following modification of Chapin's method (A., 1920, ii, 645). Kerosene (50 c.c.) is added to 10 c.c. of the aqueous solution of the mixture, and the

phenol is extracted by shaking three times with 100-c.c. portions of water and then determined in the combined extracts. Chapin's method for unsaponified cresols gives very high results due to hydrolysis of the methyl salicylate, and the method for saponified cresols gives only slightly high results.

E. H. SHARPLES.

Viscosity of glycerin solutions. COCKS.—See XII. **Acetic and lactic acids from sawdust.** ALLGEIER and others.—See XVIII.

PATENTS.

Refining raw carbon disulphide. P. SIEDLER and E. SCHULTE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,733,171, 29.10.29. Appl., 11.10.26. Ger., 24.10.25).—See B.P. 260,236; B., 1927, 907.

Manufacture of esters. R. WIETZEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,732,392, 22.10.29. Appl., 28.9.26. Ger., 5.10.25).—See B.P. 259,204; B., 1927, 571.

Isolation of alcohols or phenols from mixtures. F. O. ZEITSCHEL (U.S.P. 1,733,440, 29.10.29. Appl., 13.7.25. Ger., 1.8.24).—See B.P. 252,570; B., 1926, 691.

Condensing organic [perylene] compounds by means of aluminium chloride. A. ZINKE, Assr. to F. BENSA (U.S.P. 1,733,768, 29.10.29. Appl., 7.4.25. Austr., 10.4.24).—See B.P. 232,265; B., 1925, 911.

Production of compounds containing carbocyclic or heterocyclic rings. E. HOFFA and W. LUCE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,733,458, 29.10.29. Appl., 24.4.26. Ger., 5.5.25).—See B.P. 251,997; B., 1927, 550.

Electrochemical processes (B.P. 303,027).—See XI. **Citric acid** (B.P. 302,338).—See XVIII.

IV.—DYESTUFFS.

Influence of chlorine, alone and in conjunction with the sulphonic acid group, on the colours of substituted benzeneazophenols. H. H. HODGSON and W. ROSENBERG (J.S.C.I., 1929, 48, 287–289 T).—The influence of chlorine substituted in all available positions in benzeneazophenol is found to have varying effects on the photo-activation of the system $C_6H_5 \cdot N : N \cdot C_6H_4 \cdot OH \rightleftharpoons C_6H_5 \cdot NH \cdot N : C_6H_4 \cdot O$ the state of strain of which will be largely determined by its degree of ionisation (incipient or otherwise) and assumed in the paper to be shown by the resultant dyeing. The electronics of such a system are tentatively discussed on current theories. In all cases the chlorine-substituted benzeneazophenols are deeper in colour than is the unsubstituted product, showing the influence of chlorine to be bathochromic, i.e. to cause a resultant increase in electronic mobility, and the descending order from light yellow to pronounced orange is: aniline \rightarrow phenol, 4-chloroaniline \rightarrow phenol, 3-chloroaniline \rightarrow phenol, aniline \rightarrow 2-chlorophenol, 3:5-dichloroaniline \rightarrow phenol, sulphanilic acid \rightarrow phenol, 2-chloroaniline \rightarrow phenol, sulphanilic acid \rightarrow 2-chlorophenol, aniline \rightarrow 3-chlorophenol, aniline \rightarrow 2:6-dichlorophenol, 2:5-dichloroaniline \rightarrow phenol, sulphanilic acid \rightarrow 3-chlorophenol, sulphanilic acid \rightarrow 2:6-dichlorophenol, aniline \rightarrow 2:5-dichlorophenol, 2:6-dichloroaniline \rightarrow phenol, sulphanilic acid \rightarrow 2:5-

dichlorophenol, aniline \rightarrow 3:5-dichlorophenol, and sulphanilic acid \rightarrow 3:5-dichlorophenol, the dyestuffs above being designated by their component intermediates. Evidence in favour of the "general effect" of chlorine is given by 3-chloro- being deeper than 4-chloro-benzene-azophenol, and by the 2-chloro-isomeride having a deeper shade than that resulting from the combined general effects of both chlorine atoms in 3:5-dichlorobenzene-azophenol. The somewhat anomalous positions in the order of benzeneazo-2-chloro- and -2:6-dichloro-phenols are probably due to partial co-ordination at the *o*-chloro-phenol end. In all cases examined the presence of the *p*-sulphonic acid group in the benzene residue is accompanied by a deepening of colour. Attempts to prepare unique benzenoid and quinonoid forms of *p*-nitrobenzene-azo-2-chloro- and -2:6-dichloro-phenols and also of *p*-nitrobenzeneazo-2-chloro-1-naphthol were unsuccessful.

PATENTS.

Manufacture of dyestuff preparations. F. FELIX and O. ALLEMANN, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,733,057, 22.10.29. Appl., 28.5.27. Switz., 29.5.26).—See B.P. 271,898; B., 1928, 781.

Manufacture of [vat and acid] dyes. J. BADDILEY, P. DOOTSON, A. SHEPHERDSON, and S. THORNLEY, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,734,789, 5.11.29. Appl., 2.2.28. U.K., 8.2.27).—See B.P. 289,188; B., 1928, 517.

Manufacture of sulphur dyes. A. BERGDOLT, W. NEELMEIER, and T. NOCKEN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,733,443, 29.10.29. Appl., 13.8.27. Ger., 20.8.26).—See B.P. 299,909; B., 1929, 352.

Production of ice colours. W. CHRIST, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,733,447, 29.10.29. Appl., 19.11.25. Ger., 1.12.24).—See G.P. 433,149; B., 1927, 276.

Manufacture of water-soluble arylazodiarylamine dyes. H. EICHWEDE, E. FISCHER, and A. SIEGLITZ, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,734,246, 5.11.29. Appl., 13.8.27. Ger., 21.8.26).—See B.P. 276,372; B., 1929, 239.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of caustic soda on wool. J. B. SPEAKMAN (J.S.C.I., 1929, 48, 321—324 r).—No explanation has previously been given of the increased strength of wool yarns after immersion in 38% caustic soda solution for 5 min. A study of the elastic properties of single fibres shows them to be completely unaffected by similar treatment, and their immunity is referred to the low partial pressure of water vapour in equilibrium with the caustic soda solution and to the formation of the complex hydrate $2\text{NaOH} \cdot 7\text{H}_2\text{O}$. The increased strength of wool yarns, following treatment with concentrated caustic soda solution, is due to surface gelatinisation of the fibres produced by the dilute alkali formed during subsequent neutralisation and washing processes; this binds the fibres firmly together in the dried yarn.

Adsorption of vapours by animal and vegetable fibres. E. V. ALEKSEEVSKI (J. Appl. Chem., Russia, 1928, 1, 184—189).—Experiments with trichloronitro-

methane, arsenic trichloride, methyl sulphate, benzyl chloride, and bromoacetone on various fabric fibres are described.

CHEMICAL ABSTRACTS.

Chemical injury of vegetable fibre products. H. SOMMER (Mitt. Materialprüf. Berlin, 1929, No. 6, 51—55; Chem. Zentr., 1929, i, 3053).—Weakening or rotting is due to the formation of oxy- and hydro-cellulose. Methods for the detection of these substances and of photocellulose resulting from the action of light or weather are considered. A. A. ELDRIDGE.

Wood cooking with magnesium bisulphite solution. VON POSSANNER (Papier-Fabr., 1929, 27, 537—543).—Methods of preparing magnesium bisulphite liquors from dolomite and from the magnesium chloride waste-liquors of the potash industry are described. Dolomite is calcined and treated with water and sulphur dioxide in Mitscherlich towers, thus giving a mixture of calcium and magnesium bisulphite, whilst magnesium salt solutions are treated with lime, and the hydroxide so obtained is converted into bisulphite by means of sulphur dioxide. If magnesium sulphate is used instead of the chloride, insoluble calcium sulphate is formed and interferes with the working of the absorption towers. In cooking, magnesium bisulphite is more resistant to heating under pressure than is the calcium salt, and no separation of monosulphite occurs, whereas with the calcium salt both monosulphite and sulphate are formed and interfere with the penetration of heat through the pulp. Admixture of magnesium bisulphite with calcium bisulphite in the liquor shortens the time of cooking. This time is shortest if 75% of the former and 25% of the latter are present, but the product is more readily bleached and a higher yield is obtained if the magnesium salt alone is used. The yield is higher, and the percentage of ash and lignin and the Sieber numbers of the product are lower, when pure magnesium liquor is used than when either a mixed liquor or calcium bisulphite solution is employed. Although cost is the deciding factor, if large quantities of magnesium chloride liquor are available as raw material, considerable advantages result from the use of magnesium bisulphite.

B. P. RIDGE.

Determination of α -cellulose. G. J. RITTER (Papier-Fabr., 1929, 27, 678—682).—Modifications of the ordinary method of determining α -cellulose have been examined for the purpose of establishing a simple standard procedure which will give accurate and reproducible results. Two samples of cotton linters and three of sulphite-pulp were analysed in several different laboratories by four modifications of the same general method which differed according to the concentration of the sodium hydroxide solution used for mercerisation, time of steeping before maceration, total time of mercerisation, volume of water used to dilute the mercerising alkali, etc. As a result, the fourth modification (known as Method IV of the Cellulose Section of the American Chemical Society) is recommended. In this procedure 3-g. samples of material are treated with 7.5% sodium hydroxide solution, the mixture is kept for 5 min., and is then macerated while further quantities of sodium hydroxide are added. After mercerisation for 45 min., the mixture is diluted with a quantity of water equal to

that of the sodium hydroxide added, and the whole is filtered through a Gooch crucible. The filtrate is poured back through the cellulose mass until all suspended particles are retained, and the residue is washed with water and acetic acid, and dried to constant weight at 105°. All solutions and washing liquids are maintained at 20° throughout the operations. With those materials which presented no difficulties in maceration etc. the same results were obtained in all the laboratories concerned using this procedure, but with one sample of cotton and one of sulphite-cellulose, known to give trouble, the results were less uniform. B. P. RIDGE.

Fractionation of cellulose esters. J. DUGLAUX and R. NODZU (*Rev. gén. Colloid.*, 1929, 7, 241—250).—Fractional precipitation of commercial nitrocellulose from its acetone solution by gradual addition of water or aqueous acetone leads to a separation into several fractions, each giving solutions of very different viscosities; those of high viscosity, in general, precipitate spontaneously, whilst those of low viscosity may be isolated by ultrafiltration or by coagulation. A nitrocellulose may be characterised by its specific viscosity deduced from the viscosity of its 2% solution in methyl ethyl ketone. Fractionation appears to be determined by the different size of the micelles and not by composition, the different fractions having nearly the same nitrogen content. Other methods of fractionation consist in diffusing a nitrocellulose gel in alcohol or in ultrafiltration of a dilute solution, the diffused portion or the ultrafiltrate containing the fine micelles of low viscosity; in this way very pure products may be obtained free from substances in suspension.

F. R. ENNOS.

Effect of thinners on the viscosity of nitrocellulose solutions. B. M. PAM (*J.S.C.I.*, 1929, 48, 223—226 T).—With a mixture of solvents and non-solvents for nitrocellulose which represented a possible schedule for dope manufacture, it was found that the order in which the solvents and non-solvents were added had no influence on the viscosity of the product, provided sufficient time and agitation were allowed. The influence on the viscosity of the addition of excess of liquids already present both individually and in mixture has been examined. The "15% rule" is found to be far from true even with the most efficient thinners, and the reduction in viscosity % on the addition of thinners is proportional not to the percentage of thinner added, but to the logarithm of that addition.

Storage properties of transparent celluloid. O. C. ELLINGTON (*J.S.C.I.*, 1929, 48, 267—276 T).—Fourteen samples of transparent sheet celluloid typical of the manufacturers of six countries have been examined. The samples fell into two distinct classes, viz., class *a*, characterised by inferior stability, by acidity, and by high contents of sulphates and mineral matter, and class *b*, characterised by superior stability and the relative absence of the above impurities. The results were confirmed by a preliminary warm-storage deterioration trial. It was shown that the use of inadequately purified nitrocellulose is the main cause of the liability of transparent celluloid to rapid deterioration and to spontaneous inflammation or even explosion under

adverse conditions of storage. The discrimination of celluloids as to stability in storage should be based on tests which indicate with certainty the purity of the nitrocellulose ingredient used. Such tests are described and limits are suggested. Urea has a pronounced stabilising effect on celluloid made from impure nitrocellulose, but by addition of urea such celluloid cannot be rendered as stable as material made from pure ingredients. The behaviour of celluloid in the later stages of its deterioration is discussed. The occurrence of mineral matter in excessive quantity in incompletely stabilised nitrocellulose is explained and the mechanism of the stabilisation process is discussed.

Effect of temperature on the strength of bag-paper. W. HERZBERG (*Mitt. Materialprüf. Berlin*, 1929, No. 6, 4—6; *Chem. Zentr.*, 1929, i, 3050).—In 24 hrs. at 80° soda-paper gained 22% and sulphite-paper 10% in strength. A. A. ELDRIDGE.

[Test for] paper constituents injurious to metals. W. HERZBERG (*Mitt. Materialprüf. Berlin*, 1929, No. 6, 18; *Chem. Zentr.*, 1929, i, 3050).—The paper is compared with a standard by wrapping around a polished steel plate and preserving in a moist atmosphere, or by clamping the paper between zinc and copper plates for 1 hr. in a moist atmosphere. A. A. ELDRIDGE.

Influence of atmospheric moisture on the properties of fibres and the measurement of atmospheric moisture. G. HERZOG (*Mitt. Materialprüf. Berlin*, 1929, No. 6, 24—30; *Chem. Zentr.*, 1929, i, 3052).

Differentiation of viscose and copper-silk. O. RÜNZI (*Kunstseide*, 1929, 11, 158—160; *Chem. Zentr.*, 1929, i, 3055).

Liquorice root. HOUSEMANN and LACEY.—See XX.

PATENTS.

Washing or cleaning of wool. H. W. FAWCETT (B.P. 319,026, 14.3.28).—Wool-washing liquor is purified by passing through a tank containing a series of inclined plates at a speed below that at which the deposited dirt is remingled with the liquid, and thence to a centrifuge comprising a number of rotating, superimposed, conical plates. F. R. ENNOS.

Treatment of jute to obtain a fibre similar to wool. TEXTILES (NEW PROCESS), LTD., Assees. of J. VIALLET (B.P. 309,021, 15.1.29. Fr., 3.4.28).—Waste jute materials, after treatment in a bath containing 2—3% of an enzyme, e.g., diastase, together with soda in order to remove starch and glue, are submitted to the fermenting action of micro-organisms from virgin jute at 30—35° in the presence of potassium phosphate, and are finally unravelled in a damp state. F. R. ENNOS.

Treatment of cellulose. A. THIRIET (B.P. 318,868, 8.3.28).—Wood cellulose prepared by the soda or bisulphite treatment is purified by subjecting it while flowing down an inclined gauze to jets of water under pressure. After a thorough oxidation it is then washed with water free from alkaline-earth salts, treated with sodium hydroxide solution containing less than 0.001%

of alkaline-earth salts at ordinary or elevated temperature, washed again, and finally bleached.

F. R. ENNOS.

Acetylation of cellulose. RUTH-ALDO CO., INC., Assees. of H. L. BARTHELEMY (B.P. 303,099, 3.8.28. Fr., 28.12.27. Addn. to B.P. 282,791; B., 1929, 429).—The reaction cylinder, which is mounted on hollow trunnions permitting the introduction of the reagents and also of the inlets and outlets of heating or cooling coils, is made to rotate about an axis which is parallel to but off-set from the plane passing at right angles through the centre of its longitudinal axis.

F. R. ENNOS.

Manufacture of chloroacetic cellulose esters. SOC. DES USINES CHIM. RHÔNE-POULENC (SOC. CHIM. DES USINES DU RHÔNE), H. GAULT, and F. BIDAUD (B.P. 318,908, 12.6.28).—Cellulose is heated with monochloroacetic anhydride and a catalyst, *e.g.*, sulphuric acid, the ester being separated by diluting with a non-solvent such as ether either before or after dissolution of the cellulose.

F. R. ENNOS.

Manufacture of cellulose derivatives and solutions of cellulose. L. LILIENFELD (B.P. 318,088, 22.3.28).—Cellulose is pretreated with alkali and carbon disulphide under such conditions that at least a substantial part of the resulting product is insoluble in water. After filtration and washing, if necessary, the cellulose product is converted into viscose, cuprammonium cellulose, or cellulose esters or ethers by the usual methods.

F. R. ENNOS.

Manufacture of cellulose esters. I. G. FARBENIND. A.-G. (B.P. 292,929, 25.6.28. Ger., 25.6.27. Addn. to B.P. 284,298; B., 1929, 593).—Solubility of the esters is assisted by heating them at a high temperature in an anhydrous liquid with an acid halide (inorganic or organic) and, if desired, an acid, an acid anhydride, a salt of a strong acid with a weak base, or a mixture of these.

F. R. ENNOS.

Manufacture of artificial silk yarns and the like. W. P. DREAPER (B.P. 320,100, 6.6.28).—The "dry"-spun artificial silk yarn is removed from the centrifugal box in the form of a cake and wound directly on to a suitable holder; while passing from cake to holder it may be dried, treated with oil solution or sizing material, or given an extra twist.

F. R. ENNOS.

Manufacture of yarns, filaments, ribbons, fabrics, etc. from organic derivatives of cellulose. H. DREYFUS (B.P. 320,363, 5.4.28).—Materials having less tendency to lose their lustre and become opaque and crinkled when treated with hot water are made by spinning cellulose acetate of acetyl value (calc. as acetic acid) 52.5–56% (especially 54.5%) from solution in aqueous acetone of 97.5–100%, of 88–72%, or of lower concentration, under conditions precluding precipitation of the ester.

F. R. ENNOS.

Production of compound materials containing sheets of cellulose derivative. BRIT. CELANESE, LTD., and J. H. ROONEY (B.P. 320,374, 6.7.28).—Sheets of cellulose acetate which have been treated with a mixture of a non-solvent, *e.g.*, methylated spirit, and a plasticiser, *e.g.*, triacetin, are applied while wet to one or more sheets of glass and subjected to heat and pressure; the

edges may then be sealed by means of a solution containing acaroid resin.

F. R. ENNOS.

Desulphurising artificial products made from viscose. O. Y. IMRAY. From HERMINGHAUS & Co. G.M.B.H. (B.P. 319,098, 18.7.28).—The artificial silk is treated with a liquid organic desulphurising agent, *e.g.*, pyridine, or with a solution of one or more desulphurising agents in one or more organic solvents, *e.g.*, phenol or sodium sulphide in ethyl alcohol, which are either free from water or contain insufficient water to have any appreciable swelling action on the product.

F. R. ENNOS.

Surface-treatment of celluloid articles to render the same practically unflammable. A. WEBER, H. HÖPPNER, JUN., and H. WEICH (B.P. 316,276, 25.9.28. Ger., 27.7.28).—The articles are sprayed with a solution of acetate-celluloid in acetone containing amyl acetate, a surface gloss being imparted, if desired, by addition of shellac dissolved in alcohol or varnish lac.

F. R. ENNOS.

Covering articles with non-inflammable celluloid. R. J. CARRUTHERS (B.P. 320,504, 20.9.28).—Non-inflammable celluloid is softened by steeping in a mixture of petrol, acetone, and methylated spirit, wiped dry, and applied to the article which has previously been coated with an intermediate adhesive consisting of cuttings of the celluloid dissolved in methylated spirit and camphor; the covering is then stretched over the article, allowed to dry, and polished.

F. R. ENNOS.

Manufacture of ethers or esters of carbohydrates of the type $(C_6H_{10}O_5)_x$. I. G. FARBENIND. A.-G. (B.P. 293,316, and Addn. B.P. 293,757, [A] 2.7.28, [B] 5.7.28. Ger., [A] 30.6.27, [B] 11.7.27).—(A) After removal of the associated air by passing a current of gas or vapour capable of being absorbed by alkali, *e.g.*, carbon dioxide, acetaldehyde, through the carbohydrate contained in a vessel which is maintained under a vacuum, the alkali compound is formed by treatment with caustic soda and esterification is completed in the usual manner. (B) A current of gas or vapour, which is absorbed or condensed on access of the esterifying liquid, is passed through a vessel containing the carbohydrate under a vacuum until the associated air is eliminated.

F. R. ENNOS.

Treatment of flax. O. D. LUCAS, ASST. to VICKERS, LTD. (U.S.P. 1,733,742, 29.10.29. Appl., 13.1.28. U.K., 5.7.27).—See B.P. 298,178; B., 1928, 889.

Manufacture of textile yarns. ABBEY SYND., LTD., and D. R. NANJI (B.P. 320,860, 20.7.28).

Manufacture of composite yarns and the like. W. P. DREAPER (B.P. 318,531, 5.6.28).

[Pump] apparatus for spinning artificial silk. H. WADE. From SYNTHETA A.-G. (B.P. 320,858, 18.7.28).

Drying of films, bands, etc. of cellulose and the like. WOLFF & Co. KOMMANDIT-GES. AUF AKT., and R. WEINGAND (B.P. 300,496, 27.9.28. Ger., 12.11.27).

Manufacture of tubular bodies from cellulose solutions, particularly viscose. KALLE & Co. A.-G. (B.P. 297,103, 26.7.28. Ger., 15.9.27).

Method and apparatus for coating paper, fabrics, etc. C. MÜNCH (B.P. 291,031, 23.5.28. Ger., 24.5.27).

Purification of [zinc chloride] solutions (B.P. 294,259).—See VII. **Cellulose-resin product** (B.P. 305,671).—See XIII. **Leather substitute** (B.P. 320,444).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of artificial silk. IV. Conditions for uniform dyeing of artificial silks (except acetate silk) with substantive dyes. W. WELTZIEN and K. GÖRTZE (Seide, 1929, 34, 136—143; Chem. Zentr., 1929, i, 3038).—For uniform dyeing the degree of dispersion of the dye is of little importance. The temperature of the bath is related to the amount and uniformity of the dye taken up. Simple rules governing the dependence of uniformity on the operating conditions are elucidated. A. A. ELDRIDGE.

Dyeing wool with direct cotton dyes. A. E. PORAI-KOSHITZ (J. Appl. Chem., Russia, 1928, 1, 11—20).—Experiments with benzopurpurin 4B and diamine-violet N show that, unlike cotton, wool adsorbs only sulphonic acids. CHEMICAL ABSTRACTS.

Action of caustic soda on wool. SPEAKMAN.—See V.

PATENTS.

Bleaching of fur skins. II. STEIN, W. E. AUSTIN, and I. LIEBOWITZ (B.P. 318,471—2, 3.5.28).—The skins, e.g., brown moufflons which have been washed with sodium carbonate solution, rinsed, and hydro-extracted, are (A) treated (preferably brushed) with an aqueous solution containing about 3—7.5% of hydrogen peroxide, 0.5—1.5% of concentrated ammonia, and 1—2% of concentrated potassium soap solution, after which they are dried in the air at about 15—38°, or (B) brushed with a solution containing a bleaching agent, preferably 4—7.5% of hydrogen peroxide, and an organic aromatic reducing agent which may (e.g., *o*- or *p*-aminophenol, *p*-phenylenediamine) or may not (e.g., quinol, phenylhydrazine) impart a desired tint to the skin, with the addition, if desired, of an inorganic reducing and accelerating agent, e.g., ferrous sulphate, after which they are air-dried. L. A. COLES.

Removal of ink spots and the like. C. H. KIDWELL, Assr. to KIDWELL & Co., Inc. (U.S.P. 1,732,606, 22.10.29. Appl., 4.1.24).—Ink spots containing an iron compound are removed from paper, cloth, etc. by moistening them with water, soaking with 5% sodium sulphide solution, washing, acidifying with 5% oxalic acid solution, and again washing. H. ROYAL-DAWSON.

Production of coloured woollen and worsted materials in two or more harmonious shades. F. BAUER (B.P. 319,112, 9.8.28).—Two-tone coloured wool piece-goods are obtained by weaving together unbleached white yarn and yarn mordanted by boiling with chromium fluoride and formic acid (this mordant protects the wool during subsequent dyeing) and afterwards dyeing; the mordanted wool dyes to a darker shade. A. J. HALL.

Printing on wool. I. G. FARBERIND. A.-G. (B.P. 298,648, 12.10.28. Ger., 13.10.27).—Wool may be printed with water-soluble ester salts of the leuco-compounds of vat dyes and afterwards fully oxidised, without staining the white unprinted parts, by using acidified persulphate solution as a developer. E.g., wool is printed with Indigosol Orange HR made into a paste with a dilute solution of British gum, then dried, steamed for $\frac{1}{2}$ hr., developed in a bath at 50—70° containing potassium persulphate acidified with hydrochloric acid (d 1.16) or its equivalent of sulphuric acid, rinsed, soaped, rinsed, and dried. A. J. HALL.

Multi-colour printing process. A. SARK and J. KOROLEW (B.P. 318,844, 8.5.28).—A multi-coloured printing block is prepared by assembling according to a desired pattern a number of shaped units pressed or moulded from coloured plastic masses prepared by suitably mixing kaolin, chalk, white lead, and similar substances with adhesive, water-soluble, viscous, colloidal substances such as syrups, and colouring matters; the joining edges of the shaped units may be blended or effaced by use of a heated tool. Before printing, the fabric, paper, or other material is moistened with a solvent (e.g., dilute alcohol or glycerin) to facilitate transference of colour from the block to the material. A. J. HALL.

Improvement of artificial fibrous materials. L. LILJENFELD (U.S.P. 1,724,670, 13.8.29. Appl., 22.5.26. Austr., 17.6.25).—To increase its strength, artificial silk is treated with a solution of caustic alkali containing not more than 5% by wt. of alkali hydroxide (calc. as NaOH), while stretching the material during at least a part of the treatment. F. R. ENNOS.

Shrinking and felting of animal fibres. J. H. MARTIN (U.S.P. 1,727,374—5, 10.9.29. Appl., 7.2.28. Renewed 31.1.29).—The process is carried out in the presence of a liquid to which is added the condensation product of an aldehyde with (A) a bisulphite or (B) sulphur dioxide. F. R. ENNOS.

Improving [raising the ironing temperature of] textile and other materials. BRIT. CELANESE LTD. (B.P. 304,596, 17.1.29. U.S., 21.1.28).—Material composed wholly or partly of cellulose acetate is treated at 70—90° with a solution containing 0.3—0.7 g./litre of caustic soda, the alkalinity being maintained within these limits by additions of soda from time to time, whereby a regular and partial (not more than 6%) hydrolysis of the cellulose ester takes place. F. R. ENNOS.

Rendering material mothproof. LARVEX CORP., Assecs. of M. G. MINAEFF (B.P. 301,421, 8.11.28. U.S., 29.11.27).—The material is treated with a 2% solution of thiourea or phenylthiourea in aqueous acetone containing a sulphonated oil to facilitate wetting. F. R. ENNOS.

Treatment of vegetable textile fibres to render them non-putrefiable. J. J. LAMBRECHT (B.P. 294,101, 20.6.28. Fr., 16.7.27).—A mixture of oleic acid, soda lye, and a light-coloured mineral oil in water is agitated to cause saponification of the acid, and is then emulsified with boiling water containing sodium carbonate and soda lye. The textile material is heated with this liquor

either in an open vessel or under 3—4 atm., and afterwards bleached and washed in acidulated water.

F. R. ENNOS.

Dyeing of vat colours on fabrics. J. WAKEFIELD (U.S.P. 1,733,439, 29.10.29. Appl., 21.5.28. U.K., 23.6.27).—See B.P. 307,238; B., 1929, 354.

Washing, bleaching, or dyeing artificial silk on cross-wound bobbins. MASCHINENFABR. SCHWEITER A.-G. (B.P. 304,310, 17.12.28. Ger., 19.1.28).

Treatment with liquids of [twisted] artificial threads prepared from cellulose solutions. W. W. GROVES. From HERMINGHAUS & Co. G.M.B.H. (B.P. 297,063, 13.9.28).

Acidification of fabrics. F. R. GROSSER (B.P. 320,996, 27.8.28).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Ammonia oxidation in [relation to sulphuric acid] chamber plants. T. R. OLIVE (Chem. Met. Eng., 1929, 36, 614—617).—The use of nitrogen oxides derived from ammonia is increasing in sulphuric acid manufacture not only on economic grounds, but also owing to the regularity and capability of control of the process. An alloy of platinum and rhodium is more efficient, and has a longer life than platinum as a catalyst and the newer forms of design enable the external heating of the catalyst to be dispensed with. Careful filtering of the gases before they reach the catalyst is necessary, and in the particular converter described the catalyst consists of four layers of 80-mesh gauze made of platinum-rhodium alloy wire (No. 750 alloy; diam. 0.003 in.) with a service life of 6—12 months.

C. A. KING.

Recent developments in the electrochemical production of hydrogen peroxide. L. LOWENSTEIN (Chem.-Ztg., 1929, 53, 821—822).—The electrolysis of sulphuric acid for the manufacture of hydrogen peroxide works with a low current efficiency, but the subsequent distillation of persulphuric acid *in vacuo* is simple and almost automatic. The use of ammonium sulphate solution, with which the efficiency is much better, has hitherto involved difficulties in the distillation, or if it is converted into potassium persulphate the labour cost is much increased. A process has now been worked out in which the anode solution is drawn into tubular distillation vessels in the upper part of which fractionation takes place. This permits direct working from ammonium sulphate. The manufacture of 1 kg. of 30% hydrogen peroxide thus requires only about 4½ kw.-hrs. of direct current and 1 kw.-hr. of alternating current.

C. IRWIN.

Burkheiser ammonium sulphite-bisulphite process. III. **Oxidation of ammonium sulphite and bisulphite in aqueous solution.** E. TERRES and A. HEINSEN (Gas- u. Wasserfach, 1929, 72, 994—998, 1022—1026, 1050—1054. Cf. B., 1928, 156).—The velocity of oxidation is reduced with increasing concentration of sulphite or bisulphite, whence it may be concluded that it is the dissociated part of the salt which participates chiefly or entirely in the reaction.

The rate of oxidation of bisulphite increases with the rise of temperature much more quickly than that of sulphite. Lead peroxide catalyses the former reaction, but no catalyst has been found to accelerate air oxidation of sulphite. The reaction velocity with constant mass of sulphites was determined by agitating a bulb containing the solution together with oxygen and connected to a manometer. The partial pressures of ammonia and sulphur dioxide under reaction conditions are obtained from previous experimental results. Then $K = 1/t \cdot \log [a/(a-x)]$, where a is the barometric pressure less partial pressures, and x the manometer reading in time t . K was found to decrease with decreasing oxygen concentration and (*vide supra*) with increasing sulphite concentration. The rate of oxidation of the solutions used in the Burkheiser process will be quite small, but repeated use of wash-liquors will make the method practical. Concentrated bisulphite solutions are the more readily oxidised. The formation of "Burkheiser salt" is sufficiently explained by these results, and the conditions for its avoidance are established. An acid bisulphite solution should be oxidised to bisulphate and used as wash-liquor. The ammonium sulphate solution remaining after neutralisation should be saturated with sulphur dioxide and used for washing, whereby a sulphite-sulphate solution is obtained which is oxidised by aeration before it returns to process. By the interchange of wash-liquor the deposition of sulphite should be avoided. A temperature of 40—50° is best for oxidation. Experiments with catalysts which led to no very good result are described in detail.

C. IRWIN.

Hyposulphite meter. R. FEIBELMANN and W. MEVES (Chem.-Ztg., 1929, 53, 749).—The meter consists of a glass-stoppered cylinder graduated from 0 to 115%, the zero graduation being some distance from the bottom. The hyposulphite preparation (1 g.) is placed in the cylinder and cold, air-free water is added up to the zero mark. After stirring, a standard solution of ferric sulphate containing thiocyanate is added until, after mixing, the solution remains pink. The percentage of hyposulphite present is then read directly from the height of the liquid above the zero mark.

A. R. POWELL.

Electrolytic production of potassium ferri-cyanide from potassium ferrocyanide. I. G. SHCHERBAKOV (J. Appl. Chem., Russia, 1929, 2, 155—165).—The use of the liquid mercury cathode is described. The average current density should be 1.5—2 amp./in.² at 20—30°. If electrolysis is continued after all the ferrocyanide has been oxidised acid salts are formed.

CHEMICAL ABSTRACTS.

Electrolytic preparation of hydrogen. II. K. TERANO and H. SHIMOYAMA (Bull. Coll. Eng. Kyushu, 1928, 3, 227—241).—Electrode corrosion diminishes in the order: nickel steel, cobalt steel, steel, nickel-plated steel, nickel. Corrosion is marked with alkali carbonate solutions. The cathode overvoltage is lowest with nickel and with rough surfaces. As electrolyte 20% sodium hydroxide solution is preferably employed with an asbestos diaphragm.

CHEMICAL ABSTRACTS.

Determination of inert gas content of gas mixtures. LEATHERMAN and BARTLETT.—See I.

Magnesium bisulphite solution. VON POSSANNER. See V. **Lime putty.** COWPER and WILLIAMS.—See IX. **Arsenious iodide and its solutions.** COCKING.—See XX.

PATENTS.

High concentration of nitric acid. E. I. DU PONT DE NEMOURS & Co. (B.P. 292,951, 15.6.28. U.S., 27.6.27).—Dilute nitric acid is passed as vapour up a tower in which it is brought in contact with a countercurrent of cold, concentrated sulphuric acid; the vapour issuing from the top is conveyed to a reflux condenser which returns to the top of the tower a portion of the condensed acid (1.5–1.75 lb. per 1 lb. removed from the system at 20°); this cools and further dehydrates the emergent vapours. The spent sulphuric acid is denitrated with just sufficient steam for the purpose, and the dilute nitric acid vapour liberated passes into the bottom of the tower. L. A. COLES.

Manufacture of hydrochloric acid. G. P. ADAMSON, Assr. to GEN. CHEM. Co. (U.S.P. 1,729,431, 24.9.29. Appl., 20.12.27).—A mixture of sodium chloride and sulphuric acid is heated at 113–118° to produce pure, gaseous hydrogen chloride. H. ROYAL-DAWSON.

Conducting chemical reactions [manufacture of hydrogen cyanide]. G. D. BAGLEY, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,731,331, 15.10.29. Appl., 26.2.25).—Nitrogen is brought into reactive relationship with a bath of hydrocarbon oil, in the vicinity of which is an electric arc maintained under the surface of the bath. H. ROYAL-DAWSON.

Production of ammonium nitrate. APPAREILS ET EVAPORATEURS KESTNER (B.P. 305,121, 20.11.28. Fr., 31.1.28).—Ammonium nitrate solution in a container situated below a tower and provided with an overflow for draining off excess liquor is pumped through a cooler to the top of the tower; after admixture with about 2% of nitric acid, the liquor is neutralised in the tower with a countercurrent of ammonia and the neutral solution, the temperature of which should not exceed 35°, flows back into the container. L. A. COLES.

Preparation of [anhydrous] calcium sulphate. P. SPENCE & SONS, LTD., and S. F. W. CRUNDALL (B.P. 319,228, 14.6.28).—The product is obtained in a finely-divided, amorphous form by treating the hydrated sulphate, *e.g.*, gypsum, at raised temperatures with dehydrating agents, *e.g.*, concentrated sulphuric acid, sulphur trioxide, phosphoric acid, acid sulphates. L. A. COLES.

Manufacture of calcium cyanide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,733, 15.9.28).—Calcium oxide and/or hydroxide is treated with dry hydrogen cyanide and the water formed during the reaction is withdrawn as vapour or gas by mixing with the hydrogen cyanide dry nitrogen or other inert gas. The process is carried out at 10–40° and at ordinary pressure. (Cf. B.P. 300,348; B., 1929, 53.) W. G. CAREY.

Production of chemically active anhydrous aluminium oxide, and its uses. G. A. BLANC (B.P. 319,850, 6.7.28).—Hydrated aluminium nitrate pro-

duced, *e.g.*, as described in B.P. 181,677 or 246,827 (B., 1923, 402 A; 1927, 75) is heated at 140–180° under atmospheric pressure. This oxide has a special application in the manufacture of aluminium sulphate and the electrolytic preparation of the metal. L. A. COLES.

Production of alkali phosphates and ammonium phosphates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,318, 8.6.28).—A mixture of dicalcium phosphate and an alkali or ammonium bisulphate is heated below sintering point, *e.g.*, at 50–60°, for $\frac{1}{2}$ –1 hr., and the product is leached with water to extract the desired alkali or ammonium phosphate. L. A. COLES.

Production of lasting, storable carbon dioxide developing powder for foods etc., as, *e.g.*, baking powder. L. WEIL (B.P. 303,353, 15.12.28. Ger., 31.12.27).—A neutral, anhydrous alkaline-earth or magnesium pyrophosphate in quantity sufficient to maintain the gas-forming constituents in a dry state is added as filler to baking powder etc. L. A. COLES.

Generating energy from water or aqueous solutions. T. D. KELLY (B.P. 319,604, 23.6.28).—Water containing acids, alkalis, salts, or gases to render it unstable, *e.g.*, 2–10% of nitric acid, calcium nitrate, or nitrous oxide, is injected, with or without subjection to the action of an alternating or direct electric current, into a hot chamber which is preferably lined with silicon carbide and contains air under pressure. The solution is thereby split up into hydrogen, oxygen, etc., and these gases are subsequently fired, the pressure generated being used for the production of power. Air, steam, lubricating oil, etc. may flow through the apparatus to keep it cool. L. A. COLES.

Purification of metallic salt solutions contaminated by organic substances. I. G. FARBENIND. A.-G. (B.P. 294,259, 20.7.28. Ger., 21.7.27).—The solutions, *e.g.*, spent zinc chloride liquor from the manufacture of parchment paper, are treated with oxygen or gases containing it at about 130° under raised pressure. L. A. COLES.

Removal of halogen ions from metal salt solutions containing same as impurities. METALLGES. A.-G. (B.P. 301,512, 30.11.28. Ger., 1.12.27).—Salt solutions for use, *e.g.*, in the electrodeposition of metals flow through a cell containing mercury or a mercury amalgam as anode, under conditions such that the quantity of mercury dissolved is just sufficient to precipitate halogen ions as mercurous halide. L. A. COLES.

Colloidal iodine preparations in gel form. J. COFMAN-NICORESTI (B.P. 320,719, 24.8.28).—Iodine is dissolved in a hydrocarbon oil or oleic acid, the solution is dispersed in colloidal state by an alcohol, *e.g.*, ethyl, methyl, isopropyl alcohols, or mixtures of these, and is stabilised by fatty acids or their salts, *e.g.*, palmitic or stearic acid, or by agar, gelatin, pectin, gums, resins, tallow, etc.; metallic salts or hydroxides may also be added. W. G. CAREY.

Production of hydrogen from methane. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,957, 18.10.28).—Methane is treated with steam in proportions

and under conditions such that the issuing gases contain 2–15% (preferably 4–5%) of unchanged methane, which is subsequently removed, *e.g.*, by combustion with oxygen or, together with carbon dioxide, by extraction with solvents (gasoline) under pressure. Removal of carbon monoxide from the products may be facilitated by converting, prior to the removal of undesired gases, the greater part of it into carbon dioxide and hydrogen by the action of steam and the remainder catalytically into methane.

L. A. COLES.

Apparatus for making hydrogen peroxide by distillation of persulphuric acid and persulphate solutions and for the concentration of hydrogen peroxide solutions. OESTERR. CHEM. WERKE G.M.B.H. (B.P. 293,755, 4.7.28. Austr., 11.7.27).—Heated portions of the apparatus, the inner surfaces of which are covered continuously with a layer of the liquid under treatment, are constructed of a metal, *e.g.*, lead, acid-resisting iron alloys, which, although having a catalytic action on persulphuric acid, persulphates, and hydrogen peroxide, is not attacked by the residual liquor or solvent. The portions in contact with the cold liquid and with hydrogen peroxide are constructed of ceramic ware; *e.g.*, the liquid under treatment is drawn by suction from a ceramic container through a metal tube heated externally by steam, and thence into a separator constructed of ceramic ware.

L. A. COLES.

Production of dense carbon dioxide snow from liquid carbon dioxide. MIDDEN-EUROPEEISCHE OCTROOIMAATSCHAPPIJ (B.P. 314,371, 1.1.29. Switz., 26.6.28).—Liquid carbon dioxide is converted into wet carbon dioxide snow by expansion to a pressure below the triple-point pressure, compression to above the triple-point pressure, and expansion still further, thus freezing the moist snow by sucking the vapour evolved from the bottom of the expansion chamber through a permeable plate upon which the snow rests.

W. G. CAREY.

Filtering and extracting sulphur. A. J. CROWLEY, ASSR. to HUMBOLDT SULPHUR Co. (U.S.P. 1,731,562—3, 15.10.29. Appl., [A, B] 16.7.27).—(A) Molten sulphur is passed through a filtering medium so that a constant volume is maintained on each side of the latter. (B) Steam is used to superheat a mixture of water and the material containing sulphur, whereby the sulphur meets and continuously enters a tubular passage; the coalesced sulphur and residue pass on to a larger part of the tube in which the sulphur solidifies on cooling.

H. ROYAL-DAWSON.

Catalytically manufacturing phosphoric acid and hydrogen. A. MITTASCH and G. WIETZEL, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,732,373, 22.10.29. Appl., 14.11.25. Ger., 22.11.24).—See G.P. 431,504; B., 1926, 916.

Separation of solid salts of ammonium and of the alkalis or alkaline earths. P. OSSWALD and K. SCHAD, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,733,272, 29.10.29. Appl., 10.10.25. Ger., 31.10.24).—See B.P. 242,975; B., 1927, 251.

Production of pure aluminium hydroxide particularly from aluminium-containing raw materials rich in silica. E. L. RINMAN (U.S.P. 1,732,772, 22.10.29.

Appl., 14.11.25. Swed., 22.11.24).—See B.P. 243,356; B., 1927, 74.

Recovery of vanadium [from solutions]. R. H. STEVENS, G. C. NORRIS, and W. N. WATSON, ASSRS. to RHODESIA BROKEN HILL DEVELOPMENT Co., LTD. (U.S.P. 1,733,700, 29.10.29. Appl., 24.9.26. S. Afr., 7.4.26).—See B.P. 269,780; B., 1927, 555.

Preparation of agglomerated solid carbon dioxide. W. HESSLING (B.P. 294,584, 26.7.28. Ger., 27.7.27).

Crystallisation (B.P. 301,496).—See I. Copper oxide-ammonia cellulose solution (U.S.P. 1,728,565).—See V. Sulphur from pyrites (U.S.P. 1,731,516).—See X. Electrochemical processes (B.P. 303,027).—See XI. Fertilisers (B.P. 320,708).—See XVI. Oxygen-evolving preparations (B.P. 319,393).—See XXIII.

VIII.—GLASS; CERAMICS.

Determination of fluorine and of silica in glasses and enamels containing fluorine. J. I. HOFFMAN and G. E. F. LUNDELL (Bur. Stand. J. Res., 1929, 3, 581–595).—The method of Berzelius for the separation of silica and fluorine is tedious and gives low results for both substances, but the following method is rapid and gives accurate results for quantities of fluorine between 0.01 and 0.1 g. in the presence of boric, chromic, sulphuric, or phosphoric acid, although it is not satisfactory for the analysis of phosphate rock or of materials with a high magnesia content. None of the usual constituents of enamels or glasses interferes. The sample (0.5 g.) is fused with 5 g. of sodium potassium carbonate and the product is leached with hot water. The residue is collected and boiled with 50 c.c. of 2% sodium carbonate solution to remove the last of the fluorine. The combined filtrates (300 c.c.) are boiled for 1 min. with a solution of 1 g. of zinc oxide in 20 c.c. of 1 : 9 nitric acid to precipitate the greater part of the silica; the precipitate is collected and washed with hot water, and the filtrate nearly neutralised with nitric acid, evaporated to 200 c.c., neutralised exactly with 1 : 9 nitric acid until methyl-red indicator becomes a very faint pink, and boiled with a solution of 1 g. of zinc oxide in aqueous ammonia and ammonium carbonate until the odour of ammonia disappears. The precipitate is collected, washed with hot water, and, together with those previously obtained, evaporated with 25 c.c. of hydrochloric acid on the steam bath. The dry residue is moistened with 10 c.c. of hydrochloric acid and 150 c.c. of hot water are added. The silica is collected after 15 min., washed, dried, and ignited at 1200°; the filtrate is evaporated with 10 c.c. of sulphuric acid until copious fumes are evolved, water is added, and the remaining silica collected and added to that previously obtained. The usual purity test is applied. The filtrate from the zinc oxide precipitation is treated with 2 drops of a 0.4% solution of bromophenol-blue and 3 c.c. of 10% sodium chloride solution, the volume adjusted to 250 c.c., and dilute nitric acid added until the colour changes to yellow followed by dilute sodium hydroxide solution until the blue is just restored. The solution is treated with 2 c.c. of 1 : 1 hydrochloric acid and 5 g. of solid lead nitrate, heated until the latter

dissolves, and for a further 30 min. after addition of 5 g. of sodium acetate crystals, and set aside overnight. The precipitate of lead chlorofluoride is collected on a close-textured filter paper, washed once with cold water, five times with a cold saturated solution (0.325 g./litre) of lead chlorofluoride, and again once with cold water. Precipitate and paper are then stirred with 100 c.c. of 5% nitric acid on the water-bath, an excess of 0.2*N*-silver nitrate is added to precipitate the chlorine, and the excess is titrated with thiocyanate, using iron alum as indicator (1 c.c. of 0.2*N*-silver nitrate = 0.00380 g. of fluorine). For good results the above details must be followed exactly.

A. R. POWELL.

Tenacity of the system clay-water. O. BARTSCH (Ber. Deut. keram. Ges., 1929, 10, 146—184; Chem. Zentr., 1929, i, 3025).—Measurements of the deformation-resistance were made for various clay-water mixtures, to some of which electrolytes had been added.

A. A. ELDRIDGE.

Clay slip. I. Electrolytes, fluidity, and hydr-o-gen-ion concentration. T. YOSHIOKA and T. IRIE (J. Japan. Ceram. Soc., 1928, 36, 312—323).—Addition of alkali (within certain ranges) increases, and of acids decreases, fluidity; sodium silicate or hydroxide (*e.g.*, 0.16—0.24%) has a greater deflocculating action than the carbonate. The fluidity is slightly increased by ageing.

CHEMICAL ABSTRACTS.

Ceramic coatings. MORRELL and FARAGHER.—See II.

PATENTS.

Manufacture of compound [unsplinterable] glass. RÖHM & HAAS A.-G. (B.P. 297,413, 11.7.28. Ger., 21.9.27).—Sheets of glass are united by polymerisable, unsaturated organic compounds, *e.g.*, methyl cinnamate, ethyl crotonate, which may be polymerised, *e.g.*, by the action of heat or light, before or after application to the glass and may, if desired, be applied in solution, the solvent being subsequently expelled. Colouring and/or strengthening material may also be added.

L. A. COLES.

Colouring glassware [for electric lamp bulbs]. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of M. PIRKIN (B.P. 300,179, 7.11.28. U.S., 7.11.27).—Bulbs frosted on the interior are dusted inside with a dry powder comprising tungstic oxide and other suitable pigments, *e.g.*, zirconium oxide, cadmium sulphide, ultramarine, with the addition, if desired, of red phosphorus.

L. A. COLES.

Apparatus for treating clay. J. C. BLACK, Assr. to CONTACT FILTRATION Co. (U.S.P. 1,731,702, 15.10.29. Appl., 2.8.27).—Clay is heated and agitated with acid, in tanks provided with separate acid and water supplies, and is then transferred to water wash-tanks, in which it is again heated and agitated, the wash water being removed by means of siphons. After passing to a steam-heated receiver, the product is filtered, dried, and pulverised. It has clarifying and decolorising properties.

F. G. CLARKE.

Manufacture of [ceramic] abrasive wheel. H. R. SIMONDS, Assr. to A. A. SIMONDS-DAYTON Co. (U.S.P. 1,732,287, 22.10.29. Appl., 1.7.25).—An abrasive is

bonded with compounds of the alkali and alkaline-earth metals and boric oxide in such quantity that, when fused together into the completed wheel, the expansion of the wheel in use will be negligible.

H. ROYAL-DAWSON.

Manufacture of abrasive wheels. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 319,221 and 319,200, 15.5.28).

Manufacture of bricks and operation of brick kilns. J. S. WHITEHOUSE (B.P. 321,023, 26.9.28).

IX.—BUILDING MATERIALS.

Cellular cement mortars. C. R. PLATZMANN (Cement, 1929, 2, 263—269).—The results of two processes (U.S.P. 1,087,098 and G.P. 327,907; B., 1914, 355; 1921, 180 A) which aim at the production of cellular building material by means of the generation of gas in a cement mixture during the initial setting stage are compared. In the former process, in which 0.1—0.25% of powdered aluminium is incorporated, fineness of grain of the metal and of any filling material, *e.g.*, coke, is necessary to prevent segregation. Increases in volume up to 130% have been obtained without sand, which reduces the expansion to 60—75%, though up to 30% of sand improves the strength. The German method, which employs zinc in a similar manner, showed less satisfactory results with only 30—50% increase in volume, and then only when extraneously heated; better results were obtained when calcium chloride was also introduced. Other gas-forming chemicals, *e.g.*, carbide, were tried, but with negative results; it is considered that the lime in the cement furnishes a valuable third component to the reaction.

C. A. KING.

Influence of aluminium on mortar strength. H. W. LEAVITT, J. W. GOWEN, and L. C. JENNESS (Proc. Nat. Acad. Sci., 1929, 15, 740—742).—A consistent increase (up to 8%) in tensile strength is developed by mortar on the 28-day test as the aluminium content of the sand increases. The breaking strength also increases in the same way. The aluminium content does not affect the compressive strength to such a marked extent.

A. J. MEE.

Joint influence of iron and aluminium in native [Maine] sands on mortar strength. H. W. LEAVITT, J. W. GOWEN, and L. C. JENNESS (Proc. Nat. Acad. Sci., 1929, 15, 742—743; cf. B., 1927, 483).—Iron and aluminium jointly account for about 16% of the variation in the strength developed by mortars made from native sands containing these substances. In general, the chemical condition of the sand used in cement mortars materially affects the strength of the hardened mortars.

A. J. MEE.

Influence of sugar on the setting and hardening of Portland cement. H. W. GONELL (Zement, 1929, 18, 372—375, 437—441, 472—474; Chem. Zentr., 1929, i, 3026).—In presence of sugar, the gel formation in the setting and hardening of Portland cement takes place at the expense of crystal formation. Addition of sodium carbonate favours crystal formation, considerable separation of calcium carbonate taking place.

A. A. ELDRIDGE.

Volume-yield and density of lime putty. A. D. COWPER and J. F. WILLIAMS (J.S.C.I., 1929, 48, 276—279 t; cf. B., 1928, 194).—A rapid method for determining "volume-yield" of lime putty in standard tests of building limes is described, involving only determination of density. The density of dispersed wet calcium hydroxide is found to be constant (≈ 2.12) over a wide range of conditions.

Heat-insulating materials. STILES.—See I. **Effect of stored coal on concrete.** WOLF.—See II. **Liquor-ice root.** HOUSEMAN and LACEY.—See XX.

PATENTS.

Manufacture of quick-setting lime products. C. C. LOOMIS, and A. D. MACDONALD, Assrs. to COLLOIDAL LIME PLASTER CORP. (U.S.P. 1,732,409, 22.10.29. Appl. 5.4.26).—Quicklime, pulverised to pass through a 5-mesh/in. sieve, is partly converted into carbonate by intimate contact with carbon dioxide, such process being continued until the increase in weight is within the range 3—40% of the original weight of the lime.

H. ROYAL-DAWSON.

Manufacture of [coloured] cement. H. C. BANNER (B.P. 320,597, 26.4.28).—Mixtures of finely-ground material comprising a pure base of Portland or aluminous cement, metal oxides, carbonates or salts, or earths or pigments adapted to produce the desired colour, and a flux, are heated to fusion under oxidising conditions.

L. A. COLES.

Manufacture of cellular concrete. K. P. BILLNER, Assee. of N. E. BROANDER (B.P. 306,444, 13.8.28. U.S., 20.2.28).—The use is claimed of finely-ground mixtures of hydraulic cement, puzzuolanic material (blast-furnace slag, lava, trass), and material for producing a cellular structure, e.g., aluminium powder, lime and gypsum being added, if necessary. [Stat. ref.] L. A. COLES.

Production of asphalt-like road building and the like material, from coal tar or pitch. W. SCHROTH (B.P. 291,749, 7.6.28. Ger., 7.6.27).—The phenol content of tar or pitch is adjusted to about 8—10% by suitably blending tars of high and low phenol content, or by adding phenols to pitch, before the material is treated with sulphur and/or oxidising agents, with or without the addition of contact material, for the production of asphalt substitutes. L. A. COLES.

Brick kilns. J. S. WHITEHOUSE (B.P. 320,741, 26.9.28).

Compositions for road making etc. HEWITT (DARLSTON), LTD., and A. HEWITT (B.P. 319,904, 4.9.28).

Making bituminous emulsions and distributing same on road surfaces. H. WADE. From SOC. PROVIA (B.P. 320,919, 19.6.28).

Treatment of crude oils, residues, etc. (B.P. 319,673 and 319,718).—See II. **Conversion of smelts** (B.P. 294,175).—See X.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Hadfield steel. Correlation of physical properties with microstructure and thermal treatment. V. N. SVETCHNIKOV (Rev. Mét., 1929, 26,

401—408).—Measurements of the resistance to shock on a Charpy pendulum machine, and to wear by loss of weight when rubbed on moist carborundum, were carried out on a Hadfield manganese steel containing 1.23% C, 12.67% Mn, 8.35% Si, and 0.1% P. Specimens were used (1) as cast, (2) forged but not thermally treated, (3) forged and quenched in water at 850°, 950°, 1050°, and 1250°, and the results obtained with the treated specimens were plotted against the quenching temperatures. The curves for shock and wear tests both show a sharp change in direction for an optimum quenching temperature of 1150°, whilst the curves for Shore hardness, Brinell hardness, and sp. gr. show a similar but less well-defined change in direction at the same temperature. The cast steel consists of austenite and the eutectic surrounded by a network of carbide. After forging, the metal consists of austenite and a network of a granular constituent. Quenching the forged specimens at increasingly high temperatures results in a progressive diminution in quantity of the granular constituent, which is accompanied by an increase in the viscosity and the resistance to wear. The last traces of the granular constituent disappear at a quenching temperature of 950°, whilst the maximum viscosity is reached only at 1150°. Quenching at 1050° produces a fine-grained structure of pure austenite, slip-bands being very prominent; at 1150° the structure is similar but the grains are larger and more irregular, whilst at 1250° another constituent appears, partly present as a network and partly in the form of rounded grains which treatment with Stead's reagent shows to be rich in phosphorus; the decrease in shock-test value and wearing properties of test-pieces quenched at this temperature is probably due, therefore, to the presence of a network of this phosphoric constituent. The superiority of Hadfield steel over other steels of equal wearing properties is due to its high viscosity, whilst its high resistance to wear is due to the rapidity with which it undergoes cold-hardening. When quenched at 1150° and reheated for 2 hrs. at 570°, it loses much of its viscosity, but its resistance to wear is not affected. The micrographic results can all be attributed to a high phosphorus content.

M. E. NOTTAGE.

Hot aqueous solutions for quenching of steels. H. J. FRENCH and T. E. HAMILL (Bur. Stand. J. Res., 1929, 3, 399—418).—The rates of cooling from 875° of the surface and central portions of steel (0.96% C) cylinders 0.5 in. diam. and 2 in. high in two oils, water, 5% sodium hydroxide, and 5% sodium chloride solutions at 20—100° have been determined and cooling curves plotted and correlated with the microstructures and hardness obtained. The rate of cooling was reduced by increase in temperature of the cooling medium, but was regular in the central zones with aqueous solutions up to 80°, above which it became irregular. The results indicate that warm salt or alkaline solutions are suitable quenching media when rates of cooling intermediate between those obtained in water and oils are required.

A. R. POWELL.

Determination of manganese in steel and iron by the persulphate-arsenite method. A. A. BRIGHT and C. P. LARRABEE (Bur. Stand. J. Res., 1929, 3,

573—579).—The following modified procedure gives good results with up to 20 mg. of manganese. The iron or steel (1 g.) is heated with 30 c.c. of a mixture of 100 c.c. of sulphuric acid (d 1.84), 125 c.c. of 85% phosphoric acid, and 250 c.c. of nitric acid (d 1.4) diluted to 1 litre. When red fumes cease to be evolved, 100 c.c. of cold water, 10 c.c. of 0.8% silver nitrate solution, and 10 c.c. of 25% ammonium persulphate solution are added and the solution is boiled briskly for 30 sec., cooled, diluted with 75 c.c. of water, and titrated with 0.03*N*-sodium arsenite to a clear yellow end-point. The procedure is the same for cast iron except that the graphite is filtered off before oxidation of the manganese. Chromium-tungsten steels (0.5 g.) should be dissolved in 50 c.c. of 1:9 sulphuric acid and 3 c.c. of 85% phosphoric acid; 40 c.c. of water are then added and the solution is boiled with 10 c.c. of 1:1 nitric acid until the tungsten carbide is all dissolved. The analysis is then finished as described above.

A. R. POWELL.

Composition of ϵ -bronze. W. HUME-ROTHERY (Phil. Mag., 1929, [vii], 8, 114—121).—It is shown that under ordinary conditions of examination the composition of ϵ -bronze can vary between the limits 24.5—25.1 at.-% Sn, but there is no conclusive evidence to show whether ϵ -bronze is a solid solution within these limits or whether there exists the compound Cu_3Sn lying in this range, in which there is slight solubility of copper on the one hand and of tin on the other. It is suggested that the ultimate answer to the question will be provided by a detailed X-ray examination, and the precautions to ensure adequate annealing for this purpose are indicated.

A. E. MITCHELL.

Relation between adsorption, wetting power, and flotation. W. LUYKEN and E. BIERBRAUER (Metall u. Erz, 1929, 26, 197—202; Chem. Zentr., 1929, i, 3030—3031).—Experiments with apatite, calcite, wollastonite, magnesite, celestine, and galena in relation to the adsorption of sodium palmitate are described. The adsorption, which is attributed to the exceptional affinity of the sodium palmitate for the alkaline-earth metals, takes place only when the adsorption compound is less soluble than the adsorbent. The sodium palmitate becomes concentrated at the air-water interface, and the individual molecules become oriented. After adsorption the minerals show a diminution of wetting power. The flotation of cassiterite is discussed from the authors' point of view.

A. A. ELDRIDGE.

Extraction of zinc from sulphide ores. V. IL'INSKI and A. SAGAI DATSCHNI (Trans. Sci. Inst. U.S.S.R. Sci. Tech. Dep., Russia, 1928, No. 259, 187—193; Chem. Zentr., 1929, i, 2915).—At 650—700° in an atmosphere of sulphur dioxide conversion into sulphate is complete. Unroasted zinc sulphide ores yield up to 95% of the zinc to sulphuric or hydrochloric acid; under the conditions described only small amounts of sesquioxides pass into solution. Treatment of the roasted ore with alkali dissolves 80% of the zinc, no iron, and only traces of copper.

A. A. ELDRIDGE.

Effect of oxidising conditions on accelerated electrolytic corrosion tests. H. S. RAWSON and W. A. TUCKER (Bur. Stand. J. Res., 1929, 3, 375—390).—The tests were made on sheet copper and nickel, both

anode and cathode being of the same metal, in dilute sodium chloride solution, and the results are expressed in the form of potential-loss in weight curves. The effect of partial immersion as compared with complete immersion was to increase appreciably the anode losses, especially when the overlying atmosphere was oxygen. A nitrogen atmosphere reduced the losses considerably in both cases, showing that the presence of oxygen reduces the potential required to produce a given rate of corrosion. The effect of differential oxidation (aeration), whereby those parts having free access to the air are rendered cathodic and those having less access anodic, originates within the cell and is additional to that resulting from the applied external *E.M.F.*; it therefore tends to increase the corrosion losses, the oxygen acting as a cathode depolariser. In practically all cases the observed corrosion losses were greater than those corresponding with a 100% anode efficiency as calculated in accordance with Faraday's laws. These irregularities show that the accelerated electrolytic corrosion test as at present conducted cannot be considered a satisfactory routine test for the behaviour of a metal under corrosive conditions.

A. R. POWELL.

Electrodeposition of silver from argentocyanide solutions. S. GLASSTONE and E. B. SANIGAR (Trans. Faraday Soc., 1929, 25, 590—598).—The silver deposits formed on prepared sheet cathodes by electrolysis of alkali argentocyanide solutions containing an excess of cyanide and one of the following anions: formate, acetate, hydroxide, phosphate, sulphate, carbonate, chloride, borate, for 1 hr. with a cathode current density of 0.35 amp./dm.² and using pure silver anodes were investigated. Carbonate or an excess of free cyanide decreased the hardness of the deposits, whilst the other anions each increased the hardness, especially borate and chloride. The presence of the anions investigated caused an increase in the polarisation during deposition, but it was very small in the case of solutions containing a large excess of cyanide. Sodium solutions produced slightly more polarisation than the corresponding potassium solutions. Cathodic polarisation and the quality of the silver deposits did not appear to be inter-related, the former appearing to be connected with the alkali metal ions and the latter with the anions. The silver concentration and not the conductivity of the electrolyte determines the limiting current density at which the metal can be deposited with 100% efficiency.

H. T. S. BRITTON.

[Catalyst for] ammonia oxidation. OLIVE.—See VII. **Soils and iron.** HOLLER.—See XVI. **Liquorice root.** HOUSEMAN and LACEY.—See XX.

PATENTS.

Conversion of smelts [molten blast-furnace slag] into foaming highly porous masses. T. GILLER, Assee. of P. OHRT (B.P. 294,175, 13.7.28. Ger., 19.7.27).—The slag is poured successively into a series of small containers arranged on the periphery of a vertical revolving wheel which automatically empties the granulated slag from the containers and refills them with the regulated quantity of water sufficient to convert the slag into a dry porous mass.

A. R. POWELL.

Employment of cerium in the production of steel and iron. I. G. FARBERIND. A.-G. (B.P. 302,574, 21.11.28. Ger., 17.12.27).—The cerium is added as a scavenger in the form of an alloy with iron and another metal, *e.g.*, vanadium, aluminium, silicon, calcium, magnesium, or titanium, the reaction products of which increase the fluidity of the reaction products of the cerium. A. R. POWELL.

Production of metals [iron, by the carbonyl process]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 320,177, 13.8.28).—The gases obtained by the thermal decomposition of iron carbonyl at 280° contain about 2% CO₂, which is removed by means of quicklime, and the residual carbon monoxide is passed over metallic iron produced by the reduction of iron oxide to regenerate iron carbonyl. The yield is increased by 40% as a result of the purification of the gas. A. R. POWELL.

Soldering of iron and steel parts. F. VON WURSTEMBERGER (B.P. 309,032, 2.4.29. Ger., 3.4.28).—The parts are first nickel-plated then soldered with a copper alloy. A. R. POWELL.

Alloy for high-speed steel. M. S. CLAWSON (U.S.P. 1,729,154, 24.9.29. Appl., 26.6.20).—The alloy contains 13–40% W or Mo, 5–20% Cr, 14–75% Fe, 0.5–3% V, 4–20% Co, and 0.85–3.5% C. H. ROYAL-DAWSON.

Hard-metal alloy for [cutting] tools. J. BERTRAM (B.P. 320,991, 23.8.28. Addn. to B.P. 232,591; B., 1925, 962).—In alloys of the type described in the prior patent, the nickel is replaced up to one third of its amount by cobalt. F. G. CROSSE.

Alloy of aluminium, silicon, and iron. B. D. SAKLATWALLA, ASST. to VANADIUM CORP. OF AMERICA (U.S.P. 1,732,915, 22.10.29. Appl., 2.2.28).—A densifying and cleaning alloy for steel contains 5–30% Al, 60–90% Si, and the remainder is chiefly iron; not more than 0.4% C is present. H. ROYAL-DAWSON.

Welding rod. J. R. DAWSON, ASST. to ELECTRO-METALLURGICAL Co. (U.S.P. 1,728,174, 17.9.29. Appl., 8.3.24).—The rod consists of an iron alloy containing about 0.5% V, less than 0.2% C, and about 1.2% Cr. H. ROYAL-DAWSON.

Aluminium-welding rod. H. GILBERT (U.S.P. 1,728,052, 10.9.29. Appl., 30.7.28).—The rod consists of 88–74% Al, 2–42% Cu, 8–06% Cd, and 0.78% Bi. H. ROYAL-DAWSON.

Magnetic alloys. SIEMENS & HALSKE A.-G. (B.P. 290,658, 20.4.28. Ger., 20.5.27).—Alloys having high permeability with low magnetic forces and containing 30–80% Ni, 20–70% Fe, and 5.5–20% Si, the nickel in which may be wholly or partly replaced by cobalt, are claimed. The magnetic properties of such alloys are increased by heating for 1 hr. at 900°, slowly cooling to about 625°, keeping at that temperature for 15–30 min., and finally cooling in air. F. G. CROSSE.

[Non-magnetic] ferrous alloys. S. E. DAWSON (B.P. 320,332, 4.4.28).—A non-magnetic alloy highly resistant to corrosion and having an austenitic structure comprises iron with 0–2% Si, 0.2–0.35% C,

3–14% Mn, 3–12% Ni, and 5–15% Cr. The preferred composition is 0.2% C, 7% Mn, 7% Cr, 7% Ni, and 79% Fe. [Stat. ref.] A. R. POWELL.

Hard metal composition [tungsten-cobalt alloy]. BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of E. G. GILSON (B.P. 289,477, 27.4.28. U.S., 28.4.27).—A mixture of tungsten, carbon, and cobalt, all in powdered form, is pressed into a carbon mould and heated to 1300–1450° (the sintering temperature of the mixture) between electrodes while subjected to a pressure of 1000 lb./in.²; the temperature should be maintained for several minutes. A suitable mixture contains 5–25% of cobalt and an amount of carbon equal to about 6% of the tungsten content. F. G. CROSSE.

Extraction of copper-nickel matte. W. J. HARSHAW and G. L. HOMER, ASSTS. to HARSHAW CHEM. Co. (U.S.P. 1,729,423, 24.9.29. Appl., 28.4.26).—The matte after being ground is leached with acid, lightly roasted, and leached again with acid. H. ROYAL-DAWSON.

Manufacture of shaped pieces consisting of homogeneous alloys of lead with alkali or alkaline-earth metals. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 300,129, 5.11.28).—The alloy of lead with one or more of the metals is extruded at a temperature between 200° and the m.p. of the alloy. Thus, a lead-sodium alloy (20% Na) is extruded at 260–330° under 800–3000 kg./cm.² M. E. NOTTAGE.

Alloy of magnesium. J. A. GANN, ASST. to DOW CHEM. Co. (U.S.P. 1,729,339, 24.9.29. Appl., 27.10.21).—The alloy contains 90–95% Mg and 10–5% Cd. H. ROYAL-DAWSON.

Solder. J. SILBERSTEIN, ASST. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,728,772, 17.9.29. Appl., 9.9.27).—A lead-thallium alloy is claimed containing 99–80% Pb and 1–20% Tl. H. ROYAL-DAWSON.

Treatment of zinc ores. SOC. MINIERE & METALLURG. DE PENARROYA (B.P. 313,430, 29.6.28. Fr., 11.6.28).—The finely-divided zinc ore is mixed with cellulosic organic material such as sawdust, chaff, or paper pulp, the charge being artificially humified if the necessary degree of humidity does not already exist in the cellulosic material. The subsequent calcination of oxide ores or desulphurisation of sulphur ores may be effected, if desired, simultaneously and in the same apparatus. M. E. NOTTAGE.

Recovery of precious metals by amalgamation. D. ENZLIN and J. A. EKLUND (B.P. 320,185, 20.8.28).—The finely-ground ore is mixed with a dilute solution of zinc chloride, mercuric chloride, hydrochloric acid, and chlorine, and the pulp is passed over iron or nickel plates coated with zinc or tin amalgam wherein the precious metals collect. The tailings are dewatered, the solution is electrolysed to regenerate free chlorine, mercuric chloride is added, and the liquor returned to the circuit. A. R. POWELL.

Thermostatic element. W. M. CHACE, ASST. to W. M. CHACE VALVE Co. (U.S.P. 1,729,245, 24.9.29. Appl., 19.12.27).—A bimetallic thermometer consists of two metal parts welded together, the composition of which, respectively, is: 0.35% C, 2% Si, 40% Ni,

57.65% Fe; and 0.25% C, 1.75% Si, 13.25% Cr, 2.5% Ni, 5% Cu, and 77.25% Fe (wrought).

H. ROYAL-DAWSON.

Production of metal plates and sheets for offset printing. I. G. FARBENIND. A.-G. (B.P. 293,834, 9.7.28. Ger., 13.7.27).—The plates or sheets are made of magnesium or a high-grade magnesium alloy, e.g., 5.2% Al, 1% Zn, 93.8% Mg, which is grained before use by immersion in 1–3% nitric acid for 10–100 sec. according to the fineness required. A. R. POWELL.

Electrolytic manufacture of iron. E. KELSEN (B.P. 306,151, 15.2.29. Austr., 16.2.28).—The conductivity of the usual ferrous chloride electrolyte is increased by diluting it slightly and adding a mixture of two other chlorides, e.g., potassium and sodium or potassium and calcium chlorides. A suitable mixture is FeCl_2 122.5 g., CaCl_2 109 g., KCl 110 g., and H_2O 525 g.

A. R. POWELL.

Apparatus for electrolytically depositing chromium. SOC. CHIM. DE LA SEINE, and V. SZIDON (B.P. 320,440, 28.7.28).—The articles to be plated are supported upon a horizontal buss-bar which is mounted so as to be capable of vertical movement in the bath, and which is provided with a brush moving over an electrical resistance so that the current is adjusted automatically to the area immersed. Movable anodes shaped to correspond with the shape of the articles to be treated are mounted in a frame supported inside the bath so that the distance between the anodes and cathodes is kept uniform.

A. R. POWELL.

Electrolytic preparation or purification of metals [beryllium, tungsten, tantalum, or molybdenum]. L. ST. C. BROUGHALL (B.P. 320,818, 31.12.28).—A solution of a salt of the metal in liquid ammonia is electrolysed at a low temperature or under pressure between electrodes of non-rusting steel.

A. R. POWELL.

Heat treatment of steel. S. MAKITA, Assr. to KABUSHIKI KAISHA NIHON SEIKOSHO (U.S.P. 1,733,669, 29.10.29. Appl., 19.4.27. Jap., 22.6.26).—See B.P. 273,248; B., 1928, 451.

Improving the strength qualities of steel wire. W. PÜNGEL, Assr. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,732,615, 22.10.29. Appl., 1.6.27. Ger., 4.6.26).—See B.P. 272,240; B., 1928, 820.

Reduction of ores and the like. W. BUDDÉUS (U.S.P. 1,732,788, 22.10.29. Appl., 14.1.27. Ger., 20.1.26).—See B.P. 264,834; B., 1927, 942.

Treatment of ores for recovery of titanium. A. W. GREGORY (U.S.P. 1,734,034, 5.11.29. Appl., 16.10.26. U.K., 20.10.25).—See B.P. 251,527; B., 1926, 591.

Recovery of zinc oxide. F. RÜSBERG and P. SCHMID, Assrs. to KALI-CHEMIE A.-G. (U.S.P. 1,732,662, 22.10.29. Appl., 24.10.28. Ger., 11.11.27).—See B.P. 300,271; B., 1929, 718.

Aluminium solder. A. J. LINE (U.S.P. 1,732,727, 22.10.29. Appl., 7.6.26. U.K., 8.9.25).—See B.P. 262,192; B., 1927, 80.

Preparation of thorium alloys. H. S. COOPER, Assr. to KEMET LABS. Co., INC. (U.S.P. 1,732,326,

22.10.29. Appl., 4.5.25).—See B.P. 279,274; B., 1928, 20.

Moulds for casting metals. A. E. WHITE. From ALUMINIUM, LTD. (B.P. 320,947, 27.7.28).

Making of metal wool. BRILLO MANUFACTURING Co., INC., Assees. of C. FIELD (B.P. 292,473, 18.6.28. U.S., 18.6.27).

Polishing preparation for metal and other surfaces and its manufacture. A. VAN DER BRUGGEN (B.P. 309,853, 16.6.28. Fr., 16.4.28).

Furnaces (U.S.P. 1,725,879 and 1,727,898). Reverberatory furnaces (B.P. 318,748).—See I. Washing of minerals (B.P. 296,775).—See II. Anhydrous aluminium oxide (B.P. 319,850).—See VII. Separation of minerals (B.P. 302,725).—See XI.

XI.—ELECTROTECHNICS.

Dielectric losses at high frequencies. G. E. OWEN (Physical Rev. 1929, [ii], 34, 1035–1039).—The power loss in very small pieces of dielectrics in high-frequency alternating fields was measured by a calorimetric method in which the heat produced in the dielectric was compared with that produced in a resistance carrying a measured direct current. For vulcanised fibre, celluloid, rubber dam, and glass the loss was found to be proportional to the frequency and to the square of the applied voltage.

N. M. BLIGH.

Chemistry in incandescence lamp manufacture. W. J. BARTLETT (Ind. Eng. Chem., 1929, 21, 970–973).—Chemical processes used in the manufacture of such lamps are briefly reviewed, with special reference to the preparation of tungsten for filaments, gases for filling the lamps, getters, basing cements, inside frosting, and lamp coatings.

C. J. SMITHELLS.

Rugged type of calomel electrode vessel. L. V. SORG (Cereal Chem., 1929, 6, 344–346).—A new type of calomel electrode intended for routine work and giving reasonable accuracy combined with the prevention of diffusion into and out of the cell is described. The liquid junction is easily renewable. The whole cell, constructional details of which are shown by diagram and photograph, can easily be made in the laboratory.

E. B. HUGHES.

Coking of pitch. KOETTNITZ.—See II. Potassium ferricyanide. SCHERBAKOV. Hydrogen. TERANO and SHIMOYAMA. Hydrogen peroxide. LÖWENSTEIN.—See VII. Electrolytic corrosion tests. RAWDON and TUCKER.—See X. Electrodeposition of silver. GLASSTONE and SANIGAR.—See X. Automatic pH recorders. HOLVEN.—See XVII. Sodium glutamate. HAN.—See XIX. Purification of water. BLOCHER. Chlorination of drinking water. BAILEY.—See XXIII.

PATENTS.

Separation of materials of different physical qualities, especially of raw mining products. F. KRUPP GRUSONWERK A.-G. (B.P. 302,725, 13.12.28. Ger., 21.12.27).—In two oscillating electric circuits, tuned to the same frequency and each actuating an amplifying valve, a phase displacement is produced by

entrance of material to be treated into the field of an induction coil and/or condenser in one of the circuits, and is used to generate current or voltage which is used, either directly or after amplification, to control the separating apparatus. J. S. G. THOMAS.

Electrochemical [oxidation and reduction] processes. E. CROVICH (B.P. 303,027, 24.12.28. Ger., 24.12.27).—The processes are conducted with a dilute electrolyte at a temperature between 100° and its critical temperature, and with the use of oxidation or reduction electrodes consisting of solid conductors, *e.g.*, metals, alloys, metal oxides or sulphides, solid fuels, the electrodes being activated by the gases generated during the process. A suitable galvanic battery is built on the filter-press system, and is so constructed that gases may be manufactured under high pressure and that the conveyance of the reacting materials and the regulation of the temperature are effected by the circulation of the electrolyte or the gas. Examples are given of the accumulation of energy by the decomposition of water and subsequent recombination of the high-pressure gases, the generation of pure high-pressure hydrogen, the recovery of pure oxygen and nitrogen from the air, and the generation of working energy from substances of different potential. Other possibilities cited are the production of chlorine, bromine, and iodine, and the reduction of nitrobenzene to aniline by the process.

L. A. COLES.

Apparatus for measuring ultra-violet radiations. A. K. CROAD. From HANOVIA CHEM. & MANUF. Co. (B.P. 320,794, 8.11.28).—Ultra-violet radiation and visible light from a source of controllable intensity, after incidence on a fluorescent material and a non-fluorescent reflector placed in an opaque chamber, are transmitted through a colour filter. J. S. G. THOMAS.

Electrolytic rectifier. E. P. LUNDEEN, Assr. to WILLARD STORAGE BATTERY Co. (U.S.P. 1,729,429, 24.9.29. Appl., 14.4.27).—The breakdown voltage of an electrolytic cell having a film-forming electrode is increased by being heated prior to the formation of the film thereon. Such an electrode having a homogeneous structure of amorphous and crystalline metal thoroughly intermixed and containing less than a normal amount of amorphous metal is claimed.

H. ROYAL-DAWSON.

Electric battery. SCHMID-PATENT CORP., Assecs. of A. SCHMID (B.P. 293,299, 15.6.28. U.S., 2.7.27).—A cathode composed largely of aluminium is immersed in a flowing electrolyte, *e.g.*, a solution of sodium chloride, containing substances such as sodium chlorate and sulphuric acid, and a mixture of sodium chloride and hydrochloric acid, respectively, for depolarising the anode and for preventing polarisation of the cathode. A porous carbon anode, through which the electrolyte is forced, may be used. J. S. G. THOMAS.

Electric discharge tube. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 320,783, 1.11.28).—The wall of an electric discharge tube containing helium and using the positive-column light is composed partly or wholly of a material, *e.g.*, yellow glass, which absorbs violet light. J. S. G. THOMAS.

Getter for vacuum devices. L. McCULLOCH, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,729,888, 1.10.29. Appl., 14.10.24).—For thermionic discharge devices a combination of calcium oxide and the product obtained by baking nickel and magnesium oxalates is used. H. ROYAL-DAWSON.

Manufacture of electron-emitting material. W. B. GERO, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,731,244, 15.10.29. Appl., 29.6.26).—A mixture of pure powdered tungsten, molybdenum, and thorium is subjected to pressure. The intimate mixture of particles is made to coalesce by heating, while maintaining a high vacuum. F. G. CLARKE.

Tungsten electrode for determining [hydrogen] ion concentration. J. R. BAYLIS, Assr. to LEEDS & NORTHRUP Co. (U.S.P. 1,727,094, 3.9.29. Appl., 16.7.24).—The electrode comprises an ordinary tungsten-filament lamp with a hole cut in the top for admission of the solution, the p_H of which is to be determined. The second electrode comprises a calomel half-cell, external to the lamp, having a capillary connexion tube passing just below the surface of the liquid in the lamp, the lead-in wires of which are connected to the usual electrical apparatus and galvanometer. A. R. POWELL.

Manufacture of filament supports [for electric incandescence lamps]. C. J. SMITHELLS, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,733,076, 22.10.29. Appl., 25.4.27. U.K., 14.5.26).—See B.P. 275,735; B., 1927, 786.

Recovery of condensates from coal-distillation gases (B.P. 294,106).—See II. **Hydrogen cyanide** (U.S.P. 1,731,331). **Removal of halogens from salt solutions** (B.P. 301,512).—See VII. **Colouring glass** (B.P. 300,179).—See VIII. **Magnetic alloys** (B.P. 290,658). **Iron** (B.P. 306,151). **Chromium** (B.P. 320,440 and U.S.P. 1,730,349). **Purification of metals** (B.P. 320,818). **Insulating materials** (B.P. 320,370).—See XIV.

XII.—FATS; OILS; WAXES.

Detection and determination of carbon disulphide and sulphur in fluids [*e.g.*, olive oil]. J. A. PIERCE (Ind. Eng. Chem. [Anal.], 1929, 1, 227—228).—Five c.c. of a solution of equal volumes of the oil to be examined and pure chloroform are placed in a test-tube, 2 c.c. of a reagent (freshly prepared by dissolving 1 g. of copper sulphate, nitrate, or chloride in a small amount of water, adding 4 c.c. of ammonia solution followed by 3 g. of hydroxylamine hydrochloride, and making up to 50 c.c. with water) are added, and the tube is stoppered and tilted to and fro for $\frac{1}{2}$ min. The reagent reacts with carbon disulphide instantly with the formation of an opaque, chocolate-coloured aqueous solution which quickly clears, and a heavy slimy precipitate of the same colour floats at the interface of the two liquids. The reaction is sensitive to solutions containing 0.0033% CS₂. With dissolved sulphur no preliminary colouring of the solution occurs, but a dense, deep black metallic-lustrous precipitate of copper sulphide instantly forms. With oils containing both carbon disulphide and sulphur, microscopical examination shows the presence of a brown and a black precipitate both clearly distinguishable. H. S. GARLICK.

Oil content of Malayan estate copra. C. D. V. GEORGI (Malayan Agric. J., 1929, 17, 335—340).—Copra obtained from six representative plantations lost on the average 6.9% of moisture when heated at 100° for 6 hrs. and the moisture-free material contained 65.6% of oil.

E. HOLMES.

Malayan lumbang oil. T. H. BARRY (J.S.C.I., 1929, 48, 289—290 T).—The examination of a recent sample of the Malayan lumbang oil (from *Aleurites moluccana*) indicates that the oil is equal to that obtained from other sources, and of similar chemical and physical properties. Preliminary experiments on its use in paints, however, are not promising owing to the softness of the film and the slowness of drying. It is shown that tung oil can be adulterated with nearly 20% of lumbang oil before the "Brown heat test" exceeds the maximum limit of the present American specifications for tung oil.

Composition of neem oil. So-called margosic acid. A. C. ROY and S. DUTT (J.S.C.I., 1929, 48, 333—335 T).—The fatty acids from neem oil are shown to contain no "margosic acid," which is proved to be a mixture of stearic, palmitic, oleic, linoleic, and small quantities of lower fatty and unsaturated resinous acids, together with a very small quantity of arachidic and lignoceric acids.

Fatty acids and glycerides of kusum oil. D. R. DHINGRA, T. P. HILDITCH, and J. R. VICKERY (J.S.C.I., 1929, 48, 281—286 T).—The properties and constitution of kusum (macassar) fat from the seeds of *Schleichera trijuga*, which grows abundantly in various parts of India, have been studied. The fat is semi-solid and yellow or cream in colour, and is accompanied by unsaponifiable matter and resinous compounds, the properties of which render its suitability for general edible purposes somewhat uncertain. The mixed fatty acids from the fat have setting point 50.5—51°, and are composed approximately as follows: oleic (60%), linoleic (3—4%), arachidic (20—25%), palmitic (5—8%), stearic (2—6%), and myristic acid (1%). These acids are apparently distributed more or less evenly in the form of mixed glycerides; fully-saturated glycerides (triarachidin) occur only in minute amounts, but there is probably some free triolein present. Arachidic acid is present in abnormally large quantities, and the oil is a suitable source of vegetable arachidic acid. The presence of arachidic acid in large proportions renders the soap from kusum fat somewhat hard and granular, although its lathering power is fair; the qualities of the soap are reminiscent of those of a hydrogenated fish oil. Employed in conjunction with other oils consisting largely of combined oleic acid, it should find use as a soap-making material. Of other acids which have been reputed to be present in the oil, lauric acid has been definitely shown to be absent, and no evidence of the presence of butyric acid is forthcoming; small amounts of acetic acid are present, but this acid is probably not in combination as glyceride, and arises from some of the non-fatty compounds which invariably accompany the fat.

Viscosity of glycerin solutions. L. V. COCKS (J.S.C.I., 1929, 48, 279—280 T).—Tables compiled from literature and experimental determinations are given

for the viscosity of glycerin solutions from 10% to 99.2% over a temperature range 1—100°. Attention is directed to similarity between the viscosities of 99.2% glycerin and castor oil. Small quantities of soap, represented by sodium oleate, have the effect of increasing the viscosity of glycerin solutions, the influence of the addition of quantities of the order 0.1% and 0.5% being similar on the average to increasing the glycerin concentration of 80% solution by 3.6% and 7.4% of glycerin, respectively. At higher concentrations of glycerin the effect of small amounts of soap is less marked.

Iodine values. Report of Committee of Society of Leather Trades' Chemists on Oils and Fats. IV. V. KUBELKA, J. WAGNER, and S. ZURAVLEV (J. Soc. Leather Trades' Chem., 1929, 13, 437—442; cf. B., 1929, 924).—The Hanus, Margosches, and Rosenmund-Kuhnenn methods of determining the iodine value have been used on olive, castor, arachis, and dark fish oils, oleic acid, and coconut oil fatty acids. The Hanus method proved the most useful and convenient. Difficulty was experienced with the Margosches method because the mixture of fat with alcohol had to be heated, and this affected the analytical results; also the result was dependent on the weight of fat used. In the other methods the result was unaffected by excess of fat where the iodine value was below 100, but with fats having iodine values above 100, the Rosenmund method proved unreliable with excess of fat. The Margosches method was very sensitive to the effect of increase in the time of reaction.

D. WOODROFFE.

Transformation of fatty acids. STADNIKOV and WEIZMANN.—See II.

PATENTS.

Production of [hard] soap. G. PETROV (U.S.P. 1,732,656, 22.10.29. Appl., 16.1.26. Ger., 21.12.25).—Drying and semi-drying oils are treated with a water-insoluble metal chromate in the presence of a mineral acid, other than chromic acid, capable of forming an insoluble salt with the metal radical of the chromate.

L. A. COLES.

Production of alkali soaps, including ammonium soap, and obtaining fatty acid therefrom. V. GRAUBNER (B.P. 320,331, 4.4.28).—The soap is made by passing a mixture in molecular proportions of fat, alkali, and water continuously in thin streams under pressure through a saponifying apparatus where it is in contact with a heated body, a zig-zag path being formed by the alternate arrangement of projecting baffle-ribs on the heated body and surrounding casing. The saponified mass is forced in a finely-divided state by means of a fine nozzle into a hot saturated salt solution to separate the glycerin from the "soap core"; alternatively, the soap may be distributed into a continuously acidified solution of the corresponding salt in order to separate the fatty acids.

E. LEWKOWITSCH.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Iodine value of turpentine oil. C. KRANZ, V. HRACH, and I. FRANTA (Chem. Obzor, 1928, 3, 365—367).—The maximum value, 370—380, is obtained by Hübl's

method when about 0.1 g. of oil is treated with about 0.4*N*-iodine solution.

CHEMICAL ABSTRACTS.

Determination of the opacity [of pigments] by painting methods. F. SCHMID and K. WEISE (*Farben-Ztg.*, 1929, 35, 229—230).—The limitations of the method of comparing opacity of pigments by conversion into paint and ascertaining the weight required for obliteration of a striped undercoat are indicated. The standard undercoat for white paints recommended by the Reichsausschuss für Lieferbedingungen in this connexion comprises a central black stripe over a white background, the three stripes thus formed having equal width. This is criticised in that on such an undercoat there is a tendency to apply more white paint over the central stripe than over the remainder of the panel, and that the error due to losses at the edge are accentuated. A further source of error is the use of paints of equal consistency but containing different amounts of thinners, leading to inequality in the thickness of the paint coats which are being compared. In an improved undercoat (which should be flat) the edges of the panel are occupied by black stripes, and these preponderate in the striping scheme. Similar considerations apply to undercoats for other colours. With this improved type of undercoat the obliteration method yields results concordant with those attained by exact physical methods.

S. S. WOOLF.

Low-temperature tar and the artificial resin industry. EHRMANN.—See II. **Viscosity of nitro-cellulose solutions.** PAM.—See V. **Malayan lumbang oil.** BARRY.—See XII.

PATENTS.

Manufacture of [fire- and water-proof] paint, adhesive, etc. R. J. CARRUTHERS (B.P. 320,735, 20.9.28).—Celluloid, preferably cuttings or waste (4 pts.), camphor, boric acid, sodium bicarbonate, and zinc chloride (1 pt. of each) are dispersed in 1 pt. of methylacetone, and the whole is thinned if desired with amyl acetate or other suitable thinner. Suitable dyes may be added.

S. S. WOOLF.

Production of palette ["stopping"] materials. G. TRÜMLER (B.P. 294,262, 20.7.28. Ger., 21.7.27).—Quick-drying, non-porous, and non-clogging surfacing or stopping pastes for coating metal, wood, or other surfaces comprise solutions, preferably highly concentrated, of one or more cellulose esters and/or ethers, containing resins if desired, in which solutions one or more finely-divided metals or alloys, and, if desired, filling substances, *e.g.*, kieselguhr, mica, are incorporated.

S. S. WOOLF.

Coating of surfaces. BRIT. THOMSON-HOUSTON Co., LTD., A. T. WARD, and G. R. R. BRAY (B.P. 320,649, 18.7.28).—A coating composition made from a solution of a phenol (preferably di- or poly-hydric, *e.g.*, resorcinol) in furfuraldehyde is applied to a surface and hardened by exposure to the action of a suitable catalyst, *e.g.*, gaseous hydrochloric acid. The addition of plasticisers, *e.g.*, tolyl phosphate and/or toughening agents, *e.g.*, cellulose acetate, improves the characteristics of the film.

S. S. WOOLF.

Manufacture of a white-pigment base. A. E. MARSHALL, Assr. to MARYLAND PIGMENTS CORP. (U.S.P.

1,728,296, 17.9.29. Appl., 1.12.23).—Iron is removed from ilmenite sand containing silica by phosgene, the remaining silica and titania are fused with alkali, and the oxides are precipitated.

H. ROYAL-DAWSON.

Manufacture of synthetic resins. G. T. MORGAN and H. A. HARRISON (B.P. 319,444, 23.7.28).—A condensed hydrocarbon of the type containing one or more unreduced benzene rings and also a five-membered hydrocarbon ring containing one or more methylene groups, *e.g.*, acenaphthene, fluorene, indene, or a monohalogen derivative of such hydrocarbon, is condensed with formaldehyde, or a substance yielding formaldehyde, in the presence of an acid catalyst (sulphuric acid).

S. S. WOOLF.

Cellulose[artificial] resin product. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of H. C. ROHLFS (B.P. 305,671, 8.2.29. U.S., 9.2.28).—Layers of cellulose material are cemented together by means of an "alkyd" (glyptal-type) resin in the intermediate ("B") stage, and the composite material is subjected to pressure, *e.g.*, 1000 lb./in.², at a temperature sufficiently high to soften the resin, *e.g.*, 125—140°. These conditions are maintained for a sufficient time, *e.g.*, 14—16 hrs., to "cure" the resin without affecting the character of the sheet material.

S. S. WOOLF.

Production of paint or waterproofing material. F. W. McRAE (B.P. 320,886, 19.7.28).—See U.S.P. 1,684,593; B., 1928, 903.

Synthetic resins and the like. NOBEL INDUSTRIES, LTD., Assees. of C. E. BURKE and H. H. HOPKINS (B.P. 289,784, 30.4.28. U.S., 28.4.27).—See U.S.P. 1,667,189; B., 1928, 457.

Grinding mill (B.P. 318,873).—See I. **Low-temperature tar and distillates** (B.P. 318,116).—See II. **Colour-binding agents** (B.P. 289,053).—See XVII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Preparation and properties of aqueous rubber dispersions. H. L. TRUMBULL (*Coll. Symp. Mon.*, 1928, 6, 215—224).—Aqueous dispersions of rubber, obtained by mastication with protective colloids, are free from pear-shaped particles and give a dried film of controllable properties.

CHEMICAL ABSTRACTS.

Vulcanisation without sulphur. **Vulcanisation by heat or ultra-violet light with the aid of trinitrobenzene and picric acid.** F. KIRCHHOF (*Gummi-Ztg.*, 1929, 44, 252—253).—Contrary to its behaviour in vulcanisation by sulphur, litharge does not assist hot vulcanisation by trinitrobenzene or picric acid; lamp-black aids such vulcanisation, but the products are of poor mechanical quality and resemble regenerated rubber in physical character. Vulcanisation of rubber in ultra-violet light by such compounds, especially trinitrobenzene, yields better results comparable with those of cold vulcanisation; these products, like ordinary vulcanisates, also show strong absorption of ultra-violet radiation suggestive of reduced unsaturation.

D. F. TWISS.

Ageing of rubber. B. V. BYZOV (*J. Appl. Chem.*, 1928, 1, 6—11).—Properly vulcanised rubber undergoes smaller changes on ageing than does uncured rubber.

The changes are probably due to a continuation of the vulcanising process at the temperature (*e.g.*, 70°) used in artificial ageing tests, and differ from those which occur in natural ageing.

CHEMICAL ABSTRACTS.

Carbon black [in rubber]. C. M. CARSON and L. B. SEBRELL (Ind. Eng. Chem., 1929, 21, 911—914).—The absorptive power of a carbon black for iodine is a measure of the rate of cure of a rubber mix, low absorptive power corresponding with rapid curing rate. The effect of heating blacks over the temperature range 500—1200° is to increase their absorptive power and, at the same time, to increase both the rate of cure of a rubber mix made from them and also the modulus of the product. The stiffening action of a carbon black may be estimated by measuring the amount of the substance which is retained in a thin rubber cement after centrifuging for a given time. Carbon blacks when boiled with sulphur and zinc oxide in xylene liberate a substance which serves to accelerate the rate of cure of a pure gum mix.

H. INGLESON.

PATENTS.

Production of articles substantially made of organic materials from aqueous dispersions containing such materials. DUNLOP RUBBER CO., LTD., G. W. TROBRIDGE, and E. A. MURPHY (B.P. 320,387, 12.4.28).—Substantially adhering fluid layers of an aqueous dispersion of organic materials, such as rubber, are applied to non-porous, inert surfaces by repeated dipping, spreading, and/or pouring; the setting and drying of each layer are effected by heat applied to the side of the layer adjacent to the deposition surface prior to the deposition of the next fluid layer.

D. F. TWISS.

Coating or plating apparatus. ANODE RUBBER CO., LTD., Assees. of G. F. WILSON (B.P. 298,650, 13.10.28. U.S., 13.10.27).—In order to prevent flocculation or agglomeration, *e.g.*, by frictionally sliding surfaces, in colloidal dispersions such as of rubber during the deposition of material such as rubber from colloidal dispersion on to a rotating article, the latter is so supported that the liquid is excluded from the rotating or moving parts.

D. F. TWISS.

Manufacture of artificial compositions, especially those resembling rubber. I. G. FARBENIND. A.-G. (B.P. 294,474, 6.7.28. Ger., 23.7.27).—Artificial compositions, especially such as resemble rubber, are obtained from organic esters or ethers of vinyl alcohol by polymerisation and incorporating therewith, before, during, or after polymerisation, such nitrogenous organic compounds and/or aromatic hydrocarbons as are solid at ordinary temperature. Polymerisation may be effected by known methods, *e.g.*, heating or irradiation, or a combination of such methods, in the presence or absence of accelerators, the resulting products being finally compounded and vulcanised as required.

D. F. TWISS.

Electrical insulating materials. W. S. SMITH, H. J. GARNETT, J. N. DEAN, B. J. HABGOOD, and H. C. CHANNON (B.P. 320,370, 4.7.28 and 28.3.29).—By hydrolytic treatment, *e.g.*, by the action of water at an elevated temperature, in the presence of glycerin or other hydroxy-compounds, the ordinarily water-insoluble impurities of raw rubber are rendered remov-

able by extraction with water. Heated glycerin effects a swelling of the rubber protein, and thereby aids the hydrolytic influence of the water. Alternatively, the raw rubber may be caused to absorb a solvent, *e.g.*, about 10% of benzene, and then be subjected to hydrolytic treatment with superheated water and steam.

D. F. TWISS.

Manufacture of synthetic rubber. R. P. DINSMORE, ASST. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,732,795, 22.10.29. Appl., 13.9.27).—See B.P. 297,050; B., 1929, 829.

XV.—LEATHER; GLUE.

Pine bark and the manufacture of rich pine bark extracts. P. JAKIMOV (Collegium, 1929, 334—356).—The tannin content of pine bark varies from 6.5% to 11.5%, due to climatic conditions, method of harvesting, and mould attacks. Only the extract can be obtained of standard composition. Cold leaching of the bark extracts all the non-tans, but only 60—65% of tannin. The best extract was obtained by extracting pieces of bark, 2—3.5 mm. thick, at 95—96° in a battery of 8 leaches, allowing 2 hrs. in each. A crude, evaporated extract (*d* 1.180) contained tans 35.5%, non-tans 49.3%, sugars 11.3%, insoluble matter 15.2%, and tans: soluble matter 41.3%. This ratio was low, and was improved by subjecting the unground bark to a slow current of water for 24 hrs., pressing it to remove excess water, cutting it into small pieces, and leaching in the usual manner. The maximum amount of insoluble matter was rendered soluble by treatment of the improved extract for 20 hrs. at 96° with 35% (on the weight of insoluble matter) of a mixture of sodium sulphite and bisulphite. The solubilised extract contained tans 55.7%, non-tans 41.3%, and insoluble matter 2.98%. The greater content of non-tans in the untreated extract renders the viscosity higher, yields a greater amount of acid on fermentation, hinders the salting out of the tannin by sodium chloride, and adversely affects the speed of tannage with this material.

D. WOODROFFE.

Tannin analysis: Report of Committee of Society of Leather Trades' Chemists. J. G. PARKER (J. Soc. Leather Trades' Chem., 1929, 13, 412—421).—The Koch extractor is too small and frequently chokes; materials occasionally separate into two layers. The degree of concordance is bad and reinstatement of the Procter extractor is advocated. For the extraction 4 hrs. is ample time. Different results were obtained in the determination of the moisture by vacuum oven, electric oven, water oven, and combined evaporator and drier, respectively. The moisture should be determined by drying a small quantity of the powdered extract in a small weighing bottle in an oven at not above 100°. Lower percentages of total soluble matter were obtained by the filter-paper method than by use of the Berkefeld filter-candle, and the former method possesses no advantage in manipulative detail or accuracy over the latter. The filter-candles do not vary in degree of porosity beyond the limits of ordinary experimental error. The chroming of the hide powder with chrome alum was found to be highly satisfactory and better than the old method.

D. WOODROFFE.

Action of neutral salts on the enzyme activity of tryptic bates. I. V. KUBELKA and J. WAGNER (Collegium, 1929, 328—334).—The activity of tryptic enzymes on casein was increased by the addition of ammonium salts, and at low salt concentrations the increase was almost proportional to the salt concentration. Maximum activity was attained with an amount of ammonium salt 2—4 times that of standard bating material present. This corresponds to 33% of ammonium salt in a commercial bating material, and since this amount is usually exceeded, the enzyme activity of the bating material is always at its maximum, hence the author's method of analysis (B., 1929, 140, 755) is free from objection on this ground. With enzyme preparations which contain less than 33% of ammonium salt it will be necessary to add a further supply of the salt sufficient to bring the proportion present above 33% in testing them by the casein substrate method.

D. WOODROFFE.

Vegetable-tanned sole leathers. A. COLIN-RUSS (J. Soc. Leather Trades' Chem., 1929, 13, 443—462).—As a result of the analysis of a considerable number of such leathers it is suggested that they fall into three well-defined groups of chemical composition and quality with hide substance, water-soluble matter—hide substance ratio, leather substance, and ashed water-soluble matter, respectively, as follows: grade I $> 42\%$, < 0.3 , $> 72\%$, 0.9% ; grade II 42 — 32% , 0.3 — 0.9% , 72 — 56% , 1.65% ; grade III $< 32\%$, > 0.9 , $< 56\%$, 3.1% . All leathers should pass the thymol-blue test for free acid. Leathers containing less than 36% of hide substance are liable to spue. Photomicrographs are given of sections of a number of different grades and manufactures of sole leather. The quality of leathers of grade I is the best, and of grade III the worst.

D. WOODROFFE.

Effect of heat on wetted vegetable-tanned leathers. IV. W. J. CHATER (J. Soc. Leather Trades' Chem., 1929, 13, 427—437).—The effect of various buffer solutions on the shrinkage temperature of calf pelt has been determined, and also shrinkage curves for a number of insole leathers, and various other leathers including sulphur-tanned formaldehyde leather and several defective leathers. The initial shrinkage temperatures of chrome-tanned leathers stripped with Rochelle salt, $0.1N$ -sodium hydroxide, and pyridine, respectively, were 81° , 65° , and above 95° .

D. WOODROFFE.

PATENTS.

Tanning substances. J. R. GEIGY, SOC. ANON. (J. R. GEIGY, A.-G.) (B.P. 305,013, 12.9.28. Ger., 28.1.28).—Tanning agents are prepared by condensing in strongly acid solution water-soluble sulphonation products of phenol or its derivatives with formaldehyde or material yielding it, in the presence of urea, or material yielding urea, or of previously prepared urea-formaldehyde condensation products, and subsequently neutralising the products with alkalis.

L. A. COLES.

Tanning-material products. R. AISTRUP (B.P. 320,344, 7.7.28).—Extracts of tanning materials are concentrated in a triple-effect apparatus to a water content of 60—70%, then spray-dried to produce a

thread-like product of 1—5% water content which may be briquetted.

D. WOODROFFE.

Manufacture of a leather substitute. J. M. GONNISEN (B.P. 320,444, 31.7.28).—Celluloid waste, e.g., scrap film, is dissolved in acetone and ethyl acetate, kneaded together with leather dust, compressed, and dried.

D. WOODROFFE.

Production of tough transparent foils from gelatin. O. KLOTZ (B.P. 320,509, 24.9.28).—On a backing or an endless running band, gelatin and rubber latex are alternately applied to form a film of superimposed gelatin and rubber layers. The outer layers consist of gelatin. The product is dried, stripped off, and wound.

D. WOODROFFE.

Bleaching of skins (B.P. 318,471—2).—See VI. **Vegetable-glue base** (U.S.P. 1,726,824).—See XVII.

XVI.—AGRICULTURE.

Soil genetics. I. H. T. JONES and J. S. WILLCOX (J.S.C.I., 1929, 48, 304—308 r).—Work which has been carried out on seven Millstone Grit profiles from various parts of Yorkshire is described. The results of analyses of these profiles are given and evidence is adduced to show that the soils belong to the "podsol" class, the nature of which is explained. The various current theories of iron-pan formation are briefly discussed. Experiments are described on which a further explanation is based. It is shown that tartaric acid and oxalic acid extracts of soil deposit compounds of iron on keeping. It is suggested that sesquioxides are dissolved by soil organic acids, the iron and aluminium entering into the electronegative portion of the molecule; these compounds are then leached through the soil in solution and are ultimately precipitated as basic salts, thus giving rise to a zone of sesquioxide accumulation and finally to a pan.

Adsorbing soil complex and adsorbed cations as a basis for genetic classification of soils. K. K. GEDROIZ (Kolloidchem. Beih., 1929, 29, 149—260).—The following classification of soils is proposed. (1) Soils containing no hydrogen in the adsorbing complex (soils saturated with bases). These are divided into two sub-groups: (a) Soils which contain adsorbed calcium or magnesium in the adsorbing complex. The adsorbing complex is in the form of aggregates of the primary colloidal particles, the aggregates having, in general, a diameter greater than 0.1μ ; the aggregates are broken down, dissolved, or chemically decomposed by water only with difficulty. (b) Soils containing adsorbed sodium in addition to calcium and magnesium; the sodium salts may be present in an easily soluble excess or may be present in small amount strongly adsorbed; in this case the ability of water to break up the aggregates and to dissolve and chemically decompose the constituents of the complex is greater than for any other type of complex. (2) Soils containing adsorbed hydrogen in the adsorbing complex. In regard to the loosening and decomposing action of water, these complexes occupy an intermediate position between those containing only adsorbed calcium and magnesium and those partly or completely saturated with sodium.

E. S. HEDGES.

Hypothesis of "unfree water" in soils. A. N. PURI (Agric. J. India, 1929, 24, 318—324).—Four dry soils tested were unable to remove any water from sugar solutions of concentrations 4—8%. At higher concentrations there was evidence that a more dilute solution was taken up by the soil, leaving the sugar solution more concentrated, but it is considered unlikely that any "unfree water" existed in these soils.

E. HOLMES.

Reaction between soils and metallic iron. H. D. HOLLER (J. Washington Acad. Sci., 1929, 19, 371—378).—Evolution of hydrogen from equal weights of soil and steel turnings or pulverised cast iron in water serves as a convenient guide to the relative corrosiveness of different soils on iron. The amount of hydrogen evolved is not dependent on p_H alone, but if a soil has a low p_H value together with material capable of buffering the solution at this p_H , then a high rate of evolution may be expected. Clays and silt loams are therefore more active than sands. The activity of a soil on iron as measured by hydrogen evolution bears a direct relation to soil acidity as measured by titration. In the absence of air or oxygen, hydrogen evolution takes place up to p_H 9.4.

E. HOLMES.

Dispersing soils for mechanical analysis. A. N. PURI (Agric. J. India, 1929, 24, 330—331).—It is claimed that the dilute hydrochloric acid used for the preliminary treatment of soils for mechanical analysis, by the method recommended by the International Society of Soil Science, may be replaced with advantage by *N*-sodium chloride.

E. HOLMES.

Composition of commercial acid lead arsenate and its relation to arsenical injury. H. S. SWINGLE (J. Agric. Res., 1929, 39, 393—401).—At low concentrations (less than 0.01% As_2O_5) arsenic and arsenious acid solutions were equally injurious to peach foliage. Arsenic acid is the more injurious at the higher concentrations, the increased toxicity corresponding to p_H 2.6 in the solution. The minimum concentration of As_2O_5 harmful to peach leaves was 0.0012%. Acid lead arsenate containing less than 0.25% of soluble arsenic produced minimum leaf injury. Reduction of soluble arsenic in this substance sufficiently to prevent injury appears impossible. The initial concentration of soluble arsenic (within the limits of ordinary spray preparations) had little or no effect on the toxicity of lead arsenates to insects.

A. G. POLLARD.

Effect of external growth conditions on germination of cereals in sugar solutions. K. MEYER (J. Landw., 1929, 77, 97—138; cf. B., 1928, 421).—The germination of individual species of cereals varies with their origin, age, and method of storage. The significance of the suction power of seeds as a measure of their soundness is therefore restricted. It is necessary to differentiate between the osmotic power and germinative capacity of seeds in the application of these methods.

A. G. POLLARD.

Reduction of soil nitrates during the growth of soya beans. E. P. DEATRICK (J. Amer. Soc. Agron., 1928, 20, 947—958).—Nitrates in soils under maturing soya beans are very low. A long period for nitrification between harvesting and replanting is desirable.

CHEMICAL ABSTRACTS.

Sugar beet top silage. A. W. OLDERSHAW (J. Min. Agric., 1929, 36, 634—640).—Good silage may be made from sugar beet tops. Two analyses record: starch equivalent 5.6, 6.8; nutritive ratio 1:6.7, 1:7.7. The inclusion of adhering soil in the silo must be minimised. Pit- and trench-silos must be well drained; and clamp-silos made on the ground level should be covered with straw and banked with soil to a depth of 1 ft.

A. G. POLLARD.

Low-temperature tar as fungicide. EHRMANN.—See II. **Liquorice root.** HOUSEMAN and LACEY.—See XX.

PATENTS.

Manufacture of fertilisers containing ammonium nitrate. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 320,708, 7.8. and 21.9.28).—Finely-divided calcium carbonate, other than that obtained in the manufacture of ammonium sulphate from ammonium carbonate and calcium sulphate, is mixed intimately with fused ammonium nitrate, or with a hot, concentrated solution of it, and the product is solidified into granules by spraying or by compression between cooled rollers.

L. A. COLES.

Insecticide. Parasiticide. R. G. MEWBORNE, Assr. to CONSUMERS TOBACCO Co. (U.S.P. 1,731,677—8, 15.10.29. Appl., [A] 6.10.25, [B] 23.10.25. Renewed [A] 13.1.28, [B] 6.8.27).—(A) Tobacco is treated with an alkali in the presence of moisture, and with sufficient dehydrating agent to give a dry, readily pulverised product. (B) Tobacco is mixed with bentonite. The product yields free nicotine on mixing with water, and may be used as a dip.

F. G. CLARKE.

Insecticidal solution. A. W. C. MENZIES (U.S.P. 1,732,240, 22.10.29. Appl., 7.2.27).—An aqueous solution containing about 0.5% of sodium fluoride and about 0.2% of sodium taurocholate and sodium glycocholate is claimed.

H. ROYAL-DAWSON.

(A) **Extirpation of insects noxious to plants.** (B) **Insecticide.** E. VON AMMON and K. SZOMBATHY (B.P. 294,646—7, 10.7.28. Ger., 29.7.27).—(A) Insects which are protected by enveloping webs may satisfactorily be sprayed with mixtures of the alkaloid lupanine (0.3%), an alcohol such as amyl alcohol (1.5%), and an alkali, e.g., caustic potash (0.2%). (B) Insects which are normally protected by a layer of waxy secretion, particularly *Schizoneura lanigera*, may be killed by spraying with an emulsion containing lupanine (0.25%) and resinic salts and free resinic acid (1.2%) which dissolve the wax layer.

E. HOLMES.

Juices from plants (B.P. 320,369).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Colloids in molasses. A. VON BRODOVSKI (Kolloid-chem. Beih., 1929, 29, 261—353).—An investigation has been made of the physico-chemical properties of beet-sugar molasses, and the various colloidal constituents have been isolated and analysed. The surface tension of the solution of molasses at first falls with increasing concentration, then passes through a minimum, and thereafter rises. The surface tension is highest for neutral solutions. The relative viscosity of the solution of molasses is lower than that of a sucrose solution of

the same concentration, and little effect is produced by altering the hydrogen-ion concentration, although the viscosity rises in strongly alkaline solutions. The colour of the solutions deepens with increasing p_H . The colloid content of the molasses amounts to 1.07%, and the isolated colloids are divided into two groups according to whether they are soluble or insoluble in water (reversible or irreversible). The greater part of the colloidal material is insoluble in water, ether, alcohol, or benzene. In the irreversible part of the colloidal material can be distinguished three fractions with acid reaction and three fractions which behave amphotERICALLY. The reversible part consists of at least two substances, but it was not possible to fractionate it; about 25% consists of araban, and the presence of hexoses in the products of hydrolysis was established. The irreversible acid fractions are soluble in pyridine, whilst the amphoteric fractions and the water-soluble colloids are not. All the isolated fractions contain nitrogen (about 7.5–8.7% in the irreversible colloids and 4% in the reversible colloids). The irreversible colloids are also richer in carbon. Phosphorus is also present. One of the acid fractions appears to be similar to the fuscazinic acid isolated by von Staněk (cf. B., 1917, 935); it is tri- or tetra-basic. It has not been possible to recognise the presence of albumins, and it is suggested that these may have suffered degradation during the manufacturing process. Salts of most heavy metals precipitate the colloids, but mercuric chloride is a notable exception; the salts of the alkaline earths have only a small precipitating power. In feebly acid solution the reversible colloids lower the surface tension of water more than do the irreversible colloids, but the reverse is the case in weakly alkaline solutions; the viscosity of the reversible colloids is greater than that of the irreversible colloids. The colouring matter in molasses is in the irreversible colloidal part. When the isolated colloids are added to sucrose solutions the surface tension of the latter is reduced, but practically no effect is produced on the viscosity. E. S. HEDGES.

Advantageous zone of p_H for [beet sugar] refinery liquors. M. I. NAKHMANOVICH (Nauch. Zapiski Sak. Prom., 1929, 7, 265–281).—Filtration is best effected at p_H 7.0–7.5. Destruction of sugar begins below p_H 7.0, whilst colour is developed by heating at p_H 8.

CHEMICAL ABSTRACTS.

Automatic p_H recorders for sugar-refinery alkalinity control. A. L. HOLVEN (Ind. Eng. Chem., 1929, 21, 965–970).—Various methods for the automatic determination of p_H have been investigated in order to control the addition of lime in the refining of sugar products, but none has proved entirely satisfactory. The quinhydrone, the antimony, and the tungsten-manganese sesquioxide electrodes were found to be unreliable, owing to failure to hold the calibration (cf. Parker, B., 1927, 634; 1928, 627). The bare-wire tungsten electrode in conjunction with a calomel half-cell (cf. Balch, B., 1928, 461; 1929, 32) proved more suitable, but required elaborate compensatory devices owing to the variations of temperature, viscosity, density, and chemical character of the products to be tested. The temperature coefficient of the electrode is about 2 millivolts, and was compensated by a

thermopile of 50 copper-constantan couples giving the same current but in opposition to the electrode. The difference in the readings for two different products at the same p_H was found to be constant, so that the zero of the electrode could be adjusted for each product by means of a cell and potentiometer. Screening of the liquors also presented difficulty as the electrode is very susceptible to poisoning. It is concluded that, although more sensitive, the method offers no advantage over the colorimetric method. H. J. DOWDEN.

Application of the Selivanov reaction [for detection of lævulose]. S. MALOWAN (Chem.-Ztg., 1929, 53, 800; cf. Ofner, B., 1929, 832).—The colour given by lævulose in the Selivanov test is attributed not to the sugar itself but to its aldehyde and ketone decomposition products; the coloured compounds are in the nature of xanthene derivatives. A. R. POWELL.

Acetic and lactic acids. ALLGEIER and others.—See XVIII. **Sodium glutamate.** HAN.—See XIX.

PATENTS.

Sugar manufacture. D. STEWART & Co., LTD., and J. B. TALBOT-CROSBIE (B.P. 320,826, 14.1.29).—In the diffusion process where carbonatation is employed, the liquor from exhausted pulp is mixed with press cake, thereby reducing acidity of the liquor, then filtered, and used again at any suitable stage in the diffusion.

E. B. HUGHES.

Recovery of sucrose from cane molasses. H. DE F. OLIVARIUS, Assr. to CALIFORNIA PACKING CORP. (U.S.P. 1,730,473, 8.10.29. Appl., 22.12.25. Renewed 25.2.29).—Invert sugar present is eliminated by fermentation, an alkaline-earth oxide or hydroxide and alcohol are added to precipitate organic impurities, and the liquor is filtered. Sucrose is then precipitated by means of an alkaline-earth oxide or hydroxide, after distilling off the alcohol. F. G. CLARKE.

Treatment of starch. INTERNAT. PATENTS DEVELOPMENT Co. (B.P. 294,979, 3.7.28. U.S., 3.8.27).—Modified starches (from thin-boiling starch to highly-soluble dextrin) are prepared by drying to 2% of moisture, then treating with chlorine gas (0.03–0.12% of the weight of starch) at 115–135° for at least 2 hrs. E. B. HUGHES.

Manufacture of dry colour-binding agents soluble in cold water. HENKEL & CIE., G.M.B.H. (B.P. 289,053, 12.4.28. Ger., 21.4.27).—Gelatinised starch preparations soluble in cold water, obtained by heating starch with water or by treating it with sodium hydroxide, with magnesium, zinc, or calcium chloride, or with ammonium thiocyanate, are slightly disintegrated by treatment with, e.g., hydrogen peroxide, per-salts, hypochlorites, or chloroamines, in the presence of alcohol as diluent, and the products are dried and ground. Alternatively, disintegration can be effected before or during gelatinisation. L. A. COLES.

Vegetable-glue base. E. H. HARVEY, Assr. to PERKINS GLUE Co. (U.S.P. 1,726,824, 3.9.29. Appl., 26.1.23).—Dry starch substantially free from gluten is mixed with not more than 5% of a non-hygroscopic oxidising agent, e.g., barium dioxide, which does not convert the starch on admixture, but renders the finished glue suitably fluid on addition of water or caustic alkali solution. F. R. ENNOS.

Carbohydrate esters or ethers (B.P. 293,316 and 293,757).—See V. Yeast (U.S.P. 1,732,922).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Nitrogen requirements of yeast. A. A. D. COMRIE (J. Inst. Brew., 1929, 35, 541—547).—The author has reviewed the data published on the nitrogenous nutrients of yeast. From 0.025—0.026% of the total nitrogen is assimilated by yeast during fermentation of wort under brewery conditions. Data relating to the most propitious state of combination of the nitrogen are contradictory, and although leucine, asparagine, and ammonium sulphate are quoted as substances favourable to yeast, on occasion, they are condemned by other workers as detrimental to fermentation. Substances in which the nitrogen is not combined either as amino-nitrogen or ammonia-nitrogen, such as alkaloids or pyridine, are of no value. Protein, nitrates (other than ammonium), and, according to one authority, acid amides, excepting formamide and those of a complex nature, are non-assimilable. Peptones, aliphatic amines, aromatic amines with side-chain nitrogen, vitamins, diastase, and amino-acids, in general, are stated to be assimilable. The examination of the published work has also yielded a great number of compounds of known chemical constitution with possible nutrient properties which have been classified according to their assimilability by yeast and their action on fermentation.

C. RANKEN.

Methylene-blue studies [in the staining of yeast]. J. FUCHS (Woch. Brau., 1929, 46, 437—440).—By observation of individual cells of yeasts 8—30 days old in water suspension it was noted that certain cells remained unstained for as much as 30 min. and then stained dark blue in a few seconds. In other cases staining occurred gradually after several hours. These two types of resistant cell were characterised by bright plasma and well-defined vacuoles. The proportion of cells stained at once by methylene-blue in concentrations of 0.001, 0.0002, and 0.0001% differs little, but the difference increases and is marked at 15 min. Stained cells may become decolorised on keeping owing to reductase action, and this is hastened by slight acid reaction of the medium. Certain weakly stained cells are capable of reproduction; these are considered to be stained only on the surface. In testing yeast a 0.0001% solution of methylene-blue should be employed, and only sufficient added to impart a greenish-blue colour to the suspending medium. Only deeply stained cells should be considered as dead (cf. B., 1929, 792).

F. E. DAY.

Brewers' pitch [for lining casks]. F. KUTTER (Woch. Brau., 1929, 46, 357—361, 372—376).—A cask-lining composition was heated at 170° and 200° under various conditions of aeration. The loss in weight was noted at intervals, also the softening and melting points as determined by the Hoffmann-Herbst and Klinger-Lampe methods, and the viscosity. At 170° in a closed or open vessel without aeration the physical constants were little changed, with 0.6 litre of air per hr. there was comparatively slight change, but with 1.2 litres/hr. a decided raising of the m.p. and viscosity took place after 67 hrs. This was much more marked

with 1.0 litre/hr. of air at 200°, the m.p. (Hoffmann-Herbst) rising from 50.7° to 76.5°, and the viscosity at 140° from 1.80 to 23.25 (Engler). Correspondingly the cooled material became darker, harder, and more brittle. In the chemical examination the neutral and acid constituents were determined (cf. B., 1929, 34), and in place of the "oxidation value" a bromine value was determined by treating 0.7 g. of the material dissolved in 10 c.c. of glacial acetic acid for 15 min. at 70° with 27—30 c.c. of a solution (N) of bromine in glacial acetic acid. The reaction flask was closed with a ground-in two-bulbed absorption funnel containing 8 c.c. of 0.5N-potassium iodide. This solution prevented escape of bromine, and on cooling it was drawn back into the flask; the iodine liberated by the excess of bromine was titrated with 0.5N-thiosulphate. The bromine value was found to fall during heating with aeration in a manner corresponding to the changes in the physical properties. The proportions of neutral and acid constituents showed more complex changes, the latter at first decreasing, then increasing in amount. It is suggested that this is possibly due to loss of volatile acids in the early stages, followed by oxidation of neutral to acid substances later, accompanied by rise of m.p. and viscosity. An apparatus suitable for carrying out the hot aeration test is figured.

F. E. DAY.

Mechanism of enzyme action and its action in breadmaking. F. F. NORR (Z. angew. Chem., 1929, 42, 1022—1025).—Various theories of the mechanism of the action of yeast-cell enzymes on hexoses are discussed. From experiments on the action of yeast on dough under various physical conditions, a theory of the mechanism of enzyme action is developed based on the equilibrium at and adsorption on the yeast-cell wall. This theory serves to explain the observed phenomena of stimulation and depression of fermentation.

A. A. GOLDBERG.

Production of acetic and lactic acids [by fermentation] from mill sawdust. R. J. ALLGEIER, W. H. PETERSON, and E. B. FRED (Ind. Eng. Chem., 1929, 21, 1039—1042).—Fir, spruce, and pine sawdust hydrolysed by sulphuric acid gives wood-sugar liquors fermentable by a unique lactic acid organism capable of fermenting both pentoses and hexoses (cf. Marten and co-workers, B., 1927, 889). Fermentation is carried out in presence of 5% (wt./vol.) of malt sprouts as a source of nitrogen and excess calcium carbonate, which may be added at the commencement or at intervals, as neutralising agent. The presence of added colloids or phosphates has no accelerating effect on the fermentation. Liquors of 7—10% concentration of sugar are fermented in 7—9 days with destruction of about 85% of the sugar, which is almost quantitatively recovered as products. Dilution of solutions aids fermentation. The mixed acid obtained contains, in small- and large-scale experiments, respectively, about 10 and 5% of acetic and about 90 and 95% of inactive lactic acid. Soft-wood liquors are more readily fermented than those from hard woods. The dextrose tetrameride produced by the Rheinau process (cf. J.S.C.I., 1926, 45, 267 r) after first hydrolysing with water is as readily fermented as are the liquors from the sulphuric acid process, and yields similar products.

D. W. HILL.

Recalculation of the densities of water-alcohol mixtures from the experimental data of Mendeléev. A. N. GEORGIEVSKI (J. Appl. Chem., Russia, 1928, 1, 207—219).

Densities of water-alcohol mixtures. B. M. KOYALOVICH (J. Appl. Chem., Russia, 1928, 1, 219—260; cf. also Vredski, *ibid.*, 1928, 1, 160).

PATENTS.

Production of yeast. J. R. WHITE, Assr. to H. LEEDS (U.S.P. 1,727,847, 10.9.29. Appl., 29.4.27. Renewed 31.1.29).—Seed yeast is successively propagated in a series of sacchariferous solutions, each containing 0.5—5% of sodium chloride.

H. ROYAL-DAWSON.

Manufacture of diastatic composition. J. TAKAMINE, JUN., Assr. to TAKAMINE FERMENT CO. (U.S.P. 1,731,400, 15.10.29. Appl., 20.8.18).—The composition is precipitated from a diastatic solution containing sodium bicarbonate, by means of magnesium chloride.

F. G. CLARKE.

Cooling of worts during fermentation. F. E. LICHTENTHAELER (U.S.P. 1,731,073, 8.10.29. Appl., 14.3.25).—The carbon dioxide produced during fermentation is drawn off, compressed, cooled, and passed again into the wort.

A. COUSEN.

Aerobic fermentation. G. S. BRATTON, Assr. to ANHEUSER-BUSCH, INC. (U.S.P. 1,732,921, 22.10.29. Appl., 6.3.26).—A stream of air is circulated over and over again through the fermenting material, and the loss of oxygen is compensated by replacing part of the escaping exhaust air by an equal quantity of fresh air.

C. RANKEN.

Production of citric acid by fermentation. MONTAN U. INDUSTRIALWERKE VORM. J. D. STARCK (B.P. 302,338, 7.9.28. Czechoslov., 16.12.27).—Mashes such as are used in the production of alcohol are fermented by moulds which have been cultivated on solid vegetable culture media containing citric or tannic acid and vitamins. Nutrients which include a certain proportion of organic amino-acids are added in amount so that the growth of the moulds is restricted to a minimum necessary for the optimum conversion of sugar into citric acid. Destruction of the citric acid is prevented by "protective" substances, *e.g.*, hydrolysed glue, which unite with the acid to form complexes of high mol. wt. and which do not readily penetrate the cells of the fungi.

C. RANKEN.

Manufacture of yeast. R. HAMBURGER, S. KAESZ, and F. HARTIG, Assrs. to STANDARD BRANDS, INC. (U.S.P. 1,733,962, 29.10.29. Appl., 11.11.24. Ger., 3.3.24).—See B.P. 230,050; B., 1924, 896.

Sucrose from cane molasses (U.S.P. 1,730,473).—See XVII.

XIX.—FOODS.

Determination of carotin in flour. C. G. FERRARI and C. H. BAILEY (Cereal Chem., 1929, 6, 347—371; cf. Coleman and Christie, B., 1927, 396).—The carotin content of flour is determined by spectrophotometric examination of a gasoline extract of the flour. The extract should be perfectly clear or clarified by filtration through selected alundum thimbles or by means of a

capillary siphon. Filter paper and unglazed porcelain adsorb carotin. The amount of carotin extracted by gasoline from flour depends on the ratio of solvent to sample; for 100 c.c. and 20 g. about 10% of the carotin remains unextracted. Results are given for the carotin content of some varieties of Canadian wheat and also for the distribution of carotin in the various fractions of flour in milling.

E. B. HUGHES.

Loaf volume as produced by different flours under prolonged fermentation. R. J. CLARK (Cereal Chem., 1929, 6, 338—344).—An extension of the experimental baking test on flour is described in which a series of doughs made from the same flour are fermented for varying periods. In curves connecting loaf volume and fermentation period, a major peak is obtained corresponding with the best-quality loaf; certain minor peaks also appear, the significance of which is discussed.

E. B. HUGHES.

Composition of milk. O. R. OVERMAN, F. P. SANMANN, and K. E. WRIGHT (Univ. Ill. Agric. Exp. Sta. Bull., 1929, No. 325, 174 pp.).—A statistical study of results obtained with 1998 samples shows that the ratio protein:fat is higher than is usually reported, and that the lactose is nearly constant up to 5% of fat and decreases for higher percentages of fat.

CHEMICAL ABSTRACTS.

Influence of acidity on the coagulation of milk. C. PORCHER (Rev. gén. Colloid., 1929, 7, 260—270).—The times of clotting of milk with different rennins are studied under varying conditions of acidity with hydrochloric, acetic, and lactic acids. Carbon dioxide assists clot formation by forming hydrogen ions and by its effect on the solubility of the calcium salts present in the milk, the increase in the concentration of hydrogen and calcium ions favouring coagulation; in this way it is possible to coagulate "lazy," bad, or alkaline milks.

F. R. ENNOS.

Salts in milk. Heat coagulation of evaporated milk. H. H. SOMMER (Butter Cheese J., 1929, 20, No. 11, 44—48).—The effect of salts present in milk on its coagulation by heat is discussed. The amounts of disodium hydrogen phosphate or sodium citrate necessary to remove excess of calcium do not exceed 0.007% (as P_2O_5) and 0.092% (as citric acid), respectively. The effect of sodium hydrogen carbonate is to convert sodium dihydrogen into disodium hydrogen phosphate.

CHEMICAL ABSTRACTS.

Volatile constituents of "miso." R. TAKATA (J. Soc. Chem. Ind. Japan, 1929, 32, 628—629).—The average percentages of the volatile constituents of "miso" are: total volatile material (chiefly water) 49.38, alcohol 1.38, carbon dioxide 0.034, acetone 0.013, acetic acid 0.011, and volatile esters 0.039%. The sweet varieties of "miso" contain larger amounts of alcohol, acetic acid, and esters than the salty varieties, but the amount of acetone increases as the ripening of "miso" proceeds.

K. KASHIMA.

Dried fruit grubs. Ethylene dichloride-carbon tetrachloride fumigation process. J. E. THOMAS (J. Council Sci. Ind. Res., Australia, 1929, 2, 128—133).—A mixture of ethylene dichloride (3 vols.) and carbon tetrachloride (1 vol.) used at the rate of 14 lb. per 1000

cub. ft. at temperatures above 21° for not less than 22 hrs. in airtight compartments sterilises dried fruit as regards *Plodia interpunctella* and presumably also as regards *Ephestia cautella*. W. J. BOYD.

Low-temperature preservation of foodstuffs. T. MORAN (J.S.C.I., 1929, 48, 245—251 T).—The part played by temperature in the preservation of foodstuffs is reviewed. With living foodstuffs recent work has emphasised the importance of a limited temperature range, and in turn the concept of an optimum temperature for storage. With dead foodstuffs temperature is important in restricting the activities of moulds, bacteria, and enzymes. An account of recent work on the freezing of tissues is also given.

Monosodium glutamate as chemical condiment. J. E. S. HAN (Ind. Eng. Chem., 1929, 21, 984—987).—Monosodium glutamate has a decided meat-like taste and is used largely for flavouring vegetarian dishes. For the cheap commercial production of this condiment vegetable materials such as gluten, soya bean, and beetroot molasses are hydrolysed by hydrochloric acid. Separation of the products may be effected by electrolysis in a three-chambered cell. Amphoteric and neutral products collect in the centre, whilst alkaline and acid products (e.g., glutamic and aspartic acids) collect in the respective outer compartments, which also contain the electrodes. In the usual method for manufacturing the monosodium salt, gluten obtained from Canadian wheat and concentrated hydrochloric acid are heated in stone vessels fitted with reflux condensers until the protein has dissolved, whereon the temperature is raised to boiling and maintained until hydrolysis is complete. The acid is then neutralised with soda and the glutamate is crystallised, dried, and powdered. In an improved method the protein is dissolved in hydrochloric acid at a temperature below the coagulating point, metallic tin is introduced to catalyse the hydrolysis and to remove arsenic, and the glutamate is separated by precipitation with alcohol. The neutralised glutamic acid hydrochloride, when evaporated, gives a product containing 26% of sodium chloride, and to avoid this contamination the neutralised solution is concentrated until most of the sodium chloride has crystallised, or, alternatively, the alkali equivalent of the hydrochloric group is added, whereon the sparingly soluble glutamic acid crystallises. H. J. DOWDEN.

Carbon dioxide in [mineral] water. ANON.—See VII. **Enzyme action and breadmaking.** NORD.—See XVIII. **Liquorice root.** HOUSEMAN and LACEY.—See XX.

PATENTS.

Manufacture of cereal meals. W. N. BOYES (B.P. 310,372, 27.3.29. N.Z., 24.4.28).—Cereal meals requiring no cooking are prepared from oats, wheat, etc. by treatment of the clean grain in a digester with steam at 6—15 lb. for about 2 hrs., cooling to 15.5° to condition, and then adding about 6% of sodium chloride; the product is dried, baked at about 60°, and again conditioned. E. B. HUGHES.

Preparation of milk, skimmed milk, or butter-milk, free of milk sugar [lactose] for sufferers

from diabetes. J. POHLMANN and J. R. F. RASSERS (B.P. 320,497, 13.9.28).—The casein is precipitated by slight acidification with the addition also of a small quantity of a decoction of carrageen. The precipitate is washed free from lactose, suitable mineral salts are added, and blood-serum (e.g., of the cow) and water are added to bring the whole into suspension. Cream washed with water may also be added.

E. B. HUGHES.

Treatment of sour cream. Treatment of soured or curdled milks for recovery of butter fat therefrom. A. L. RUSHTON, M. M. SIMPSON, and H. C. BECKMAN, ASSRS. to CREAM PROCESSES, INC. (U.S.P. 1,731,868—9, 15.10.29. Appl., [A] 27.2.28, [B] 6.6.28).—The cream or milk is rendered alkaline by alkali hydroxide and the fat is then separated. E. B. HUGHES.

Preservation of peanut butter. J. H. BEATTIE and A. M. JACKSON (U.S.P. 1,731,647, 15.10.29. Appl., 18.5.28).—To prevent separation of oil, peanut butter is sealed in airtight containers and subjected to the action of steam under pressure. E. B. HUGHES.

Cleansing and preparing for storage and market fruit after harvesting. I. F. LAUCKS, H. P. BANKS, and H. F. RIPPEY, ASSRS. to LAUCKS LABS., INC. (U.S.P. 1,732,816, 22.10.29. Appl., 11.10.26).—Fruit bearing residue from spraying is pretreated with trisodium phosphate solution. E. B. HUGHES.

Production of [concentrated] non-alcoholic fruit juices. G. SCHICHT A.-G. (B.P. 299,043, 9.10.28. Czechoslov., 21.10.27).—Fruit juices are sterilised and clarified by sterilising filters and then concentrated in high vacuum at temperatures not affecting colour, taste, or vitamins. E. B. HUGHES.

Preparation of fresh cocoa beans. T. ZELLER (B.P. 304,181, 29.6.28. Ger., 16.1.28).—Cacao beans are prepared in one operation by arresting the usual alcoholic fermentation before the commencement of acetous fermentation by heating for 24—30 hrs. at a temperature (optimum range 55—65°) high enough to destroy yeasts, bacteria, and moulds, and to kill the bean entirely, whilst not high enough to destroy the enzymes. The pulp is then removed by washing, after which the beans are left for 24 hrs. at 60° in moist air and then dried at 55—65°. E. B. HUGHES.

Refrigeration of products such as foodstuffs. J. E. W. REEH (B.P. 300,209, 8.11.28. Fr., 8.11.27).

[Brine tank] freezing of fish. GOVERNOR & COMPANY OF ADVENTURERS OF ENGLAND TRADING INTO HUDSON'S BAY, and C. TOWNSEND (B.P. 320,578 and 321,117, 16.1.29).

Carbon dioxide developing powder for foods, e.g., baking-powder (B.P. 303,353).—See VII. **Juices rich in vitamins** (B.P. 320,369).—See XX.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Arsenious iodide and its solutions. T. T. COCKING (Quart. J. Pharm., 1929, 2, 409—410).—An aqueous solution of arsenious and mercuric iodides (Donovan's solution), after keeping for 14 months in a well-stoppered vessel and being tested at intervals,

contained no arsenic in the arsenious state. The solution should either be freshly prepared before use or stored out of contact with air. E. H. SHARPLES.

Stability of extracts of ergot. F. WOKES (Quart. J. Pharm., 1929, 2, 384—395).—Effects of variations in the conditions of preparation and of storage on the deterioration of ergot extracts have been studied. Under the best conditions half the activity of liquid extracts is lost in from two to three months. This loss occurs in extracts made with hydrochloric acid (cf. Prybill and Maurer, B., 1928, 912), or with citric or tartaric acid, and with alcohol contents varying from 35 to 75%. The rate of deterioration is about twice as rapid at room temperature as at 0°, and still more rapid at 37°. Concentrated (soft) extracts may retain half their activity after storage for 4—9 months at 0°. E. H. SHARPLES.

Determination of "free nicotine" in tobacco: apparent dissociation constants of nicotine. H. B. VICKERY and G. W. PUCHER (J. Biol. Chem., 1929, 84, 233—241).—The fact that part of the nicotine of tobacco is "free" (i.e., can be removed by steam-distillation without addition of alkali) is due to hydrolysis of the nicotine salts, and the amount of such free nicotine depends therefore on the p_H of the sample. The electrometric titration curve of nicotine is given, from which the proportion of the total nicotine which is free in a tobacco extract of known p_H can be deduced.

C. R. HARRINGTON.

Liquorice root in industry. P. A. HOUSEMAN and H. T. LACEY (Ind. Eng. Chem., 1929, 21, 915—917).—Glycyrrhizin is contained in the roots of the plant, which after being dug are cured in the sun and baled. In the extraction the roots are shredded and subjected to the action of water under low-pressure steam. The extract is settled in cascade tanks, the liquor being concentrated under vacuum to d 1.14. This solution is then either treated in drum dryers for the preparation of powdered liquorice, or is further concentrated to a paste containing 20—25% of moisture; on cooling it sets to the characteristic semi-plastic mass. The extract may also be treated with dilute sulphuric acid in slight excess, the precipitated glycyrrhizic acid being washed, dissolved in ammonia, and dried for sale as ammoniated glycyrrhizin. A typical Spanish liquorice mass contains glycyrrhizin 10%, sugars 12%, starch and gums 25%, moisture 15—25%, and ash 5%, whilst Oriental liquorice contains 18% of glycyrrhizin. The bulk of the liquorice is used in the tobacco industry, the remainder being converted into pharmaceutical and confectionery products. Part of the spent roots are subjected to further extraction with 5% caustic soda at 100 lb. steam pressure, and this extract is used as a foam stabiliser in fire extinguishers, as a foaming agent in froth flotation, and as a spreader for insecticides. The tough fibrous residue is utilised in the manufacture of boxboard and wallboard, the latter possessing low thermal conductivity, high water-resistance, and excellent mechanical properties.

H. J. DOWDEN.

Determination of glycyrrhizic acid in liquorice root and extract. R. EDER and A. SACK (Pharm. Acta Helv., 1928, 4, 23—48; Chem. Zentr., 1929,

i, 3017).—Methods are reviewed, and details of the determination are given.

A. A. ELDRIDGE.

Examination of medicinal substances containing anthraglucosides. P. VAN DER WIELEN (Pharm. Weekblad, 1929, 66, 877—879).—The proposals for determination of hydroxyanthraquinone compounds in pharmaceutical preparations are discussed, and a standardised colorimetric method is described.

S. I. LEVY.

Boiling points of binary mixtures of ethereal oils. E. BRAUER (Ber. Schimmel, 1929, 151—161).—The b.p./10 mm. of a large number of binary mixtures of various composition of ethereal oils have been determined. In all the cases studied, with exception of the pairs cineole-*p*-cymene, and menthenone-safrole, the b.p. of the binary mixture deviates from the value calculated by the method of mixtures (Δt). Maxima are found in the following pairs, the figure in parentheses being the mol.-% of the first component: carvacrol-carvone (70), carvacrol-menthenone (70.2), carvacrol-pulegone (70.2), cineole-phenol (13.2), and menthenone-thymol (49.6), the b.p. being, respectively, 116.5°, 119.1°, 115°, 74.2°, and 116.5°/10 mm. Minima occur with the pairs: benzyl alcohol-safrole (93.1), citronellol-eugenol (90.4), citronellol-safrole (30.8), *p*-cymene-phenol (62.1), and safrole-terpineol (9.6), the b.p. being, respectively, 91.7°, 107.1°, 102.3°, 55.8°, and 96.4°/10 mm. The greater the difference in the b.p. of the two components the greater is the possibility of the existence of maxima and minima, providing the difference is not too great, since the pair *p*-cymene-terpineol which exhibits the largest deviation (Δt 17.3°, b.p. 60°/10 mm., for a mixture containing 53.5 mol.-% of *p*-cymene) shows no minimum on the curve. Menthenone has b.p. 103°/10 mm. (lit. 98°) and terpineol, b.p. 96°/10 mm. (lit. 104°).

J. W. BAKER.

Terpene- and sesquiterpene-free ethereal oils. T. KLOPPER (Ber. Schimmel, 1929, 167—178).—Various physical constants, solubility data in aqueous alcohol, and saponification and esterification values of the terpene- and sesquiterpene-free fractions of a number of essential oils have been determined and compared with the corresponding values of the natural oils. The values of d_{25}^{25} and α_D (1 in 1-dm. tube) for such fractions of the following oils are, respectively: Japanese valerian oil 0.990, -20°; Réunion basil oil 0.980, +1.5°; birch-bud oil 0.980, -13°; tarragon oil 0.9754, slightly dextrorotatory; fennel oil 0.985, +3° (solidification point +17°); galbanum oil 0.985, slightly levorotatory; helichrysum oil, 0.9294, +0.9°; Roman camomile oil 0.914, slightly dextrorotatory; pine-needle oil 0.960, -20°; niaouli oil 0.937, slightly levorotatory; origanum oil 0.937, -6°; parsley-seed oil 1.125, 0°; American tansy oil 0.932, +38°; West Indian sandalwood oil 0.985, +4°; sassafras wood oil 1.095, slightly dextrorotatory; star anise oil 0.982, slightly levorotatory; wine-yeast oil 0.879, slightly dextrorotatory.

J. W. BAKER.

Testing of blossom extracts. I. H. WALBAUM and A. ROSENTHAL (Ber. Schimmel, 1929, 187—202).—Various physical and chemical methods for the detection of impurities in commercial blossom extracts are

described and the mean values of the various physical constants of characteristic samples are tabulated. Solid extracts (extracted with light petroleum) are best characterised by their solidification points and their saponification values, the following being, respectively, the mean values of these constants for various essences: *jasmin* 49.8°, 106.2; *Bulgarian rose* 44.4°, 37.3 (the presence of light petroleum lowers both values); *orange blossom* —, 97.07; *lavender* 15.4°, 111. Fluid extracts (extracted with alcohol) are best characterised by their saponification values, density, and solubility in dilute alcohol. Adulteration with ethyl phthalate is determined by hydrolysis with 0.5*N*-alcoholic potassium hydroxide, filtering, drying at 110°, and weighing the insoluble potassium phthalate; this weight multiplied by 0.917 gives the weight of ethyl phthalate in the weight of extract taken. A reflux apparatus is described for the determination of waxes, resins, etc. in extracts, in which the whole of the essential oil from a known weight of extract mixed with salt is steam-distilled, using a controlled air-bath at 182° (boiling tetralin), and continuously extracted with ether. The weight of residue from the dried ethereal extract is the amount of pure essential oil present. For detailed constants of various extracts the original must be consulted.

J. W. BAKER.

Japanese peppermint oil. H. WALBAUM and A. ROSENTHAL (Ber. Schimmel, 1929, 203—207).—The presence of Δ^8 -hexenol, b.p. 55—56°/9 mm., d^{15}_4 0.8508, n^{20}_D 1.48030, in Japanese peppermint oil (cf. A., 1918, i, 302) is confirmed by its catalytic reduction with nickel and hydrogen to *n*-hexyl alcohol. Hydrolysis of the ester fraction of high b.p. yields phenylacetic acid, which is separated from liquid acids by centrifuging. Fractionation of the latter gives Δ^8 -hexenoic acid, b.p. 98°/5 mm., m.p. 34—35°, identical with a specimen synthesised by condensation of *n*-butaldehyde with malonic acid in the presence of pyridine and purification through its ethyl ester, and is converted into α - β -dibromohexenoic acid by the action of bromine in carbon disulphide. From the acid fraction, b.p. 160°/4 mm., is obtained a *methyl* ester, b.p. 112—113°/4 mm., d^{15}_4 0.9719, n^{20}_D 1.44129, corresponding with an acid $C_{11}H_{22}O_2$, and an acid $C_{12}H_{18}O_2$, b.p. 156—157°/4 mm. The fraction, b.p. 170—180°/4 mm., crystallises, the equivalent corresponding with an acid $C_{15}H_{24}O_2$.

J. W. BAKER.

PATENTS.

Manufacture of juices rich in vitamins, from plants. J. KORSALT (B.P. 320,369, 2.7.28).—Raw plant juices rich in vitamins may have their chlorophyll, albumin, and oxalic acid removed, and the resultant juices rendered sterile, by treatment with calcium hydrogen citrate, lactate, or tartrate etc., followed by heating to 100° in an inert atmosphere such as nitrogen. [Stat. ref.]

E. HOLMES.

Manufacture of preparations having an action resembling that of tuberculin. I. G. FARBERIND. A.-G. (B.P. 295,629, 27.7.28. Ger., 15.8.27. Addn. to B.P. 285,087; B., 1929, 661).—The process of the prior patent is applied to preparations of *B. coli*.

F. G. CROSSE.

Artificial ageing of tobacco. T. H. KELLER (U.S.P. 1,729,482, 24.9.29. Appl., 30.12.27).—The tobacco is moistened with an extract of cereal grass, packed, and sweated.

H. ROYAL-DAWSON.

Curing of tobacco. W. E. LILIENFIELD, Assr. to LILIENFIELD BROS. & Co. (U.S.P. 1,731,018, 10.8.29. Appl., 17.3.27).—Loosened tobacco leaves, after being aged and fermented, are subjected to artificial ultra-violet rays for $\frac{1}{4}$ —2 hrs. to produce the desired mildness.

F. G. CLARKE.

Manufacture of synthetic camphor. L. DUPONT (U.S.P. 1,733,218, 29.10.29. Appl., 5.6.26. Fr., 12.6.25).—See B.P. 253,542; B., 1927, 828.

Complex metallic compound of pyrocatechol. H. SCHMIDT, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,718,492, 25.6.29. Appl., 30.4.27. Ger., 6.2.24).—See B.P. 295,734; B., 1928, 799.

Manufacture of sterols. A. GAMS and F. LOCHER, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,733,009, 22.10.29. Appl., 7.6.28. Switz., 14.6.27).—See B.P. 292,133; B., 1929, 737.

Préparation of α -hydrazino- β -nitropyridine. C. RÄTH (U.S.P. 1,733,695, 29.10.29. Appl., 1.2.26. Ger., 5.2.25).—See B.P. 255,811; B., 1927, 29.

Decreasing the toxic action of cocaines. R. ECKERMANN (U.S.P. 1,733,298, 29.10.29. Appl., 26.6.26. Swed., 13.3.26).—See B.P. 267,463; B., 1927, 974.

Colloidal iodine (B.P. 320,719).—See VII. Milk for diabetics (B.P. 320,497).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Panchromatic silver salt sensitisers and their relationship to the theory of hypersensitisation and sensitisation. A. STEIGMANN (Z. wiss. Phot., 1929, 27, 117—118).—Panchromatic sensitising agents are prepared by treating eosins mixed with sodium sulphide with a mixture of sulphuric and nitric acids, and then reducing the product with hyposulphite. These substances sensitise silver chloride directly, but do not sensitise negative emulsions unless chloride is present (cf. A., 1928, 602).

R. CUTHILL.

Desensitisers. II. (MISS) F. M. HAMER (Phot. J., 1929, 69, 409—415; cf. B., 1929, 872).—An investigation of various *p*-dialkylaminoanils of quinoline-2- and -4-aldehyde methiodides and ethiodides shows that the desensitising capacities of these compounds is comparable with that of safranin. Although this property is somewhat decreased in the presence of sulphite-carbonate solutions, they are, however, very efficient when used in a preliminary bath, owing presumably to the stable silver halide adsorption complexes formed. Their breakdown products, however, attack the latent image. The *p*-dimethylaminoanil of benzthiazole-aldehyde ethiodide, a strong desensitiser, gave chemical fog, whilst the similar anils of pyridine-2-aldehyde methiodide and ethiodide lost their desensitising capacity almost completely in sulphite-carbonate solutions, and could not be used in a preliminary bath. The substitution of acidic, basic, and acylamino-groups in various

aminoanils of quinoline-2-aldehyde methiodide cause no marked changes in the desensitising capacity, but introduction of acid substituents appeared to cause failure. Examination of a number of cinnamylidene derivatives of 2-methylquinoline alkyl salts gave only desensitisers inferior to safranin, whilst the desensitising capacity of a series of styryl compounds were shown to be completely destroyed by the sulphite in the developer. It is therefore concluded that at present there are no useful desensitisers to be derived from quinoline or related bases. J. W. GLASSETT.

Diffuse illumination of photographic plates and photographic photometry. A. DENISSOV (Z. wiss. Phot., 1929, 27, 128—144).—The effect on the relative blackening and contrast of a photographic plate of fogging by exposure to diffuse light of the same quality before, during, or after the normal exposure has been investigated. For densities not greater than unity diffuse illumination increases the relative density, and for densities of 0.05 and below the contrast is increased. These facts may be turned to advantage in photographic photometry when very small amounts of light are being dealt with. Prolonged exposure to diffuse light or exposure to intense diffuse light reduces the contrast. R. CUTHILL.

Relationship between the resolving power of a photographic material and the wave-length of the light. O. SANDVIK and G. SILBERSTEIN (Z. wiss. Phot., 1929, 27, 119—127).—Examination of a number of kinds of photographic plates has shown the resolving power over the range of wave-lengths 380—725 m μ to be maximal in the violet or ultra-violet and minimal at about 535 m μ for orthochromatic plates, and about 485 m μ for plates sensitised with dyes (cf. A., 1929, 402). R. CUTHILL.

PATENTS.

Manufacture of light-sensitive materials. KALE & Co. A.-G. (B.P. 294,247, 16.7.28. Ger., 21.7.27).—A combination of boric acid and an organic acid (e.g., citric, tartaric) is found to be more efficient than an organic acid alone in preventing the premature formation of colouring matter in diazo papers in which the coupling agent and the diazo compound are applied together. A suitable coating mixture is: stannic chloride double salt of 4-diazo-1-dimethylaniline 22 pts., boric acid 20 pts., tartaric acid 50 pts., chloroglucinol 2 pts., and water 1000 pts. J. W. GLASSETT.

Developers for photographic purposes. GEVAERT PHOTO-PRODUCTEN, NAAML. VENN. (B.P. 298,953, 17.10.28. Ger., 17.10.27).—Images of varying colour may be obtained by direct development of ordinary silver halide emulsions by the addition of sodium selenite or other selenium compound to the developer; sodium thiosulphate may also be added. Violet to blue tones are obtained in the presence of a restrainer (potassium bromide), whilst the addition of an accelerator (sodium carbonate) or an increase in the quantity of thiosulphate gives brown to red tones. J. W. GLASSETT.

Production of printing surfaces [on celluloid etc. from gelatin reliefs]. E. SAUER (B.P. 296,065, 23.8.28. Ger., 24.8.27).—Gelatin reliefs, after drying

by washing with amyl acetate or amyl alcohol, wiping off the liquid, and subsequently removing residual moisture with a blower or by suction, are coated with a liquid capable of softening celluloid but not gelatin, and a celluloid sheet is pressed on to the surface with rollers etc. L. A. COLES.

XXIII.—SANITATION; WATER PURIFICATION.

Activated sludge process of sewage treatment [at Bangalore]. M. SWAMINATHAN (J. Indian Inst. Sci., 1929, 12A, 131—151).—After four years' continuous operation, a plant, designed to treat the sewage from a population of 200 on a basis of 30 gals. per head per day, in 1926, was treating the sewage from 400 people, using only 18 gals. per head. No trouble has been experienced with the diffusers, except that due to occasional deposits of silt. It is stated that a rather high consumption of air (5.3 cub. ft.) per gal. of sewage treated has been found necessary to obtain a suitable circulation in the aeration tank (3 ft. deep), and to prevent the development of numerous insect larvae and worms and also "bulking" due to filamentous growths, principally *Sphaerotilus natans*. The effluents obtained throughout have been entirely satisfactory; a low dissolved oxygen content is shown to be due to the oxygen demand of the live population of the sludge. Biological observations indicate seasonal variations in the predominant types of organisms present. Sterilisation experiments indicated that these higher organisms utilised the bacteria, present as a source of food, but elimination of them did not appreciably increase the rate of nitrification. C. JEPSON.

Sewage purification by intermittent land irrigation at Lublino (Moscow), 1914—1923. K. K. BARSOV and K. N. KOROLKOV (Rep. Moscow Sewage Res. Comm., 1928, 10, 120 pp.).—A general description of the works, the results of operation, and analytical returns covering this period is given. C. JEPSON.

Comparison of the dilution and absorption methods for determination of biochemical oxygen demand [of sewage]. G. E. SYMONS and A. M. BUSWELL (Ind. Eng. Chem. [Anal.], 1929, 1, 214—215).—A modification of Sierp's procedure for the determination of the oxygen demand of sewage by the direct absorption method (B., 1928, 318) is outlined, and the results are compared with those of the dilution method. The former obviates the use of the large number of subsamples, is shorter and more convenient, and the condition of decreasing oxygen concentration is absent. The amount of agitation necessitated by the apparatus apparently causes some slight variation in readings, and it must be kept at constant temperature when the readings are taken. Anomalous results are not obtained by the two methods when buffered dilution water is used in the latter. Both methods show two-stage oxidation. E. H. SHARPLES.

Improvements in the Imhoff sewage-settling tank. O. MOHR (Eng. News-Rec., 1929, 103, 647—648).—Difficulties met with in the operation of Imhoff tanks, e.g., foaming, spitting, sludge deposition on the sides of the sedimentation compartment, and accumulations of floating scum, are eliminated by causing the

sedimentation compartments to be submerged. According to the analytical returns quoted, this form of tank permits a better separation of colloidal matter than the older type. No arrangement is indicated for gas collection.

C. JEPSON.

Purification of water by ultra-violet radiation.

J. M. BLOCHER (J. Amer. Water Works' Assoc., 1929, 21, 1361—1372).—The river is used by the town of Berea, Ohio, as the source of its drinking-water supply, the water being treated with aluminium sulphate followed by rapid sand-filtration and sterilisation by means of ultra-violet rays. The water is passed through four copper boxes in series, each of which contains a mercury-vapour lamp surrounded by a quartz cylinder. Two such sets are installed and may be used separately, in series, or in parallel. The plant is capable of dealing with 2 million gals. per day, and results so far obtained indicate that the sterilisation is effective, particularly with gas-forming micro-organisms, and that after-growths are negligible. There is no danger of over-dosage or of the production of disagreeable tastes. The current consumption is approx. 200 kw./million gals.

C. JEPSON.

Electrolytic cells in chlorination [of drinking water] for the destruction of algæ. W. T. BAILEY (J. Amer. Water Works' Assoc., 1929, 21, 1388—1394).—Chlorine has been successfully used to prevent algal growths in settlement basins etc. at Council Bluffs, Iowa, with consequent improvement in the quality of water supplied. Experiments to determine whether electrolytic chlorine was a more efficient bactericidal agent than liquid chlorine yielded negative results. The cost of production of electrolytic chlorine compared very unfavourably with the cost of using liquid chlorine.

C. JEPSON.

De-aeration of water in relation to character of water supply. J. R. McDERMET (J. Amer. Water Works' Assoc., 1929, 21, 1339—1344).—The efficiency of either de-aeration or de-activation of water with a view to corrosion prevention is largely dependent on the nature of the supply. It is generally sufficient to reduce the amount of oxygen present to 0.5 c.c. per litre, and, with waters containing lime or softened by the lime-soda process, protection is assisted by the deposition of a film of scale. Waters free from lime can be given useful but not complete protection if the p_H value is not below 6.0. Zeolite-softened waters which when heated are frequently very corrosive over a narrow zone of attack are rendered non-corrosive by de-aeration, provided the p_H value is not less than 9.5. When water is treated by the de-activation process, the dissolved and colloidal forms of iron taken up are difficult to remove unless p_H 9.3 can be maintained. To accomplish this with varying conditions and rates of flow a calcined and partially sintered magnesium carbonate has been successfully employed, although in exceptional cases additional adjustment is necessary.

C. JEPSON.

High-manganese effluents from idle [drinking-water] filters. A. C. JANZIG and I. A. MONTANK (J. Amer. Water Works' Assoc., 1929, 21, 1319—1328).—The presence of manganese in potable waters causes

stains and incrustation of pipes. The maximum permissible amount of manganese and iron together is quoted as 0.3 p.p.m. Periodic increase in the manganese content of the water at Columbia Heights was traced to the effluent from stand-by filters. Manganese previously deposited on the sand grains was redissolved under anaerobic conditions, probably by biological agency, and the effluents obtained when the filters were recommissioned after washing for 15 min. contained more than the normal amount for nearly 12 hrs. Similar filters which had been treated with copper sulphate for algal prevention were found to deliver a smaller quantity. Manganese interferes with the *o*-tolidine test for residual chlorine, but no direct relation was found.

C. JEPSON.

Errors in the Clark method for determining hardness [in water].

G. G. TOWN (J. Amer. Water Works' Assoc., 1929, 21, 1395—1404).—In this method of determining hardness, any iron or magnesium present is considered to have the same soap-consuming power as calcium, and no allowance is made for the requirements of the water itself. It is proposed that the amount of soap solution required for a volume of distilled water equivalent to that of the sample and the soap-consuming power of any iron present (determined separately) should be deducted from the amount required to produce the false end-point, the remainder being considered as the amount required by the calcium. The quantity now required to convert the false end-point into a final one is a measure of the magnesium present, and results may be quoted in terms of the carbonates of iron, calcium, and magnesium, respectively.

C. JEPSON.

Determination of hardness in water by means of soap solution. T. VON ESTREICHER (Chem.-Ztg., 1929, 53, 800).—The method described by Bruhns (B., 1929, 662) for determining separately the calcium and magnesium hardness of water was originally described by Olszewski (Abhandl. Krakauer Akad. Wiss., Math.-natur. Kl., 1881, 9, 173).

A. R. POWELL.

Dissociation of water. FELLOWS.—See I. **Low-temperature tar as disinfectant.** EHRMANN.—See II. **Determination of phenol in presence of salicylates.** HAMILTON and SMITH.—See III.

PATENTS.

Oxygen-evolving preparations and their application for air purification. DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER, W. ZISCH, and E. HERZOG (B.P. 319,393, 19.6.28).—The preparations comprise mixtures of alkali trioxides with alkali hydroxides prepared by heating at 50—120° mixtures of alkali hydroxides and alkali peroxides, obtained as described in B.P. 280,554 (B., 1928, 447) or by mixing alkali peroxides with alkali peroxide dihydrates, the reaction proceeding thus: $\text{Na}_2\text{O}_2\text{H} + \text{Na}_2\text{O}_2 = \text{Na}_2\text{O}_3 + \text{NaOH}$. Inert material, alkali hydroxides, and catalysts may be incorporated with the products.

L. A. COLES.

Insecticide (U.S.P. 1,732,240). **Insecticide and parasiticide (U.S.P. 1,731,677—8).**—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 27, 1929.



I.—GENERAL; PLANT; MACHINERY.

Drying of solids. II. P. K. SHERWOOD (Ind. Eng. Chem., 1929, 21, 976—980; cf. B., 1929, 153).—The rate of drying of a moist solid may be controlled by the rate of removal of vapour from the surface or by the rate of diffusion of liquid to the surface. Evaporation may take place from the surface or, in the later stages of drying, from the interior of the solid through a layer of solid which has already dried. A typical rate of drying curve has three periods. At first the rate of drying is constant, and is controlled by the rate of removal of vapour at the surface. If parts of the surface of the solid are covered with material impervious to liquid or vapour the rate of drying may not be greatly reduced, as heat may be received by conduction or radiation at these dry surfaces and may accelerate evaporation from the exposed surfaces. At a critical moisture content drying proceeds at a falling rate. This period may be divided into two parts. The reduction in rate of drying in the first part of this period is due to decrease in the area of wetted surface, evaporation taking place through a dry layer at certain points. A change in the slope of the curve occurs again when the rate of diffusion of liquid becomes the controlling factor. Equations are given for calculating the rate of drying in practice, based on the physical constants of the material and on atmospheric conditions.

C. J. SMITHELLS.

Absorption. I. Very soluble gases. W. V. HANKS and W. H. McADAMS (Ind. Eng. Chem., 1929, 21, 1034—1039).—Equations based on the two-film theory of Whitman (B., 1923, 913 A) have been derived to express the effects of composition and temperature on the rate of absorption of a gas by a liquid. The absorption of ammonia by water has been investigated for gases containing 6—65 mol.-% of ammonia, and is in accordance with the authors' equation. The nature of the gas used as carrier has a great influence on the numerical value of the absorption coefficient, which can be interpreted in terms of changes in film thickness (cf. A., 1925, ii, 106; B., 1925, 69).

C. W. GIBBY.

PATENTS.

Rotary-hearth furnace. E. S. FATKIN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,728,750, 17.9.29. Appl., 19.9.27).—A method of supporting tilting trays (for the goods) in an annular hearth furnace is described.

B. M. VENABLES.

Rotary [tubular] furnace. W. M. DUNCAN (U.S.P. 1,728,958, 24.9.29. Appl., 14.5.26).—A stepped combustion chamber is formed in the burner end of the furnace, the smallest diameter being adjacent to the fuel inlet.

Longitudinal air ducts are provided, which enter the furnace at the rising portions of the steps.

B. M. VENABLES.

[Grates for] furnaces. A. W. BENNIS (B.P. 320,347, 9.7.28).—Cast-iron fire-grate elements of the type which are supported on troughs conveying forced-draught air are formed with tubes of stronger metal cast in them longitudinally; several elements may be arranged in line, the tubes then being of different sizes, so that they can nest into each other. It is also arranged for air currents to pass through the tubes. B. M. VENABLES.

Drying methods and apparatus. J. G. OLSSON and F. I. E. STENFORS (B.P. 299,838, 5.6.28. Swed., 2.11.27).—The material to be dried is conveyed through a long passage and subjected to transverse streams of drying medium circulated by a series of fans. In the middle or some other part of the travel the circulating drying medium is heated by external heat, e.g., a steam coil; from this zone the drying medium plus vapour is passed through a nest of tubes above the long drying chamber, thus serving to heat other transverse circulations (which become progressively cooler as the moist medium becomes cooler). There is in consequence always an available temperature difference, and the final temperature of the moist medium is so low that practically all vapour is condensed. The medium is then reconditioned for re-use either by further cooling by a water-cooled coil or by admixture of a proportion of the fresh cool air.

B. M. VENABLES.

Cooling and degassing towers. F. K. T. VAN ITERSOM and P. M. KUYPERS (B.P. 320,505, 20.9.28).—A reinforced concrete cooling tower of flowing shape (as described in B.P. 108,863) is provided with one or more fans at the throat, a preferred form being an aeroplane propeller on a vertical shaft.

B. M. VENABLES.

Condenser. J. A. GIBB, Assr. to PETROLEUM DERIVATIVES, INC. (U.S.P. 1,727,403, 10.9.29. Appl., 7.5.27).—Both inlet and outlet vapour ports are close to a pool of condensate, which is maintained out of contact with the cooling tubes. The heat from the vapour passing through the condenser is intended to re-vaporise low-boiling constituents from the pool.

B. M. VENABLES.

Crystallisation of substances that crystallise exothermically. APPAREILS ET EVAPORATEURS KESTNER SOC. ANON. (B.P. 301,496, 13.11.29. Fr., 2.12.27).—For the direct production of dry crystals a hot saturated solution of a substance such as ammonium nitrate is subjected to strong agitation and a current of air. Two crystallising agitators may be fed alternately from one evaporator.

B. M. VENABLES.

Unit pulveriser. A. A. HOLBECK (U.S.P. 1,724,876, 13.8.29. Appl., 22.9.27).—The stationary pulverising element is attached to the removable cover plate of the machine.

B. M. VENABLES.

Mixer and conveyer. P. THOMSON (U.S.P. 1,731,953, 15.10.29. Appl., 16.8.26).—The loose materials are fed into one end of a worm-conveyer, which may have a diminishing pitch and be interrupted. The worm delivers the material to the interior of a screen, through which air is forced and from the exterior of which the material is removed pneumatically.

B. M. VENABLES.

Pneumatic sorting of materials of different densities. P. SOULARY, and COMP. DES MINES DE BRUAY (B.P. 320,207, 10.9.28).—A shaking table or series of tables is provided with upward air currents in a series of zones alternating with dead zones without air. The skimmers for delivering the products are situated in the dead zones. The earlier skimmers remove lighter material sideways, whereas the later ones remove the heavier layer downwards and permit the lighter layer to pass straight on.

B. M. VENABLES.

Screening centrifuges. C. G. HAUBOLD A.-G. (B.P. 310,512, 20.3.29. Ger., 28.4.28).—A centrifugal basket is driven by a sleeve shaft, within which is a solid shaft driven from the sleeve by step-up gearing. The solid shaft carries a worm-discharge device; the distance between the periphery of the worm thread and the basket increases progressively in the direction of discharge of solid material.

B. M. VENABLES.

Beating and/or mixing of liquids or semi-liquids. J. W. and J. MORTON (B.P. 318,851, 8.6.28).—The apparatus is suitable for whisking materials such as confectionery while under air pressure. The container is mounted on trunnions; the shaft for the beating device is brought through the walls of the container below the trunnion and is connected to the driving gear by dog clutches.

B. M. VENABLES.

Apparatus for treating liquids with gases, gases with liquids, and the like apparatus. G. K. DAVIS (B.P. 320,092, 4.7.28).—In an apparatus comprising a casing divided into compartments in each of which is a fanner for spraying the liquid which collects at the bottom of the compartments, gutters are arranged so that the spray caught on the vertical partition walls is diverted through the partition to the next compartment, thus producing a circulation of the liquid, which is controlled by varying the return openings below the liquid level.

B. M. VENABLES.

Producing mixtures of liquids and gases. E. T. CHAMBERS (B.P. 320,323, 3.7.28).—The apparatus described can be used as a petrol carburettor, and with modifications is suitable for use as a blow lamp or fuel-atomiser.

B. M. VENABLES.

Filter presses. FILTRES PHILIPPE (B.P. 318,794, 16.11.28. Fr., 10.10.28).—The plates are all to one pattern, one face being recessed and provided with feed port, the other being ribbed and provided with outlet port for filtrate; frames are unnecessary, and the cakes are bounded on one side by the face of a plate itself and on the other by filter medium.

B. M. VENABLES.

Filter. C. H. LOEW, Assr. to LOEW FILTER CO. (U.S.P. 1,726,035, 27.8.29. Appl., 13.3.24).—A number of circular plates are mounted on hollow hubs; each face of the plates has ribs and channels leading to the central passage through the hubs, and the periphery of each disc is provided with a rib, the cloth or filter medium being stretched over the faces of the disc by the action of friction rings applied each side of the peripheral rib.

B. M. VENABLES.

[Laboratory] filter. R. B. MILLARD (U.S.P. 1,727,554, 10.9.29. Appl., 2.10.28).—Liquid is siphoned from a high-level jar to the bottom of a filter jar. The latter is filled with filter medium and covered with a disc of paper through which the inlet pipe passes. The jar is fitted with a fluid-tight cap which secures the paper in place, and has an outlet for filtrate.

B. M. VENABLES.

Filtering devices. SOC. ANON. DES PROC. R. AUDUBERT (S.A.P.R.A.) (B.P. 301,507, 29.11.28. Fr., 2.12.27).—The filtering medium comprises fibrous material stranded in the form of sheets and rolled up like a short wick. The block thus formed is cemented into a box-like structure, and the filtration is effected under very slight pressure or vacuum.

B. M. VENABLES.

Tank[-outlet] filter. C. G. HAWLEY, Assr. to CENTRIFIX CORP. (U.S.P. 1,726,827, 3.9.29. Appl., 4.5.25).—A device attached to an outlet in the bottom of a tank causes the issuing fluid to whirl and deposit solid matter in a sludge compartment. Two cocks operated by one rod are provided; when the outlet cock for liquid is open the cock for discharge of sludge is closed and *vice versa*.

B. M. VENABLES.

Filtering or like devices. H. A. THOMPSON (B.P. 318,821, 17.1.29. Addn. to B.P. 307,267 and 308,166; B., 1929, 499).—A method of forming the bearings for the geared scrapers of the prior patents is described.

B. M. VENABLES.

Apparatus for straining liquids. R. A. BLAKEBOROUGH, J. LINDSAY, and J. B. BLAKEBOROUGH (B.P. 319,068, 19.6.28).—The screen comprises a pair of endless travelling chains to which perforated screening plates are fitted in such a manner that the joints overlap.

B. M. VENABLES.

Means for separating solids from liquids. J. R. POWELL, Assr. to ARMOUR & Co. (U.S.P. 1,729,547, 24.9.29. Appl., 20.9.26).—Sludge is drawn from the lowest corner of a tank having a sloping bottom and elevated by an ejector device to above the surface of a screen on which solid matter is separated, the strained liquid running back into the tank. The power liquid for the ejector may be pumped from the upper part of the same tank.

B. M. VENABLES.

Apparatus for separating liquids and solids. H. C. BEHR (U.S.P. 1,727,855, 10.9.29. Appl., 10.9.27).—A centrifugal screen is constructed after the manner of the blades of a centrifugal pump. The solid material is flung from the discharge end of the screen against a wall, from which it is removed by a series of movable blades.

B. M. VENABLES.

Separator of liquids from gases. T. F. ROCHESTER, Assr. to KORECT AIR METER CORP. (U.S.P. 1,731,061,

8.10.29. Appl., 14.12.27).—The apparatus comprises a glass cylinder (or other casing) suitably closed at the ends. The outlet for gases is through the top and is surrounded by a depending flange. The inlet for fluid is through the bottom, up an internal pipe leading to near the top outside the depending flange but inside the casing. The outlet passage is smaller than the inlet.

B. M. VENABLES.

II.—FUEL; GAS; TAR; MINERAL OILS.

Briquetting of coke and semi-coke dust, using coal as a binder. W. SWIENTOSLAWSKI, B. ROGA, and M. CHORAŻY (Przemysł Chem., 1929, 13, 465—473).—The binding qualities of coal dust used for briquettes made of coke or semi-coke dust depend on its coking qualities; non-coking coal is unsuitable for the purpose. The source of the coke or semi-coke dust used is of lesser importance, although better results are obtained with semi-coke derived from coking coal. Mixtures containing less than 30% of coal dust do not yield briquettes of satisfactory mechanical strength. The optimum pressure for the manufacture of briquettes is 200—400 kg./cm.², whilst the optimum temperature varies from 380° to 440°, according to the variety of coal used.

R. TRUSZKOWSKI.

Low-temperature distillation [of coal] in the "Italian system" furnace. D. MENEGHINI (Giorn. Chim. Ind. Appl., 1929, 11, 391—399).—This furnace, installed at the communal gasworks at Genoa, is a modification by Scavia of the Piron furnace, the molten lead bath being omitted. It comprises two superposed channels each 800 mm. wide and 10 m. long, the lower being 450 mm. and the upper 900 mm. high. The metallic conveyer consists of cast-iron plates articulated in chain fashion so as to form an endless band moved by toothed wheels at the extremities of the channels. The coal, ground to pass a 10-mm. sieve, is fed to give a layer about 15 mm. deep, and is distilled at 550—650°. Both Thornley gas coal and lignite were distilled, the heating being effected by means of the ordinary gas made at the works. One metric ton of the gas coal yielded 99.2 cub. m. of gas (calorific value 6700 kg.-cal./cub. m.), 667 kg. of semi-coke (6890 kg.-cal.), and 124.8 kg. of tar (8970 kg.-cal.), the gas consumed for heating being 230 cub. m. (4798 kg.-cal.). Of the calorific value of the coal consumed, 8.67% is obtained in the gas, 59.92% in the semi-coke, and 14.55% in the tar; total 83.14%. One metric ton of dry Valdarno lignite gave 144 cub. m. of gas (2390 kg.-cal./cub. m.), 645 kg. of semi-coke (3640 kg.-cal.), and 43 kg. of tar (8600 kg.-cal.), the heating gas consumed being 294 cub. m. (4752 kg.-cal.); 9.69% of the heating value of this lignite appears as gas, 66.13% as semi-coke, and 10.38% as tar.

T. H. POPE.

Gaseous explosions. VII. Effect of lead tetraethyl on rate of rise of pressure. M. S. CARR and G. G. BROWN (Ind. Eng. Chem., 1929, 21, 1071—1078; cf. A., 1928, 1331).—The influence of lead tetraethyl on the rate of rise of pressure has been investigated for both slow-burning and fast-burning mixtures containing oxygen, nitrogen, and the following fuels: *n*-heptane, *n*-octane, isooctane, benzene, toluene, methyl alcohol,

ethyl alcohol, and ethyl ether. The effect of lead tetraethyl is independent of the chemical nature of the fuel, but depends largely on the type and rate of combustion and on the normal rate of rise of pressure in the untreated fuel. In mixtures which give a maximum rate of rise of pressure below a critical rate, small additions of lead tetraethyl decrease the maximum rate of rise, but larger additions increase it. Above the critical rate of pressure rise, 0.1—1.0% by vol. of lead tetraethyl increases the maximum rate. The mechanism of the action of lead tetraethyl is discussed, and it is suggested that the decomposition products of lead tetraethyl are the active agents in retarding the rate of inflammation and rise of pressure, and that the accelerating action in fast-burning mixtures may be due to the explosive action of decomposition occurring in the flame front, the rate of inflammation being considered as greater than the rate of decomposition of the lead tetraethyl.

C. W. GIBBY.

Explosive limits of industrial gases. J. YEAW (Ind. Eng. Chem., 1929, 21, 1030—1033).—The upper and lower explosive limits of mixtures of air with a number of gaseous mixtures encountered in American city-gas manufacture have been determined. A small quantity of illuminating constituents reduces the upper limit from about 70% of gas to about 30%, but affects the lower limit to a much less extent. The upper limit is unaffected by large changes in the ratio of hydrogen to carbon monoxide, as their individual upper limits are close together, but is lowered by increasing the methane content. The limits can be calculated with considerable accuracy by the formula of Le Chatelier (Ann. Min., 1891, 19, 388).

C. W. GIBBY.

Ignition of firedamp. H. F. COWARD and R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 53, 1929, 40 pp.).—The results of earlier work by these authors (B., 1926, 179) on this subject are reviewed in the light of new data and old interpretations are confirmed or revised. Reference is also made to the results of subsequent work by Burgess and Wheeler (B., 1926, 114), Coward and co-workers (B., 1926, 426; 1929, 661; and A., 1928, 24), and by Bone and co-workers (A., 1928, 248).

C. B. MARSON.

Dissolved acetylene. W. RIMARSKI (Chem.-Ztg., 1929, 53, 725—727, 746—748).—The volume of a cylinder of dissolved acetylene is occupied approximately as follows: porous mass 25%, acetone 40%, increase in volume of acetone due to absorption of acetylene 24%, allowance for expansion on heating to 65° 4.3%, safety space 6.2%. The porous mass should have a porosity of 70—80%, should not pack when the cylinder is dropped 100,000 times from a height of 11 mm. on to a steel plate, and should be only locally decomposed when a welding burner, fed by the gas from the cylinder, is played on to the wall of the full cylinder for 15—30 min. Apparatus for testing the internal and external ignition of the gas and its tendency to explosive decomposition is illustrated and briefly discussed.

A. R. POWELL.

Coking of pitch. J. P. KOETNITZ (Brennstoff-Chem., 1929, 10, 406—407).—The manufacture of electrode carbon by the distillation of coal pitch is briefly

discussed. The chief difficulty lies in the construction of a still capable of withstanding the high temperature, above 1000°, necessary in the final stage of the process, and has only been overcome by the recent progress in the manufacture of heat-resisting steels and refractories. The process is intermittent, the complete operation with a retort holding a 2750-kg. charge of pitch taking 30 hrs. The products are 58% of coke, 36% of distillate, and 6% of gas and loss. The distillate, mixed with other tar oils, is utilised as cheap lubricating oil, heating oil, or impregnating oil. On admixture with other oils a solid is precipitated from which anthracene can be recovered.

A. B. MANNING.

[Purification from phenol of] effluents from coke ovens. M. PRÜSS (Gas- u. Wasserfach, 1929, 72, 791—801).—The river Emscher is estimated to receive 10,000 tons per annum of phenols in the form of coke-oven effluents. The work of Fowler on the bacteriological purification of such effluent has been repeated by the river authority, and an artificially aerated culture prepared which requires only 2 cub. m. of filter material per cub. m. of effluent. The effluent must be diluted in the ratio 1:4, and a time of contact of 2 hrs. provided. This Emscher filter is at work at one colliery and is described. The temperature and p_H values require careful control, and 60 cub. m. of air/cub. m. of effluent are required. It is, however, too expensive for general use. The use of the effluent for coke quenching is impracticable owing to the nuisance caused by vapours. Under these circumstances four phenol recovery plants were erected in the Emscher basin and commenced work at the end of 1926. These operated the "Pott-Hilgenstock" benzol process and were partly financed by the river authority. As the benzol takes up only 1% of phenols distillation of the benzol is too expensive and the phenols are removed by alkali washing. Costs are discussed, and it is claimed that at the present market price for carbolic acid a large coke-oven plant could work this process at a small profit. Some indirect advantages are also obtained; e.g., the effluent water can be used for works' purposes. The American Koppers process is described, but it is not believed that it will be adopted in Germany. A combination of partial extraction and bacteriological treatment may be desirable. For the effluent from lignite distillation benzol treatment is inapplicable, but experiments are proceeding using a heavy oil of b.p. 320—350°. This effluent is even more objectionable and difficult to deal with than that from coke ovens. In the discussion on this paper the use of activated carbon for phenol recovery in place of benzol was advocated.

C. IRWIN.

Crude oil produced in the Salt Creek Field, Wyoming. H. P. RUE and I. N. BEALL (U.S. Bur. Mines Tech. Paper No. 449, 1929, 27 pp.).—An apparatus for distilling 10-gal. charges of crude oil is described, and both steam and coking distillations were made in an effort to prepare distillates comparable with those obtained in refinery practice. Firstly, the crude oil was distilled with sufficient steam to prevent cracking, and the resulting distillates were fractionated and further treated. Secondly, the crude oil was distilled to coke without steam, and, after removal of the coke from the

still, the entire distillate was recharged and distilled with steam as in the first process. The final yields in the two processes were, respectively: gasoline 33.0, 41.0%; kerosene 5.0, 2.3%; gas oil 18.0, 26.4%; non-viscous lubricating oil 8.3, 11.5%; viscous lubricating oil 4.7, 2.5%; gear oil 3.8%, nil; foots oil 7.0, 5.5%; wax 15.8, 4.1%; asphalt 1.1%, nil; total coke and losses 3.3, 6.7%.

H. S. GARLICK.

Evaluation and cracking of gas oils. R. H. GRIFFITH (J.S.C.I., 1929, 48, 252—263 r).—The mechanism of gasification of gas oils has been studied in a furnace containing a silica tube heated electrically, the permanent gas produced being burned in a Boys calorimeter and all results expressed on a thermal basis. It is found that the rate at which oil is passed through the cracking zone influences the results, there being an optimum speed on either side of which loss of efficiency occurs. The changes do not, however, take place entirely on the hot surface, but the proportion of gas reaction varies with the surface:volume ratio of the apparatus. The decrease of efficiency at very low rates of oil feed is not due to further decomposition of gaseous hydrocarbons, but to other processes involving absorption of large volumes of hydrogen. When the experiments are carried out in an atmosphere of nitrogen, instead of with the addition of hydrogen, much lower figures are obtained. The temperature of cracking is also important; the best results are obtained at about 750°, lower temperatures giving higher tar yields and higher temperatures leading to formation of free carbon. Comparative tests have been performed under standard conditions on 31 oils from a very wide range of sources, and at the same time these oils have been analysed with respect to their content of unsaturated, aromatic, naphthene, and paraffin hydrocarbons (cf. B., 1929, 841). Results varying from 1.45 to 0.50 therms per gallon were obtained, and it is found that a gas oil can be valued by means of chemical analysis, as the straight-chain hydrocarbons in it are of far greater importance than those having a cyclic structure. Further preliminary work on the influence of b.p. of the oil, and on the nature of the tars produced, is also described. The conclusions drawn from these small-scale experiments have been examined on two water-gas plants during normal operation; the methods employed in obtaining the necessary measurements and in calculating the results are detailed, and it has been found that the same factors are involved in producing good or bad results; the number of gallons of oil injected into the carburettor and the temperature at which the cracking vessels are operated are of primary importance. Measurements have been made of the surfaces and volumes involved, and the figures obtained show that the same type of reaction is involved as that observed in laboratory experiments. The amounts of methane and gaseous olefins produced, and of hydrogen absorbed, per gallon of oil are shown for various operating conditions on the two plants.

Thermal treatment of natural gas. D. S. CHAMBERLIN and E. B. BLOOM (Ind. Eng. Chem., 1929, 21, 945—949).—Natural gas was thermally treated in tubes of silica, steel, copper, iron, nickel, monel, and clay. Between 500° and 900° the various tubes had

different effects in regard to converting methane into benzene, naphthalene, anthracene, acetylene, etc. Fused silica, clay, copper, and monel metal do not catalyse methane and ethane to any great extent. Copper gives the highest yields at the lowest temperatures, but the metal is consumed after a very short time in use. Silica gives high yields at intermediate temperatures, and is the most important material studied, as it is not changed by heat or by products of the thermal treatment. Iron and nickel readily decompose methane into its elements, but these metals are rapidly disintegrated by the reaction. The effects of temperature, surface, dilution, and other factors were studied, and various units are discussed ranging from apparatus of laboratory size to a semi-commercial installation. Experiments show it is the carbon, formed as a result of the decomposition of natural gas at 450° and above, which has a selective activity in the conversion of paraffins into aromatic compounds etc. This activity is easily destroyed by partial oxidation or by the formation of carbon-metal compounds, graphitic carbon being the final result. By a comparison of the results of these thermal treatments with that of gas manufacture in the by-product coke oven, assuming that the gases have reached an equilibrium, the practical yield of benzene has been obtained from natural gas amounting to 27.0–40.0 litres per 1000 m.³ of gas treated. The optimum temperature of benzene formation varies for each material and the method of carbon formation thereon. When the concentration of hydrogen has attained 50–60% of the volume of the gas being treated the oil yield becomes negligible.

H. S. GARLICK.

Analyses of some natural gasoline gases before and after treatment. H. C. ALLEN (Ind. Eng. Chem. [Anal.], 1929, 1, 226–227).—The results are given of the analysis of three natural gasoline gases before and after treatment to remove gasoline. A comparison is made between the results obtained by combustion analysis and the method chosen, viz., fractionation by means of liquid air, and analysis of the fractions by combustion methods, the results, in general, differing considerably.

H. S. GARLICK.

Relation between physical characteristics and lubricating values of petroleum oils. E. D. RIES (Ind. Eng. Chem. [Anal.], 1929, 1, 187–191).—The tests for physical characteristics are discussed in the light of their relation to lubrication. Neither gravity nor colour bears any relation to lubricating value. The A.S.T.M. cold test shows a rough parallelism to the actual point at which flow ceases, but the numerical values mean little. With Pennsylvania oils, high carbon residue as found by the Conradson test is due not to high wax content but to these oils being less volatile than those in the same viscosity range made from other crudes. In general, the removal of wax increases the Conradson carbon number of the dewaxed oil; the wax itself forms no carbon in the test. Flash and fire tests indicate nothing as regards lubricating power and give inaccurate data on evaporation. Only a small amount of a relatively volatile component will materially lower the fire and flash values without causing undue percentage loss by evaporation. A vacuum-distillation

test giving a volatility curve similar to the Engler curve for gasoline is of greater value. The temperature-viscosity coefficient does not entirely depend on the nature of the crude oil, but may be modified by the method of refining, and is lowest of all in vacuum-distilled paraffin oils. As yet no completely satisfactory test for oiliness or for resistance to oxidation has been devised, and the need for certain new tests is advocated.

H. S. GARLICK.

Reaction between lubricating oils and phosphorus pentoxide. C. C. FURNAS (Ind. Eng. Chem. [Anal.], 1929, 1, 185).—The use of yellow phosphorus to eliminate oxygen from a closed circuit in which nitrogen was circulated at room temperature by means of a rotary blower led to the pump heating up and sticking owing to the formation of a thick gum on all friction surfaces. Tests showed this residue to contain considerable phosphorus and to be formed by phosphorus pentoxide dust being carried over by the pump and reacting at 30–35° with the lubricant. If water were added to the oil the gum dissolved and did not form again even at 100°, provided sufficient water was present to form a separate phase.

H. S. GARLICK.

Variation of viscosity of [lubricating] oils with temperature. E. W. DEAN and G. H. B. DAVIS (Chem. Met. Eng., 1929, 36, 618–619).—In a scheme to formulate the viscosity-temperature coefficients of oils in simple manner it was found that the general relationship between viscosities (Saybolt) at the standard temperatures of 100° F. and 210° F. could be expressed by $Y = a + bx + cx^2$, in which Y and x are the viscosities at 100° F. and 210° F., respectively. The constants are characteristic of the series of oils. A system of so-called "viscosity indexes" indicates the viscosity-temperature coefficients of oils on a scale in which oils having a large change of viscosity are taken as zero and oils with only small changes in viscosity as 100.

C. A. KING.

Oxidising lubricating oil bottoms. A. D. DEMCHENKO and C. N. OBYRADCHIKOV (Nef. Choz., 1928, 15, 360–366).—The material was blown with air at 235–252°, and the composition of the gases, distillate, and residue was determined as the oxidation proceeded.

CHEMICAL ABSTRACTS.

Setting point of some mineral oil mixtures. J. TERPUGOFF (Petroleum, 1929, 25, 1213–1214).—Deviations between the observed and calculated setting points of mineral oil mixtures may be positive or negative according to the nature of the constituent oils. Positive deviations occur with certain blends of Russian, Texas, and Pennsylvanian oils, and negative with certain Russian, Texas, and mid-continent oils. In some cases the setting point of the mixture is lower than those of the initial oils. Since mixtures of oils of the same origin give differences between observed and calculated setting points which lie within the limits of experimental error, it follows that mixtures of oils of various origins undergo a change in their properties. With positive deviations some oils are rendered more viscous, although there is no change in viscosity at 20°, 50°, and 100°; with negative deviations some oils are rendered more liquid. The greatest deviations occur

with Texas oils, which become more liquid with rise of temperature. Such oils, the viscosities and setting points of which are decreased in mixtures, change their liquid state very quickly as the temperature rises. It is assumed that at, *e.g.*, 20° some hydrocarbons of those oils are only present in the colloidal state, and with rise of temperature, or on mixing with oils of other origins, these hydrocarbons pass into ordinary solutions.

W. S. E. CLARKE.

Alcohol fuels for use in internal-combustion engines. J. G. KING and A. B. MANNING (*J. Inst. Petroleum Tech.*, 1929, **15**, 350–368).—Experiments were conducted to determine the limiting factors in the preparation of mixed alcohol fuels and their behaviour in an ordinary standard petrol engine. Absolute or 98.6% alcohol is miscible in all proportions with petrol, but on the addition of water to the mixture petrol separates until at a certain concentration, depending on temperature, none remains in solution. Marked separation is caused by 1.8% of water, and in a 1:1 solution of petrol and absolute alcohol the amount of the separated layer at 0° may be 36% of the volume of the petrol present. The miscibility limits and limiting percentages in stable mixtures with 95% alcohol of seven petroleum spirits are given over the temperature range +15° to –10°, and vary over a fairly wide range. The ranges of miscibility at 15° of 95% alcohol and of a Persian petrol extend from 0 to 1.1% and 63.5 to 100% of alcohol by vol., the lower range being so limited as to be negligible. B.S.R. benzine and Persian spirit differ from others in having higher solubilities but steeper solubility curves, so that the solubilities are less at –15° than at 0°. Absolute alcohol and benzene are miscible at all temperatures above the m.p. of the latter. The alcohol-benzene mixtures which have initial crystallising points at 0° and –10° contain, respectively, 25.1 and 59.0% of alcohol. In the examination of ternary mixtures of alcohol, benzene, and petrol, the limiting amount of petrol in 100 vols. of mixture containing 50 vols. of alcohol was 39.5 vols. at 15° and 31 vols. at –5°. With 2% of water present these values were considerably lowered. Mixtures containing 50% of benzene were stable at –5° irrespective of the proportions of the other constituents as regards separation of solid benzene. The water separation limits for 92% and 95% alcohol are given. With mixtures containing 50% of petrol no clear solution was obtained at 0°, the critical point being about 3°. With 72% alcohol no clear solution was obtainable at 15°. The limits of stability for varying amounts of petrol were determined. Experiments in which pure benzene was replaced by commercial 90's benzol and the alcohol by power methylated spirit each showed a reduction in miscibility range, showing that the limits of safety for a mixed motor fuel would be narrowed by the use of less pure alcohols and benzol. The limits of miscibility of power methylated spirit and 90's benzol in different petroleum spirits are tabulated. The above experiments were repeated using motor spirits recovered from the low-temperature carbonisation of bituminous coal with results of the same character. Phase diagrams for petrol-benzol-alcohol mixtures are given and serve to

show up the difference in aromatic content of the petrols. From a series of engine tests on mixed fuels of the above type, one containing the maximum quantity of petrol safe at –20°, a second the maximum quantity of alcohol, and a third petrol containing the maximum amount of alcohol in solution, the following conclusions were arrived at: (1) Both petrol-alcohol-benzene mixtures are suitable for use in petrol engines without any alteration beyond enlarging the carburettor jets. (2) The petrol-alcohol mixture cannot be used satisfactorily. (3) Detonation was not observed with any of the fuels. (4) The volumetric fuel consumption increases with the volume of alcohol in the fuel. (5) The thermal efficiency obtainable is similar to that of petrol. (6) The difference between mixture strengths giving maximum economy and the theoretical mixture strengths is greater with these fuels than with petrol. (7) The mixture strength required to obtain maximum economy is nearer the limiting strength for smooth running for these fuels than with petrol.

H. S. GARLICK.

PATENTS.

Recovery of condensates from coal-distillation gases. BARRETT Co., Assees. of S. P. MILLER (B.P. 294,106, 6.7.28. U.S., 16.7.27).—The hot gases from coke ovens or gas retorts are cooled to a limited extent, *e.g.*, by spraying ammonia liquor into the goose-necks and collector main, and are then cleaned from suspended pitch particles by means of an electrical precipitator. By suitable regulation of the temperature at which the gases are cleaned, pitch of low carbon content and of any desired m.p. may be collected in the precipitator. The cleaned gases are cooled to a sufficiently low temperature to condense from them an oil suitable for use as creosote oil; or the cooling may be carried out in two stages giving, respectively, a creosote oil and a lighter tar acid oil. The gases are further treated in known manner to recover ammonia and benzol therefrom.

A. B. MANNING.

Settings for vertical retorts used in the production of gas etc. DRAKES, LTD., and J. W. DRAKE (B.P. 320,448, 4.8.28).—Vertical retort settings in which the waste combustion gases are withdrawn from the lower end of the setting have the uptake flues leading from the producer so arranged that a portion of the combustible gases can be admitted to the uppermost combustion chamber while the remainder is led to a lower combustion chamber, situated preferably about one third the height of the retort from the top. Secondary air is admitted to the combustion chambers in amounts necessary to give the desired heating effects.

A. B. MANNING.

Apparatus for production of producer gas. L. U. DE TOYTOR (B.P. 320,441, 28.7.28).—Air and steam are introduced into the centre of combustion of the fuel through a conduit projecting axially downwards into the producer. The air passes through an injector device arranged to draw in a quantity of steam proportional to the amount of air passing. The steam is supplied from a water reservoir which communicates with a boiler surrounding the nozzle through which the air and steam enter the producer.

A. B. MANNING

Hurdles for washing gas etc. W. SOMMER (B.P. 320,575, 14.1.29).—Thin wooden boards are arranged parallel to one another and spaced by slats running crosswise between the boards. Two connecting rods pass through each set of spacing slats and through the boards. Wedges passing through slots in the ends of the connecting rods serve to clamp the boards and slats together. [Stat. ref.] A. B. MANNING.

Treatment of crude oils, tars, bituminous residues, etc. L. BOLGAR (B.P. 319,673 and 319,718, 23.3.28).—(A) Crude oil, tar, or pitch, etc. is heated with an oxidising agent, *e.g.*, sulphuric acid, acid sludge, at 120–200°. The precipitate which forms is separated from the liquid and further heated at a higher temperature, preferably with the addition of a fluxing agent such as anthracene oil, until the acids are decomposed. The product forms an artificial asphalt, the physical characteristics of which may be suitably modified by the addition of tar, pitch, etc., or of sand or other filling materials. (B) The starting material is mixed with the acid and heated at a temperature above 180°, stearine pitch being added before or/and during the acid treatment. The mixture is then further heated with the addition of filling materials such as sand, stone meal, asbestos. The product is pressed while hot. It is particularly suitable for the manufacture of insulating materials, roof coverings, etc. [Stat. ref. to (A).] A. B. MANNING.

Manufacture of hydrocarbons and the like from coal, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 296,431, 8.8.28. Ger., 1.9.27).—When high-boiling oils are hydrogenated at 1000 atm. and at high temperature products of intermediate b.p. are obtained. At 200 atm. a larger proportion of light products is obtained, whilst at 20 atm. gas, light products, and carbonaceous residue without products of intermediate b.p. are obtained. Hydrogenation of suspensions of coal, tar, etc. in stages is therefore advocated. The initial material is first hydrogenated in the liquid phase at 200–1000 atm. The intermediate oils produced are then treated at 20–50 atm. in the gaseous phase whereby benzene is formed exothermically; catalysts may be used. Thus an American crude oil (40–50% boiling above 350°) is treated with hydrogen at 200 atm. and 450° in the presence of a chromium-molybdenum catalyst. Without any separation the product is expanded into a second chamber maintained at 20 atm. and 480°. The mixture of gas and vapour obtained from the second chamber is cooled under pressure, and deposits a liquid consisting of benzene of high anti-knock value and about 25% of lamp oil (b.p. above 200°). Only a small proportion boils above 235°, and there are no losses due to formation of gas and coke. Very heavy asphaltic oils are best treated in three stages at pressures of 1000, 200, and 20 atm., respectively.

T. A. SMITH.

Distillation, cracking, and hydrogenation of oils, tars, etc. KOHLENVEREDLUNG A.-G. (B.P. 293,430, 6.7.28. Ger., 6.7.27).—The raw material is atomised by being fed on to a series of rapidly rotating discs, and is simultaneously heated by circulating hot gases and vapours through the apparatus. The issuing gases and vapours are passed through a superheater and recircu-

lated through the atomiser, part being withdrawn periodically and passed to a condensing system. The process may be carried out in the presence of a hydrogenating gas, and, if necessary, under pressure.

A. B. MANNING.

Production of olefines and other hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,211, 14.9.28. Addn. to B.P. 301,775; B., 1929, 120).—Granular porous substances, *e.g.*, pumice, slag, are impregnated with tars, mineral oils, distillation residues, etc., and are then passed through a chamber heated to a high temperature, as described in the main patent. The granular material is re-impregnated and used again. When necessary, the coke deposited thereon is gasified in a suitable producer.

A. B. MANNING.

Production of artificial rock asphalt. J. KLEIN (B.P. 320,357, 9.7.28).—Asphalt, pitch, oily distillation residues, etc. are emulsified with an aqueous solution of naphthenic acid or one of its salts, and a mineral filling substance, *e.g.*, lime flour, is then added. The greater part of the excess emulsifying agent is removed in soluble form, and the remainder rendered harmless by precipitation as an insoluble metallic salt.

A. B. MANNING.

Production of gasoline hydrocarbons. W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,730,152, 1.10.29. Appl., 3.2.23).—A gas containing gasoline hydrocarbons (*e.g.*, natural gas) is stripped of its gasoline content by passing up a tower countercurrent to a suitable absorption liquid (*i.e.*, a liquid which is non-volatile under the existing conditions and miscible with the hydrocarbons). The gas is introduced at an intermediate point of a contact zone for gas and liquid, the absorption liquid entering above the gas inlet and, at least in part, adjacent the top of the contact zone. This zone is heated near the bottom by means of a coil through which passes the hot, stripped absorption oil, and is cooled at the top by means of cooling coils.

W. S. E. CLARKE.

Treatment [cracking] of hydrocarbons oils. C. P. DUBES, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,729,307—8, 24.9.29. Appl., [A] 26.2.20, [B] 24.9.23. Renewed [B] 3.8.28).—(A) Oil is heated to above 427° at a pressure high enough to prevent destructive distillation. It is then flashed, the flash chamber being at atmospheric pressure; almost complete separation into vapour and coke is thereby obtained. Vapours are fractionated and all insufficiently converted material is fed continuously to the charge stream. (B) The residual oil from the reaction chamber of a cracking process is removed and introduced into a coking still. The pressure on the still is released and the oil coked by superheated steam.

W. S. E. CLARKE.

Distillation of hydrocarbon oils. R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 1,731,479, 15.10.29. Appl., 15.1.25).—The fractionating column of a still contains a number of superposed closed cooling conduits, which are provided with valves and are connected in parallel with a pair of manifolds. Condensate accumulating in a collecting pan half-way down the cooling conduits is passed outside the column. F. G. CLARKE.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Strength of cellulose [pulp]. K. RIETH (Papier-Fabr., 1929, 27, 693—694).—Variations in the results of strength measurements on pulp, carried out by the method laid down by the German Strength Commission, may amount to 25%, and are attributed to differences in the moisture content of the paper strips tested. Since strength depends on the moisture content, and the latter depends on the relative humidity (R.H.) of the atmosphere in which the strips are conditioned, it would appear that all the tests were not carried out on strips fully conditioned at 65% R.H. In order to overcome this difficulty it is suggested that tearing-length results should be referred to the moisture content of the paper and not to any specific R.H. Methods of determining R.H. are discussed. Further errors arise in the determination of the degree of beating of the pulp. They may be due to the quantity of pulp taken for examination, the use of different sieves, non-uniformity of the apparatus used, and to the time taken for the operation. New sieves often give results which differ from those given by old sieves of the same number. The effect of different beating machines on the degree of beating, time required to reach a certain degree of fineness, and the relative merits of ball mills and hollander beaters are discussed.

B. P. RIDGE.

Rate of decomposition of viscose solutions. O. FAUST (Ber., 1929, 62, [B], 2567—2573).—The viscose solutions containing 5% of cellulose and 5% of sodium hydroxide are very greatly diluted with water containing phenolphthalein and brought into a flask fitted with a small dropping funnel. The acid is introduced through the funnel as rapidly and uniformly as possible, followed by an excess of 0.1N-iodine. The residual iodine is determined by titration. The interval between decolorisation of the phenolphthalein and addition of iodine is the measured time of decomposition. The acids used are acetic, phosphoric, oxalic, hydrochloric, sulphuric, and sulphuric treated with 5% of sodium naphthalenesulphonate previously condensed with 0.5 mol. of formaldehyde. An appreciable difference is not observed between the rates of decomposition of viscose from ripened or unripened alkali-cellulose. The greatest rate of decomposition is observed at the commencement of the addition of acid. In 3 sec. the concentration of viscose with 8.54% maximal xanthate content diminishes by 1.8—2.0% with acetic and about 2.4% with sulphuric acid. The further course of decomposition is rather more rapid with stronger than with weaker acids, but the difference gradually disappears. The rate of diffusion does not enter into the question since the solutions are vigorously shaken during the addition, and are so dilute that a membrane is not produced. It is shown to be possible to esterify more than one hydroxyl group of the $C_6H_{10}O_5$ unit of cellulose by carbon disulphide.

H. WREN.

Purified wood fibres as a papermaking material. R. H. RASCH (Bur. Stand. J. Res., 1929, 3, 469—506).—The chemical purity, colour, durability, and permanence of purified wood fibres and of papers made from them have been compared with the corresponding properties of papers made from rags and other fibres. Artificial

ageing tests (heating at 100° in dry and moist air for 72 hrs.) show that the relative permanence may be measured by determining the decrease in the α -cellulose content. Like purified rag fibres, purified wood fibres undergo little change in this test, and thus should show similar good resistance to the yellowing effects of natural ageing. A high degree of hydration is detrimental to the permanence of a paper, but inert mineral fillers tend to increase the resistance to ageing. Addition of glue and starch sizes to the pulp retards chemical deterioration. These results indicate that papers made of purified wood fibres are suitable for permanent records.

A. R. POWELL.

Hydration, paper formation, and strength. G. PORRVIK (Svensk Pappers-Tidn., 1929, 32, 191—196; Chem. Zentr., 1929, i, 2935).—The degree of hydration of bleached sulphite-cellulose is determined by the external capillary water, the internal capillary water, and the fibre-wall water. The union of the fibres in the production of paper is essentially a colloid-chemical adhesion phenomenon, and not a felting process. The effect of milling is discussed.

A. A. ELDRIDGE.

PATENTS.

Production of fibre articles. M. M. FROST (U.S.P. 1,726,818, 3.9.29. Appl., 15.7.27).—The fibre is pulped in water, an adhesive, e.g., rosin soap, is added, the water expressed, and the fibre after being moulded under high pressure is dried and impregnated with molten sulphur in a closed vessel which is first exhausted and then subjected to pressure.

F. R. ENNOS.

Manufacture of tracing cloth. POTTER, BOARDMAN & Co., LTD., and P. HAMILTON (B.P. 320,071, 3.7.28).—Ordinary oil-starch or wax-starch tracing cloth is passed through a bath of oil, such as olive oil; containing an oil-soluble blue dye, e.g., chrysoidine-blue, and after expressing the superfluous oil is treated with a volatile solvent containing shellac; the solvent is allowed to evaporate and the cloth polished by rapidly rotating rollers.

F. R. ENNOS.

Copper oxide-ammonia cellulose solution for spinning artificial silk by the stretch-spinning process. A. HARTMANN, Assr. to AMER. BEMBERG CORP. (U.S.P. 1,728,565, 17.9.29. Appl., 5.2.25. Ger., 24.6.24).—To reduce the spinning temperature requisite for obtaining the best product, cellulose is dissolved in a mixture of pure copper hydroxide and ammonia with subsequent addition of sodium sulphate.

F. R. ENNOS.

Manufacture of copper oxide-ammonia cellulose solutions for production of artificial silk. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 320,069, 28.6.28).—The cellulose used for dissolving in the cuprammonium solution is the commercial variety, consisting of thick sheet or board which has been neither chemically pretreated to modify its solubility nor treated with water to cause it to swell.

F. R. ENNOS.

Carbonising the organic constituents of sulphite-cellulose lye. C. G. SCHWALBE (U.S.P. 1,731,354, 15.10.29. Appl., 4.4.27. Ger., 13.3.26).—Less than 3.6 kg. of sulphuric acid are added to 100 litres of the spent lye, and the mixture is heated at about 180° and 10 atm. for 8 hrs.

H. ROYAL-DAWSON.

Refining of pulp. Processing of cellulose fibre. BROWN Co., Assees. of (A) M. O. SCHUR, (B) M. O. SCHUR and R. H. RASCH (U.S.P. 1,730,386—7, 8.10.29. Appl., [A] 5.8.27, [B] 10.8.27).—(A) A lime-digested pulp is deresinified before it is bleached. (B) The fibres are freed from resinous matters, by means of an organic solvent, after encrusting material has been removed.

F. G. CLARKE.

Treatment of fibrous material, for example, for the manufacture of paper. S. D. WELLS (B.P. 297,319, 6.9.28. U.S., 17.9.27).—Vegetable material, after treatment with chemicals for sufficient time to soften but not to pulp it, is mixed with water in the proportion of 5—9.5 pts. of fibre to 100 pts. of water, and pounded in a rod mill.

F. R. ENNOS.

Production of waterproof paper. A. L. CLAPP, Assr. to BENNETT, INC. (U.S.P. 1,727,003, 3.9.29. Appl., 23.2.26).—After mixing the pulp with an aqueous soap dispersion of an unsaponified waterproofing material, e.g., paraffin, together with sodium silicate, sufficient aluminium sulphate is added to precipitate the soap and silicate, whereby substantially all the dispersed material is carried down and fixed in the pulp; the latter is then run out on a paper machine, dried, and heated to fuse the waterproofing material in the paper.

F. R. ENNOS.

Stencil sheet. S. HORN (U.S.P. 1,729,072, 24.9.29. Appl., 31.12.27. Jap., 8.12.27).—The sheet consists of a base of fibrous material and a coating which includes the esters of polysaccharides and quince oil.

H. ROYAL-DAWSON.

De-inking solution [for paper]. W. LEWIS (U.S.P. 1,727,722, 10.9.29. Appl., 27.2.28. Can., 12.12.27).—Printed paper stock is saturated with an aqueous solution containing washing powder, lye, and water-glass, and the resulting pulp is subjected to pressure.

H. ROYAL-DAWSON.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of arsenic in sulphuric acid. H. A. J. PIETERS and M. J. MANNENS (Chem. Weekblad, 1929, 26, 559—560).—Hydrazine sulphate is recommended for reduction to the tervalent state, the titration being effected with potassium bromate in presence of potassium bromide in acid solution. If hydrogen iodide is used for reduction (cf. Blattner and Brasseur, B., 1904, 338), the insoluble arsenic iodide is best collected on a porous alundum cone; this method also gives very good results.

S. I. LEVY.

Analysis of lime. C. A. DOLGOV (Nauch. Zapiski, 1929, 8, 22—38).—The free calcium hydroxide may be determined by adding 400—450 c.c. of boiling water and 50 g. of sugar to 5 g. of lime, shaking, diluting to 500 c.c., filtering, and titrating the filtrate (the first 150—200 c.c. being rejected) with hydrochloric acid, using phenolphthalein as indicator.

CHEMICAL ABSTRACTS.

Preparation of basic magnesia. B. G. PANTELEIMONOV (J. Appl. Chem., Russia, 1929, 2, 199—213).—The method of preparation is detailed. Washing with

hard water introduces calcium. Magnesia is slightly hygroscopic.

CHEMICAL ABSTRACTS.

Production of pure beryllium oxide from beryllium ores. H. A. SLOMAN (J.S.C.I., 1929, 48, 309—316 r).—A new method for the extraction of pure beryllium oxide from beryllium ores, chiefly beryl, is described (B.P. 312,007; B., 1929, 597). In this the beryl is decomposed with sodium silicofluoride to beryllium fluoride and converted into sulphate with sulphuric acid. Use is made of the fact that beryllium oxide is a very weak base which is readily dissolved by concentrated beryllium sulphate solutions. As such a solution approaches saturation with the base, any impurities present, other than the alkali metals, are separated as hydroxides. After saturation, final purification is obtained by the use of hydrogen sulphide. On largely diluting the resulting liquor beryllium hydroxide with absorbed sulphate is precipitated. The dilute liquor after filtration is reconcentrated in a low-temperature evaporator to its original bulk, when it can again dissolve further beryllium oxide or hydroxide. The precipitated product is washed, then given a reducing roast with carbon black at 700°, which converts the absorbed sulphate into sulphide. An oxidising roast at the same temperature converts the entire mass into oxide of very high purity.

Apparatus for the determination of carbon dioxide in water supersaturated with the gas. ANON. (Chem.-Ztg., 1929, 53, 749).—The water (e.g., mineral water) is contained in a bottle fitted with a cork which is tapped by driving through it a cork borer with a hole in the side of the tube about half-way down, and with a glass tube and stopcock fitted tightly over the upper end and sealed with sealing wax. The gas is drawn off through an absorption vessel by opening the stopcock and warming the bottle gently in hot water. After cooling, the gas still remaining in the water is determined by titration with 0.1N-sodium hydroxide, using phenolphthalein as indicator, and the amount found is added to that collected in the absorption vessel.

A. R. POWELL.

Utilisation of seaweed. T. DILLON and E. F. LAVELLE (Econ. Proc. Roy. Dubl. Soc., 1928, 2, 407—413).—An investigation has been made of the products formed by the natural decay of seaweed. About 31 kg. of freshly-gathered *Laminaria* were allowed to decompose in the open for 7 weeks, the liquid portion (31 litres) being then drawn off and the residue exposed to the weather for 10 weeks, after adding fresh water. The combined extracts contained 21.5 g. of iodine and 684 g. of mineral matter (29% of the total). A portion of the extracts (34 litres) acidified with concentrated sulphuric acid (2.5 c.c. per litre of extract) was distilled and the acid distillate evaporated down after being rendered feebly alkaline with sodium carbonate. The solid residue (300 g.) on distillation with sulphuric acid under reduced pressure gave a mixture of acids having an odour of acetic acid and boiling range 99—162°. Very little nitrogen was extracted from the seaweed during the decaying process. After nine months a large part of the seaweed had not decomposed, but this residue was considered suitable for fuel when dry. H. J. DOWDEN.

Analysis of photographic products etc. BAINES.
—See XXI.

PATENTS.

Production of alkaline-earth oxides. W. L. LAWSON (U.S.P. 1,729,428, 24.9.29. Appl., 8.10.27).—A closed vessel containing a mixture of an alkaline-earth carbonate and a carbon catalyst is heated at a sufficiently high temperature (not above 1538°) to effect decomposition of the carbonate without substantial consumption of the catalyst, the evolved gases are withdrawn, and the products of the reaction are cooled under conditions precluding oxidation of the catalyst.

H. ROYAL-DAWSON.

Production of anhydrous aluminium chloride. I. G. FARBENIND. A.-G. (B.P. 305,578, 4.2.29. Ger., 7.2.28).—Material containing alumina and silica is treated in the presence of a reducing agent, *e.g.*, carbon or carbon monoxide, with a mixture of chlorine and silicon tetrachloride vapour; when the mixture contains about equal proportions by weight, the initial quantity of silicon tetrachloride is recovered from the products for re-use, but an excess of it may be obtained, if desired, by increasing the proportion of chlorine in the mixture.

L. A. COLES.

Production of iron-free potash alum from solutions containing ferric sulphate. A. J. MOXHAM, Assr. to ELECTRO Co. (U.S.P. 1,732,611, 22.10.29. Appl., 13.6.27).—Crude potash alum is crystallised from solutions containing potassium, aluminium, and iron sulphates, the crystals are dissolved in water, a reagent to react with the ferric sulphate is added, and, after separation of the precipitate, the iron-free potash alum is recrystallised.

W. G. CAREY.

Manufacture of yellow ferric hydroxide. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 320,409, 12.7.28).—Precipitated ferrous hydroxide or carbonate is oxidised above 40°, preferably by a current of air, in the presence of a salt or hydroxide of a trivalent metal; *e.g.*, sodium carbonate solution is run into a solution containing ferrous sulphate and ferric chloride maintained at 90° and treated with a current of air until the precipitate is of a clear yellow colour, when the product is collected, washed, and dried. L. A. COLES.

Treating the product of reaction resulting from the treatment of raw phosphates or other phosphate-containing substances with acids. STOCKHOLMS SUPERFOSFAT FABR. AKTIEB. (B.P. 300,903, 15.11.28. Swed., 19.11.27).—Solutions containing acid calcium phosphate are treated with ammonium or an alkali sulphate and, after removal of the gypsum, the solutions are treated with excess of ammonia at about 0° and, if desired, under raised pressure to precipitate triammonium phosphate or a mixture of it with a trialkali phosphate.

L. A. COLES.

VIII.—GLASS; CERAMICS.

Composition and hydrolytic stability of moulded glass. C. A. BECKER and C. KRAFT (Sprechsaal, 1929, 62, 261—262; Chem. Zentr., 1929, i, 3024—3025).—A comparison of moulded glass containing SiO₂ 74.5—76.0, CaO 4.5—8.5, and alkali 15.0—20.0% by two methods.

A. A. ELDRIDGE.

Testing of glass for chemical resistance. L. SPRINGER (Sprechsaal, 1929, 62, 187—190, 206—209; Chem. Zentr., 1929, i, 3027—3028).—The results of numerous tests are recorded. Glasses of similar composition frequently behave differently owing to differences in thermal history.

A. A. ELDRIDGE.

Colouring of glass by bismuth. II. K. FUHA (Mazda, Japan, 1928, 3, 295—324).—Various colours are obtained, according to the composition of the glass, by adding bismuth oxycarbonate or nitrate. Potassium nitrate may be added, but arsenious acid probably causes precipitation of bismuth.

CHEMICAL ABSTRACTS.

Preparation of experimental sagger bodies according to fundamental properties. R. A. HEINDL and L. E. MONG (Bur. Stand. J. Res., 1929, 3, 419—444).—The mechanical and thermal properties of 55 sagger mixes representing 39 different bodies prepared from 15 different clays and grogs made from them and fired at 1230° or 1270° have been determined and the results recorded in a number of tables and graphs. Most of the bodies were made from two clays and the corresponding grogs, both coarse-grained and fine-grained grogs being tested. The bodies could be classified into three groups according to whether the linear thermal expansion exceeded that of either clay, was equal to the mean of those of the two clays, or was equal to that of the clay with the lower expansion. Generally the mixes made with coarse-grained grog had a lower expansion than those made from fine-grained grog. A direct relation between the modulus of elasticity and the transverse strength and between the porosity and plastic flow of the bodies under load at 1000° is indicated. If R is a factor indicating the relative resistance to failure under thermal shock, M is the modulus of rupture, E the modulus of elasticity, e the coefficient of linear thermal expansion, and P the porosity, it is shown that $R = M/EeP$. The value of R decreases with rise of firing temperature, but increases with the porosity of the grog. The best sagger bodies appear to be those having a low modulus of elasticity, high transverse strength, a porosity below 25%, and a low and uniform thermal expansion between 20° and 250°.

A. R. POWELL.

Japanese kaolinite clays. M. MASUDA (J. Japan. Ceram. Assoc., 1928, 36, 433—439).—A clay produced at Jishahara, Sawatari, and consisting chiefly of minute kaolinite crystals and containing a little hydromica but no colloidal kaolin, is described. Analysis indicated: SiO₂ 47.06, Al₂O₃ 36.48, Fe₂O₃ 0.93, CaO 0.36, MgO trace, alkalis (diff.) 2.25, loss on ignition 14.02%.

CHEMICAL ABSTRACTS.

Sp. gr. of trass. A. STEPOPOE (Tonind.-Ztg., 1929, 53, 618—620; Chem. Zentr., 1929, i, 3028).—Variations in the value of the measured sp. gr. according to the state of the trass and the nature of the oil employed are described.

A. A. ELDRIDGE.

IX.—BUILDING MATERIALS.

Chemical composition of wood in relation to physical characteristics. H. E. DADSWELL and L. F. HAWLEY (Ind. Eng. Chem., 1929, 21, 973—975).—The chemical composition of wood samples from the

same tree, but having different physical properties, has been determined for various types of timber. A certain type of brash oak has a slightly higher lignin content and slightly lower cellulose content than tough wood from the same species. Compression wood has a higher lignin content and lower cellulose content than normal wood from the same tree. Summer wood bands isolated from redwood compression wood were found to have a higher lignin content than spring wood from the same annual growth rings. It is concluded that strength cannot be deduced from lignin content without knowing the portion of the wood structure concerned.

C. J. SMITHELLS.

Toxicity of water-soluble extractives and relative durability of water-treated wood flour of Western red cedar. A. M. SOWDER (Ind. Eng. Chem., 1929, 21, 981—984).—The resistance of red cedar (*Thuja plicata*) to attack by fungi has been ascribed to the presence of a toxic resin, and an attempt has been made to determine the influence of kiln-drying at 80° on the powers of resistance of the resultant timber. Extractions were performed on fine wood flour from heartwood and sapwood, both seasoned and unseasoned, with hot and with cold water. Seasoned heartwood gave a higher percentage of soluble material than did the unseasoned, but sapwoods gave about the same. The toxicity of the extracts was measured by the retardation of growth of a rot-causing fungus on malt agar. The undiluted heartwood extracts proved fatal to the fungus, and the remaining extracts were toxic to smaller degree, that from unseasoned sapwood being least effective. Culture tests on the wood flour after extraction showed that both hot and cold water treatment removed sufficient material to permit the fungus to grow, the weight of wood destroyed being, with one exception, comparable with the amount of material extracted. To determine the influence of kiln-drying, blocks of wood were heated in an electric oven at 80—100°, the volatile matter being condensed and its toxicity tested. It is concluded that kiln-drying has a negligible effect, any loss of toxic material being counterbalanced by the sterilising action of the heat treatment.

H. J. DOWDEN.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Inner structure of the pearlite grain [in steel]. N. T. BELAIEV (Rev. Mét., 1929, 26, 424—426; cf. B., 1925, 635).—In a micro-section of pearlite, if ω is the angle between the plane of the section and the equatorial plane, i.e., a plane perpendicular to the lamellæ, and Δ_ω and Δ_0 are the apparent and real distances, respectively, between any two lamellæ, then $\cos \omega = \Delta_\omega / \Delta_0$. It is suggested that the value of Δ_0 depends on the thermal conditions during the A₁ transformation and could be used to characterise the steel. As previously recorded (*loc. cit.*), the probability of obtaining a section in the equatorial plane was shown to be 20%, whilst that of obtaining a meridional section was 10%. Crystalline ferrite consists of small cubes of dimensions about 250 $\mu\mu$, whilst Δ_0 for steel not thermally treated is 300—350 $\mu\mu$; it is probable therefore that the width

of the ferrite lamellæ tends to be limited by the size of the cube. There is evidence that the mechanical qualities of pearlite are influenced by the value of Δ_0 .

M. E. NOTTAGE.

Influence of fineness of structure on the annealing of grey cast iron. A. PORTEVIN and P. CHÉVENARD (Compt. rend., 1929, 189, 759—761).—Two long cylinders, diam. 30 and 100 mm., were cast simultaneously from each of four samples of cast iron (C 2.6—3.3%, Si 0.9—2.2, Mn 0.5—1.2, S 0.06—0.08), and annealed by cooling from various temperatures from 900° downwards at an average rate of 1.4° per min. above 600°. The hardness of, and amount of combined carbon in, test-pieces from the centres of the bars show that graphitisation in the 30-mm. bars takes place much more quickly than in the 100-mm. bars. These conclusions are confirmed by micrographic and dilatometric examination, and are in agreement with the results of Le Thomas (B., 1929, 981). They explain the more extensive graphitisation of the external portions of a casting, and the considerable variation in hardness of a fractured surface thereof.

C. A. SILBERRAD.

Testing the hardness of chilled [iron] castings. O. KEUNE (Papier-Fabr., 1929, 27, 650—653).—Measurements of the hardness of chill castings by the scleroscope are shown to vary appreciably with different instruments of the same type; hence it is recommended that the instrument be standardised with a hard steel, the Brinell number of which is known, and that all readings be converted into Brinell numbers for reporting. The desired Brinell number, B_x , is given by the equation $B_x = B_1 + (S_2 - S_1) \cdot B_1 / S_1$, where B_1 is the Brinell number of the standard, S_1 its scleroscope number, and S_2 the scleroscope number of the specimen under test.

A. R. POWELL.

Determination of nickel in steels. C. L. THOMAS (J. Elisha Mitchell Sci. Soc., 1928, 43, 214—216).—If the steel is not highly alloyed and does not contain more than 1% Ni, satisfactory results are obtained by precipitating the fully oxidised solution with ammonia and precipitating the nickel in the filtrate with a 3% dimethylglyoxime solution.

CHEMICAL ABSTRACTS.

Determination of cobalt in steel. J. W. HEYMANS (Natuurwetensch. Tijds., 1929, 11, 151—153).—In the separation of iron by precipitation as ferric hydroxide by addition of zinc oxide, loss of cobalt in the precipitate may be avoided by adding the zinc oxide to the cold dilute solution, instead of to a hot less dilute solution, as previously suggested.

S. I. LEVY.

Hot tests on metals and alloys by compression and rolling. A. PORTEVIN (Rev. Mét., 1929, 26, 435—443; cf. B., 1926, 280).—Cylindrical test-pieces of pure magnesium and its alloys with aluminium, zinc, and copper were rolled at temperatures up to 500°. Up to 250° the cylinders broke by shearing at 45°, the broken pieces then reunited as the temperature increased, and at still higher temperatures the cylinders became flattened out without breaking (cf. Robin, B., 1911, 806). The compression curves show two distinct effects of rolling, viz., elastic deformation followed by (a) a permanent deformation with hardening up to the breaking point, and (b) viscous crushing without hardening,

which proceeds indefinitely without rupture. Each metal at its drawing temperature is characterised by a very low elastic limit, above which deformation with hardening first sets in up to a limit of about 6 kg./mm.², followed by viscous crushing under constant pressure.

M. E. NOTTAGE.

Separation and determination of arsenic [in metals]. B. S. EVANS (Analyst, 1929, 54, 523—535).—To determine arsenic in copper a 5-g. sample is dissolved in 20 c.c. of 1 : 3 sulphuric acid and 10 c.c. of concentrated nitric acid, and the solution evaporated to the point where drops of nitric acid which collect on the cover glass are entirely evaporated. After cooling, the copper sulphate is taken up in 75 c.c. of water, the solution boiled, transferred to a flask, 2—3 g. of sodium hypophosphite are added, and the liquid is warmed. After discharge of the colour 10 g. of hypophosphite are added, the liquid is boiled for 15 min. under reflux, cooled, filtered, the precipitate washed first with 100 c.c. of 1 : 3 hydrochloric acid to which 2—3 g. of hypophosphite have been added, and then 6—7 times with 5% ammonium chloride solution. The filter is transferred to a beaker, the funnel rinsed in, a measured excess of standard iodine run in, and sufficient titrated water (distilled water and starch titrated with 0.01*N*-iodine to a faint blue) added to cover the broken-up pulp. The whole is stirred, left for 5 min., diluted to 300 c.c. with titrated water, about 2 g. of sodium bicarbonate are added, and the solution is immediately titrated with arsenious oxide solution of the same normality as the iodine used. An approximate end-point is reached, 1—5 c.c. excess arsenic solution run in, sodium bicarbonate (about 2 g.) is added, the solution shaken, left until discharge of the blue colour, and finally back-titrated with iodine, with vigorous shaking. The difference between the total volumes of iodine and arsenic solutions added gives the volume of iodine solution reduced by the precipitated arsenic. In the case of bronze the difficulty of precipitated arsenic reducing the tin is eliminated by addition of a small amount of hydrofluoric acid, but it is necessary to dissolve the first precipitate and reprecipitate. With plain carbon steel the addition of a small amount of copper obviates the slowness and uncertainty of the reduction of ferric salts, and the reaction of insoluble carbon in the sample with iodine in the titration is prevented by oxidation with potassium permanganate and subsequent reduction with sulphur dioxide of the manganese dioxide formed. Tungsten steels and any steels rendered insoluble by heating with sulphuric acid until fumes appear may be dissolved in presence of electrolytic copper by adding 1 : 3 sulphuric acid, concentrated nitric acid and hydrochloric acid, and saturated potassium permanganate solution, followed, after boiling, by a saturated solution of sulphur dioxide and, after again boiling, by syrupy phosphoric acid and water. After boiling down and cooling, an equal volume of hydrochloric acid is added followed by water, successive portions of sodium hypophosphite, a few drops of hydrofluoric acid, and more sodium hypophosphite. The liquid is boiled under a reflux and the process finished as above. A series of results by this method is given for some British chemical standard steels, and

comparative figures by other methods, the latter showing wide variations. In the case of "white metal A" standard, containing lead, antimony, tin, copper, iron, bismuth, zinc, and 0.06% of arsenic, the method gave 0.059% As.

D. G. HEWER.

Colorimetric determination of copper in babbitt metal. N. V. MANDRIKA (J. Appl. Chem., Russia, 1929, 2, 317—319).—The metal (0.5—1 g.) is heated with concentrated sulphuric acid (15—25 c.c.), diluted, treated with concentrated hydrochloric acid (50—150 c.c.) until the precipitate redissolves, and diluted to 250 c.c. To 25 c.c. is added hydrochloric acid until the total quantity present is 30 c.c., the solution is diluted to 50 c.c. and compared colorimetrically with a solution prepared by dissolving copper (0.5 g.) in nitric acid, evaporating to fuming with concentrated sulphuric acid (5 c.c.), diluting to 500 c.c., and adding to 62.5 c.c. 120 c.c. of concentrated hydrochloric acid and water up to 200 c.c.

CHEMICAL ABSTRACTS.

Volumetric determination of vanadium, iron, and uranium with titanium salts. V. G. CHLOPIN and L. E. KAUFMAN (J. Appl. Chem., Russia, 1928, 2, 91—108).—Reduction is effected by tervalent titanium (in an atmosphere of carbon dioxide) in the following order: $V^V \rightarrow V^{IV}$, $Fe^{III} \rightarrow Fe^{II}$, $U^{VI} \rightarrow U^{IV}$, $V^{IV} \rightarrow V^{III}$. Quinquevalent vanadium is titrated with 0.05—0.1*N*-titanous solution, potassium ferrieyanide and ferric iron being used as indicators; ferric iron is determined by Knecht and Hibbert's method using potassium thiocyanate as indicator, whilst hexavalent uranium is titrated with Neublau G (C) as indicator.

CHEMICAL ABSTRACTS.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Light-fastness of lithographic ink pigments. W. D. APPEL and R. F. REED (Bur. Stand. J. Res., 1929, 3, 359—374).—A series of 136 specially prepared lithographic prints made from 31 different pigments were exposed to daylight in several different ways and to the light of a carbon arc enclosed in glass. The degree of fading was measured in a fadeometer, and from the results obtained the pigments have been classified according to their light-fastness. The concentration of the pigment in the ink and the effect of light on the varnish appreciably influence the results. A. R. POWELL.

Comparative investigation on oil- and nitro-cellulose varnishes. H. WOLFF and W. TOELDTE (Farben-Ztg., 1929, 35, 231—232).—The influence of type of nitro cotton used, addition of ester gums and plasticisers, and the use of different types of resin on the general properties of nitrocellulose lacquers is discussed. The durabilities of oil- and cellulose varnishes on metal and on wood are also compared.

S. S. WOOLF.

Nature and constitution of shellac. II. Potentiometric titrations in 95% ethyl alcohol. W. H. GARDNER and W. F. WHITMORE (Ind. Eng. Chem. [Anal.], 1929, 1, 205—208; cf. B., 1929, 404).—The acidity of shellac can be accurately titrated potentiometrically in 95% alcohol, and a comparison has been made between the acidity of various samples of shellac and other soluble gums and organic acids. Shellac

gives solutions the acidity of which is comparable with those of glutaric, benzoic, lauric, and cinnamic acids, and which are only slightly more acidic than Manila copal, kauri, and rosin solutions. E. H. SHARPLES.

PATENTS.

Manufacture of [bleachable] printer's inks. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,430, 23.7.28).—Printer's inks are based on black, acid, disazo dyes for wool, *e.g.*, Palatine Black, Agalma Black, Brilliant Black, and Diamond Black, in a water-insoluble form, preferably in the form of a lake, with or without the addition of other water-insoluble dyes or lakes, any water present being removed. The dyes are prepared in a fine state of subdivision by the use of dispersing media. Papers printed with such inks can readily be decolorised by hypochlorites etc. and worked up for fresh-printing paper. [Stat. ref.] S. S. WOOLF.

Lacquer. J. G. DAVIDSON, ASST. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,731,333, 15.10.29. Appl., 25.4.27).—A thinner for pyroxylin lacquers contains not more than 30% of ethyl acetate and ethyl alcohol, nor more than 20% of the monoethyl ether of ethylene glycol and its acetate, and a suitable hydrocarbon solvent.

H. ROYAL-DAWSON.

[Lacquer]-printing process. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 318,567, 5.6. and 27.10.28).—Anhydrous lacquers containing cellulose esters or ethers, with addition of a suitable pigment, are printed by means of deeply engraved plates or rollers on non-resilient bases, which may, if necessary, be rendered smooth and even by previous application of a coating of lacquer, varnish, etc., which is allowed to dry. The process is applicable to metals, wood, paper, etc.

F. R. ENNOS.

Urea-formaldehyde condensates. E. G. BUDD MANUF. Co., ASSECS. of L. SMIDTH (B.P. 294,253, 19.7.28. U.S., 21.7.27).—Formaldehyde, preferably in the form of slightly acid formalin, is condensed with urea in aqueous solution at a relatively low temperature, *e.g.*, 25–50°. The greater part of the water of reaction is removed by vacuum distillation at 50–100°, thiourea is added, and the solution is distilled *in vacuo* at 100–200° until as viscous as possible, nearly all the water being removed. The thiourea may be added before the first distillation. The condensation product is finally subjected to heat treatment, *e.g.*, 2–4 days at 70–95°. S. S. WOOLF.

Manufacture of condensation products of derivatives of urea and aldehydes. I. G. FARBENIND. A.-G. (B.P. 319,251, 7.5.28. Addn. to B.P. 261,029; B., 1928, 532).—The dimethylolurea referred to in the prior process is replaced by dimethylolthiourea, the products of higher mol. wt. resulting therefrom by elimination of water, or mixtures of these with each other or with dimethylolurea or its higher condensation products. The condensation with aldehydes is conducted in a considerable quantity of organic solvent until colloidal compounds are formed in the solution. [Stat. ref.] S. S. WOOLF.

XV.—LEATHER; GLUE.

Karri bark as a source of tannin. W. E. COHEN (J. Council Sci. Ind. Res., Australia, 1929, 2, 161—

165).—Samples of the bark of Karri (*Eucalyptus diversicolor*) for analysis must be dried to a moisture content of 20–30% as soon as possible after removal from the tree. The average water-soluble tannin content is about 17%. Difficulties in the drying and subsequent treatment of the bark for tannin production led to experiments with the green bark. Between stripping and treatment a considerable proportion of the tannin becomes insoluble, but by extraction in open vats for 1 hr. with water at 90–95° containing 2–3 pts. of sodium sulphite, or preferably sodium bisulphite, for every 100 pts. of bark (dry weight) almost theoretical yields are obtainable. The strong liquor is displaced by the press-leach system. A light-coloured clear extract is obtained which imparts a pale colour to hide and contains tans and non-tans in the ratio 2:1.

W. J. BOYD.

Tanning action of alcoholic tannin solutions. Tanning action of complex chromium salts. Effect of inert substances on alkaline swelling. G. GRASSER and H. NAKANISHI (J. Fac. Agric. Hokkaido Imp. Univ., 1929, 23, 87–90, 90–93, 93–110).—In alcoholic solution, formic acid (aqueous) has a marked, benzoquinone a slight, but oak-gall tannin, oak wood extract, quebracho extract, and neradol-D₁ no tanning action. Sodium chromioxalate was used for tanning in presence of sodium carbonate. Neutral salts first increase and then decrease alkaline swelling of skin; sugars decrease, and alcohols increase, swelling.

CHEMICAL ABSTRACTS.

Red and blue stains on damp chrome[tanned] leather. M. BERGMANN and F. STATHER (Collegium, 1929, 326–327).—A pack of chrome-tanned skins was left in a heap in the neutralised condition for 5 weeks, when red spots up to 2 mm. diam. appeared. Cultures of actinomyces were obtained from them, and also from blue specks on chrome-tanned leather, consisting of two thread-shaped bacteria, one rose-yellow, and the other deep rose coloured. Various colourless bacterial colonies were also obtained. D. WOODROFFE.

Determination of fat and water-soluble [matter] in leather. III. Fundamental significance of the retention coefficient, *K*. A. COLIN-RUSS (J. Soc. Leather Trades' Chem., 1929, 13, 422–426; cf. B., 1926, 70).—True water-soluble matter, like the fat in leather, exists in the form of a solid solution in the collagen-tannate complex, but not necessarily so. The equilibrium state of the system leather-adsorbent-solvent is subject to three opposing forces, viz., the lubricating action of the solid phase structure, the physical solvent action of the liquid phase, and the hydrolytic action of the liquid phase, and is expressed by $\bar{K} = E_2 / (E_1 - E_2) \times (V/m)$, where *V* is the volume of the successive quantities of solvent used on the leather, and *m* the weight of the leather when all the adsorbent has been removed. The value of *K* is not constant, but tends to increase with *V*. By plotting the experimental ratios of *E*₁/*E*₂ against *V*/*m* a new equation has been derived, the significance of the constants in which is discussed. The maximum value of *K* can be determined; it is intrinsically characteristic of a given type of leather and independent of the state of subdivision of the leather sample. D. WOODROFFE.

XVI.—AGRICULTURE.

Determination of total carbon in soils. E. WINTERS, JUN., and R. S. SMITH (Ind. Eng. Chem. [Anal.], 1929, 1, 202—203).—The dry combustion method for the determination of total carbon in soils (Salter, A., 1916, ii, 491) gives low results. The method and apparatus have been modified and, by the introduction of manganese dioxide into the boat, complete oxidation is ensured and the combustion is complete in 10 min. Results quite comparable with those obtained by the wet combustion method are obtained. E. H. SHARPLES.

Cobaltinitrite method for determination of potash with particular reference to soils. J. H. DENNETT (Malayan Agric. J., 1929, 17, 341—350).—The characteristics of potassium sodium cobaltinitrite precipitated under varying conditions are described, and the centrifugal method of separation is advocated. For rapid determinations not involving the necessity for a high degree of accuracy, and when the amount of potash lies between 2 and 33 mg., the dissolution of the precipitate in sulphuric acid and back-titration with sodium carbonate, using bromothymol-blue as indicator, is suggested. For more accurate determinations dissolution of the precipitate in acid potassium permanganate, addition of excess of 0.1*N*-oxalic acid, and back-titration with potassium permanganate is preferable. Modifications of the method for soil analysis, including the determination of potash in citric acid solution, i.e., "available" potash, are described. E. HOLMES.

Root-solubility of phosphoric acid contained in superphosphate, neutral phosphate, reform phosphate, and Algiers phosphate. C. DREYSPRING, C. KRUSEL, and R. PANTKEE (Superphosphate, 1929, 2, 61—69, 81—89).—The quantities of phosphorus pentoxide absorbed by barley seedlings in 18 days with applications of 5—80 mg. P_2O_5 per pot were: superphosphate 4.2—53.3, neutral phosphate 3.2—39.1, reform phosphate 1.4—10.7, Algerian phosphate rock 1.2—2.4 mg. The values were closely related to the amounts of phosphorus soluble in water or ammonium citrate solution. CHEMICAL ABSTRACTS.

Spontaneous combustion of hay. C. A. BROWNE (U.S. Dept. Agric. Tech. Bull. No. 141, 1929, 39 pp.).—An examination of the spontaneous combustion of oil-soaked cotton wool leads to the theory of the production in haystacks of unstable unsaturated substances by anaerobic bacteria. Subsequent heating of the stack depends on the rate at which oxidation of these intermediate substances occurs, this being controlled by the accessibility of air and the presence of sufficient water vapour as the reacting medium. The nature of the intermediate products is indicated by the isolation of hydroxyacrylic acid from the products of the reaction of dextrose and dilute lime water in the absence of oxygen. An extensive review of the literature on this subject is included. A. G. POLLARD.

Substitutes for lead arsenate as a soil insecticide. J. W. LIPP (J. Econ. Entomol., 1929, 22, 600—601).—When mixed with soil (1500 lb. per acre), sodium or barium fluosilicate, mercuric borate or iodide, or mercurous chloride kills the larvæ of *Popillia japonica*. The first three substances do not affect the growth of grasses,

although sodium fluosilicate tends to harden soil and prevent the germination of grass seed. The other substances were not tested for plant tolerance.

CHEMICAL ABSTRACTS.

XVIII.—FERMENTATION INDUSTRIES.

Proteins in brewing. S. B. SCHRYVER (J. Inst. Brew., 1929, 35, 532—540).—From determinations of the total, ammonia-, amino-, amide, and peptide nitrogen present in malt wort at intervals during its fermentation by yeast, the nitrogenous substances in wort may be classified into two groups. The first section includes those nitrogenous substances which can act as nutrients for yeast, whilst the second class comprises those which cannot be utilised as food material, which are largely colloidal in nature, and which may during the course of fermentation separate out with the growing yeast and so inhibit its normal growth. These colloidal forms are probably of the nature of "humin" and are produced during kilning by the combination of the peptones or their derivatives with the carbohydrates of the grain.

C. RANKEN.

Influence of disinfectants on the diastatic power of steeped barley. B. LAMPE (Z. Spiritusind., 1929, 52, 329).—Several samples of barley were steeped in water for 16 hrs., drained for 8 hrs., steeped in dilute disinfectant solution for 16 hrs., drained for 8 hrs., and subsequently kept moist. The diastatic power, and in some cases acrospire length, was compared at intervals against controls with the disinfectant omitted. A 0.025% solution of "montanin" was without effect on acrospire growth and raised the diastatic power slightly. Formalin at the rate of 0.15—0.20% had a tendency to reduce the diastatic power, but in neither case was the difference of any technical significance. F. E. DAY.

Physics of the "Läuterbottich." G. JAKOB (Woch. Brau., 1929, 46, 427—432, 440—446, 449—454).—A discussion of the conditions of separating the wort from the grains, sparging, and transferring to the copper. A better filter-bed results from the use of a described device for reducing the velocity of the entering mass. The nature of the grist and the viscosity of the wort in relation to temperature and concentration are considered, also the theory of washing out the extract by sparging, and the influence of the state of the grains on diffusion. To obtain even washing out of extract it is of advantage to separate the bottom space into portions controlled by each wort tap; also to provide means for measuring the fluid pressure at the taps, as the difference between this and the hydrostatic pressure due to the liquor on the grains determines the rate of filtration. Means for avoiding aeration of the wort during transference to the copper are mentioned; excessive relative darkening during sparging may be avoided by use of water at a lower temperature towards the end of the process.

F. E. DAY.

Colorimetric determination of higher alcohols in spirits. T. VON FELLEBERG (Mitt. Lebensm. Hyg., 1929, 20, 16—29; Chem. Zentr., 1929, i, 2932).—Values for the higher alcohols determined by Röse's method for grape marc spirits are too low, since methyl alcohol is present, and the colorimetric method is uncertain,

since various higher alcohols give colorations of different intensities with salicylaldehyde and sulphuric acid. *p*-Hydroxybenzaldehyde is therefore preferred to salicylaldehyde.

A. A. ELDRIDGE.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Essential oil from green violet leaves. H. WALBAUM and A. ROSENTHAL (Ber. Schimmel, 1929, 209—218).—Extraction with alcohol and subsequent steam-distillation of the extract of the leaves of the French violet gives an oil, d^{15}_4 0.9270, n^{20}_D 1.47642 (yield 30.8 g./kg.), which by fractional distillation gives an aldehyde $C_9H_{14}O$ (I), b.p. 82—86°/6 mm., d^{15}_4 0.9038, n^{20}_D 1.47446, α_D —1.22° (? in 1-dm. tube), which is oxidised with silver oxide to the corresponding acid $C_9H_{12}O_2$ (II), b.p. 132—136°/5 mm., d^{15}_4 0.9911, which is converted by the action of methyl sulphate on its sodium salt into its methyl ester, b.p. 88—90°/5 mm., d^{15}_4 0.9338, n^{20}_D 1.46191, α_D 0°. The last-named is reduced by palladium and hydrogen (with addition of 4 atoms of hydrogen) to methyl nonoate, the identity of which is proved by conversion into the amide and free acid and direct comparison with genuine specimens. Oxidation of the acid II with potassium permanganate in neutral solution at 0° yields acetic and oxalic acids only; the molecular refraction of I agrees with the presence of two unconjugated double linkings, and it is therefore a nonadienal. No hexenals could be detected.

J. W. BAKER.

Presence of sulphur compounds in mignonette extract. H. WALBAUM and A. ROSENTHAL (Ber. Schimmel, 1929, 219—221).—Phenylethylthiocarbimide, known to be present in the extract of the root of mignonette, is also isolated from the rest of the plant by steam-distillation of the wax-free extract and conversion into phenylethylthiocarbamide by the action of alcoholic ammonia.

J. W. BAKER.

Constituents of dwarf pine oil. H. WEINHAUS and H. NAHME (Ber. Schimmel, 1929, 233—265).—Large quantities of three different samples of dwarf pine oil (*Pinus pumilio* and *montana*) have been carefully fractionated and the constituents of each fraction determined. The presence of α -pinene, Δ^3 -carene, phellandrene, a sesquiterpene alcohol, and bornyl acetate is confirmed. From the fractions of lowest b.p. *n*-hexaldehyde is isolated by the action of neutralised sodium hydrogen sulphite. Traces of cuminaldehyde are also probably present. By distillation of the terpene fractions of lowest b.p. over sodium, α -pinene is obtained and is characterised by conversion into its nitroschloride, nitrosopinene, and pinocamphoroxime, whilst the presence of β -pinene in the fraction b.p. 162—166° is proved by the isolation of nopinic acid after oxidation with alkaline potassium permanganate. From the fraction having b.p. 167—175° Δ^3 -carene is isolated as its nitrosate, whilst phellandrene is obtained as its nitrite from the fraction b.p. 44—46°/10 mm. The fraction b.p. 175—180° contains dipentene and limonene (the small laevorotation suggesting the presence of only a small proportion of the latter), isolated as their tetrabromides. Sabinene is not present. Although carbonyl compounds are chiefly responsible for the odour, they only constitute 0.15% of the oil. By means of sodium

sulphite in the presence of sodium hydrogen carbonate 4-isopropyl- Δ^2 -cyclohexen-1-one (also obtained by the auto-oxidation of β -phellandrene) and an unsaturated, dicyclic ketone $C_{10}H_{14}O$ (semicarbazone, m.p. 220—222°) are isolated from the fractions of higher b.p. On the basis of its molecular composition and physical data the existence of an unsaturated dicyclic ketone pumilone (Böcker and Hahn, A., 1911, i, 519) is denied. Further treatment with concentrated sodium hydrogen sulphite yields no solid derivative, but after treatment of the aqueous liquor with sodium carbonate and steam-distillation anisaldehyde is obtained. By esterification of the fraction b.p. 80°/12 mm. with boric acid the absence of free borneol is proved, but after partial esterification of the fraction b.p. 80—122° and above/10 mm. with phthalic anhydride at 100° and subsequent partial hydrolysis of the ester fraction with cold 10% sodium hydroxide, borneol is obtained. No appreciable quantity of primary alcohols, either free or as esters, is present. Re-treatment of the unesterified portion, in the separation of borneol with phthalic anhydride at 130°, and subsequent hydrolysis yields more borneol (which is not completely separated from tertiary alcohols by the phthalic anhydride method) and an unsaturated monocyclic terpene alcohol $C_{10}H_{18}O$ named menthenol P, b.p. 103—106°/7 mm., d^{20}_4 0.9316, n^{20}_D 1.48082, α_D —12.8° (? in 1-dm. tube), reduced by palladised charcoal and hydrogen to menthanol P, b.p. 92—96°/6 mm., d^{20}_4 0.9123, n^{20}_D 1.46905, α_D —10.4°, and oxidised with Beckmann's mixture to a ketone $C_{10}H_{16}O$ isolated as its semicarbazone, m.p. 240—242°. Further treatment of the high-boiling fraction with phthalic anhydride at 130° and subsequent hydrolysis yields an unsaturated sesquiterpene alcohol pumiliol, $C_{15}H_{22}O$, b.p. 140—150°/3 mm., d^{20}_4 0.9674, n^{20}_D 1.50364, α_D —8.3°, which takes up 1 mol. of hydrogen in the presence of palladised charcoal in methyl alcohol, but no solid semicarbazone could be obtained from the product of oxidation of the dihydro-compound. From the 10% sodium hydroxide solution (above) after partial hydrolysis of the high-boiling fractions which have been treated with phthalic anhydride, acetic, propionic, and hexoic acids are isolated. Distillation of the high-boiling fractions over sodium, after removal of the alcohols and esters gives cadinene, isolated as its dihydrochloride, but the small yield of this derivative together with the dextrorotation of the high-boiling fractions suggest that it is only present in small quantity together with other sesquiterpenes.

J. W. BAKER.

Umbellulone and umbellularia oil. H. WIENHAUS and K. TODENHÖFER (Ber. Schimmel, 1929, 283—295).—Contrary to Power and Lees (B., 1904, 559) umbellulone (I), b.p. 85°/5 mm., d^{20}_4 0.949, n^{20}_D 1.48315, α_D —38.8° (? in 1-dm. tube), may be isolated from the oil of the Californian laurel (*Umbellularia californica*, Nutt.) by shaking with aqueous sodium sulphite and sodium hydrogen carbonate, and decomposition by steam-distillation of the aqueous solution of the soluble additive compound so obtained. Reduction of I with hydrogen at 75°/15 mm. and a nickel catalyst in alcoholic solution converts it directly into dihydroumbellulone II, b.p. 67°/4 mm., d^{20}_4 0.9275, n^{20}_D 1.46052, α_D —55.7° (oxime, m.p. 72°; lit. describes as a liquid), which is

reduced by sodium and alcohol to dihydroumbellulol, m.p. 55–56°, the product of the direct reduction of I with this reagent (Semmler, A., 1908, i, 92). Reduction of II at a higher temperature with sodium in toluene solution causes fission of one of the rings with the formation of a *hexahydro*-derivative, $C_{10}H_{20}O$, b.p. 80°/5 mm., d_{20}^{20} 0.902, n_D^{20} 1.46052, α_D –38.3° (purified through its borate), which is probably 1:2-dimethyl-3-isopropylcyclopentan-4-ol (possibly identical with the "tetrahydroumbellulol" of Power and Lees, *loc. cit.*). This is oxidised by chromic acid to a *ketone*, $C_{10}H_{18}O$, b.p. 73°/7 mm., d_{20}^{20} 0.902, n_D^{20} 1.45182, α_D –62.4° (*semicarbazone*, m.p. 177–178°; liquid *oxime*), reconverted by reduction into the parent alcohol. By means of the boric acid method *l*- α -terpineol is isolated from the fraction of the oil b.p. 70–87°/4 mm., after removal of umbellulone with sodium sulphite. J. W. BAKER.

Concrete essence of lavender : its constants and chemical composition. Y. VOLMAR and O. THURKAUF (J. Pharm. Chim., 1929, [viii], 10, 199–204).—Two samples of the essence, one being dark green and the other colourless, had, respectively, d_{15}^{15} 0.9381, 0.9067; $[\alpha]_D$ –6° 40', –5° 36'; n_D^{20} 1.4811, 1.4667; acid value 9.36, 2.42; saponif. value 124.8, 123.6; saponif. value after acetylation 126, 127.9; acetyl value 9.1, 10.4; ester value 115.4, 121.2; loss at 100° 65.3%, 92.1%. Phenols, aldehydes, ketones, and nitrogen compounds were absent from both samples, which contained, apart from a small amount of free acids, only hydrocarbons, alcohols, and esters. Distillation of the first sample at 12–13 mm. gave 54.4%, b.p. 79–120°, containing 84.9% of geraniol and 15.1% of linalool, both partly as esters; 6.9%, b.p. 121–160°, containing cedrene; 7.9%, b.p. 160–200°, containing 62% of methylumbelliferone and 6.3% of coumarin; 9.9% of a brown viscous mass; and 19% of a green resinous mass containing chlorophyll decomposition products. The free acids present in the original essence were formic, acetic, butyric, and coumaric. The second sample contained no chlorophyll and gave fractions having similar compositions to the above on distillation; it contained 1.9% of methylumbelliferone and 0.2% of coumarin. E. H. SHARPLES.

Norwegian juniper oil. II. Terpenes and other constituents. A. JERMSTAD (Riechstoffind., 1929, 4, 44–45; Chem. Zentr., 1929, i, 3044–3045).—*l*- α -Pinene, sabinene, terpinene, and 4-terpineol, but not bornyl acetate or cadinene, were found.

A. A. ELDRIDGE.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES

Analysis of photographic products and raw materials. I. Determination of iodide in mixtures of halides. II. Determination of halide impurities in potassium iodide. III. Conversion of silver halides into alkali halides. IV. Rapid complete analysis of iodobromide emulsions. V. Stop-watch method for rapid determination of traces of copper in silver nitrate. H. BAINES (J.S.C.I., 1929, 48, 295–299, 299–300, 300–301, 301–302, 302–304 r).—I. The starch-nitrite spotting

method of determination of iodide in mixtures of halides (B., 1927, 813) has been examined in greater detail. The dependence of the titre on end-point volume and on quantity of iodide present when 0.01N- or more dilute silver nitrate is used has been examined quantitatively, and is ascribed partly to the inability of appreciable iodide concentrations to colour starch-nitrite paper, and partly to adsorption of iodide ions on silver iodide. A calibration table and curve for correction of titres in determinations of 4–20 mg. of iodine (as iodide) are given. The relative concentrations of starch and nitrite, the refinement of the end-point, and the acidity of the solution are discussed. Detailed experimental procedure is given.

II. The halide impurity in potassium iodide may be determined with rapidity to an accuracy of within 0.05% as follows:—To 5 g. of the iodide in 25 c.c. of water, 1 c.c. of ammonia (d 0.880) and 5 g. of silver nitrate in 25 c.c. of water are added; 5 c.c. of 3.6N-sulphuric acid are added, and the residual potassium iodide is titrated with 0.1N-silver nitrate, until one drop of solution fails to give a blue colour when spotted on a filter paper soaked in a mixture of 1 c.c. of 10% sodium nitrite solution and 40 c.c. of 2% starch solution. The total silver added defines the iodide content of the sample. An excess of 5 c.c. of 0.1N-silver nitrate is added, the precipitated bromides and chlorides are coagulated by slight agitation, and the silver halides collected and washed. The silver nitrate in the filtrate and washings is determined by titration with thiocyanate by Volhard's method. The difference between this value and 5 c.c. of 0.1N-solution defines the halide impurity in 5 g. of potassium iodide, 3 c.c. of 0.1N-halide corresponding to 1% of the total iodide.

III. Alkali halides are obtained quantitatively by precipitation of silver sulphide from a cyanide solution of the silver halides. The product is fixed in a convenient quantity of potassium cyanide solution, and as large an aliquot as possible removed and boiled. An excess of sodium sulphide is added, and the precipitate coagulated by boiling. The liquid is acidified, and the precipitate collected and washed. Hydrogen sulphide and cyanide are removed from the filtrate and washings by boiling.

IV. Silver, iodide, and (if necessary) bromide are determined by successive titrations on the same sample by a combination of Liebig's cyanometric method with the sulphide method of conversion (*supra*). Two half-plates of the product are successively fixed in 50 c.c. of 0.2N-potassium cyanide containing 5 g. of sodium hydroxide per litre. The silver content of an aliquot is determined by back-titration of excess cyanide with 0.1N-silver nitrate; N-sodium sulphide solution is added, and the solution boiled for 2 min., acidified with sulphuric acid, and the precipitate washed. Iodide in the filtrate and washings is determined by the starch-nitrite method. The average error per determination of silver iodide in photographic products was 2.3% of iodide, or 0.075% of total halides.

V. The method depends on measuring the accelerating effect of copper on the oxidation of thiosulphate by a ferric salt, the disappearance of the latter being indicated by thiocyanate.